Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

Electrochemical Decarboxylative C3 Alkylation of Quinoxalin-2(1*H*)-ones with *N*-hydroxyphthalimide Esters

Kaikai Niu,[†] Lingyun Song,[†] Yanke Hao,[†] Yuxiu Liu[†] and Qingmin Wang*[†‡]

[†]State Key Laboratory of Elemento-Organic Chemistry, Research Institute of Elemento-Organic Chemistry, College of Chemistry, Nankai University, Tianjin 300071, People's Republic of China

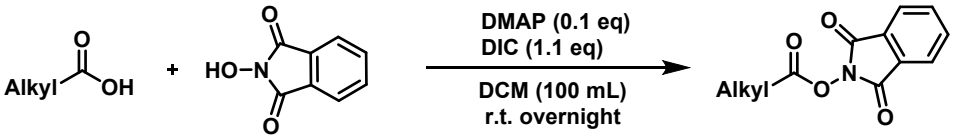
[‡]Collaborative Innovation Center of Chemical Science and Engineering (Tianjin), Tianjin 300071, People's Republic of China

Contents

1. General Information

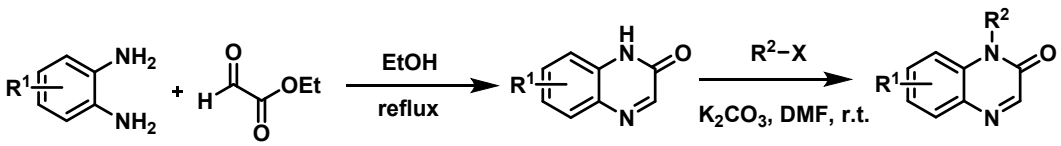
Unless otherwise noted, Reagents were purchased from commercial sources and were used as received. ^{1}H and ^{13}C Nuclear Magnetic Resonance (NMR) spectra were recorded on Bruker Avance 400 Ultrashield NMR spectrometers. Chemical shifts (δ) were given in parts per million (ppm) and were measured downfield from internal tetramethylsilane. High-resolution mass spectrometry (HRMS) data were obtained on an FTICR-MS instrument (Ionspec 7.0 T). The melting points were determined on an X-4 microscope melting point apparatus and are uncorrected. Conversion was monitored by thin layer chromatography (TLC). Flash column chromatography was performed over silica gel (100-200 mesh).

2. Preparation of *N*-hydroxyphthalimide ester



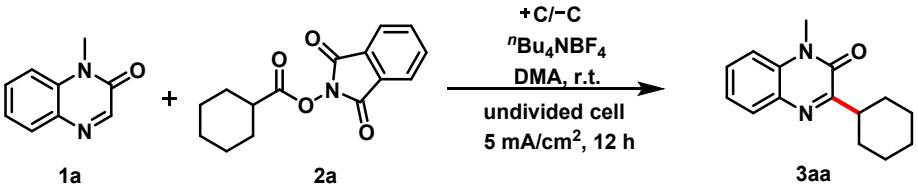
N-hydroxyphthalimide Esters was prepared from alkyl carboxylic acids following the procedure of Baran and co-workers[1] on 20 mmol scale. To a solution of the corresponding acid (20 mmol, 1.0 equiv.), 4-dimethylaminepyridine (244 mg, 2 mmol, 0.1 equiv.) and N, N'-diisopropylcarbodiimide (3.4 mL, 22 mmol, 1.1 equiv.) in DCM (100 mL) was stirred under air at room temperature for 5 min. Then, the NHPI (3.26 g, 20 mmol, 1.0 equiv.) was added into the solution and stirred overnight at room temperature. After reaction, the mixture was filtered and the filtrate was concentrated *in vacuo*. Finally, the residue was purified by chromatography on silica gel, eluted with petroleum/ethyl acetate to afford the desired product. The compound **2a-2q** was previously reported.

3. Preparation of quinoxalin-2(1*H*)-one



Quinoxalin-2(1*H*)-one was prepared from 1,2-phenylenediamines following the procedure of Cui and co-workers[2] on 5 mmol scale. To a solution of 1,2-phenylenediamines (5 mmol, 1.0 equiv.) in ethanol (40 mL) was added ethyl glyoxalate (6 mmol, 1.2 equiv.). The resultant reaction mixture was stirred at reflux until the raw material disappears. Then, the mixture was filtered and washed by ethanol. The solid was dried *in vacuo*. For alkylation, the corresponding halogenoalkane (1.6 equiv.) was added to a suspension of quinoxalinone (1.0 equiv.) and potassium carbonate (1.2 equiv.) in DMF (16 mL). The mixture was stirred at room temperature overnight. After complete reaction, brine was added, and then extracted three times with EtOAc. The combined organic layers were washed with a saturated solution of NH_4Cl then brine, dried over anhydrous Na_2SO_4, filtered and evaporated *in vacuo*. The residue was purified by column chromatography on silica gel to afford the desired product. The compound **1a-1q** was previously reported.

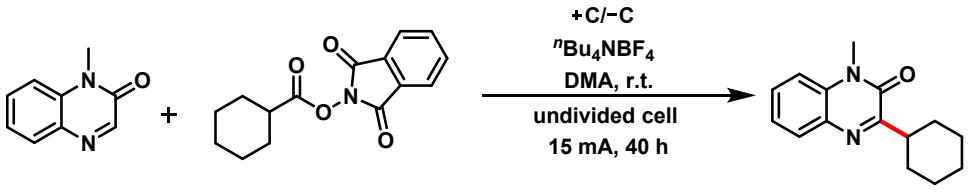
4. General procedure for the electrolysis



1a 2a 3aa

An oven-dried 10 mL undivided bottle was equipped with two graphite sheet electrodes (10 mm × 10 mm × 3 mm). The corresponding quinoxalin-2(1*H*)-one (80.0 mg, 0.5 mmol, 1.0 equiv.), the *N*-hydroxyphthalimide ester (273.1 mg, 1.0 mmol, 2.0 equiv.) and $^{n}Bu_4NBF_4$ (329 mg, 1.0 mmol, 2.0 equiv.) was added into the undivided cell. And then DMA (5 mL) was added. The reaction mixture was stirred and electrolyzed at a constant current of 5.0 mA under room temperature for 12 h. The reaction was quenched with brine (50 mL) and extracted with EtOAc (3 × 20 mL). Then the combined organic layers were filtered, dried over anhydrous Na_2SO_4 and concentrated *in vacuo*. The pure product was obtained by flash column chromatography on silica gel (petroleum ether/ethyl acetate).

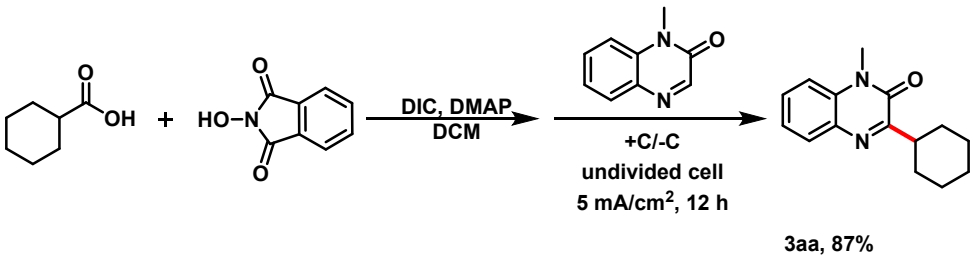
5. Gram-scale reaction



1a, 6 mmol 2a 1.28g, 88%

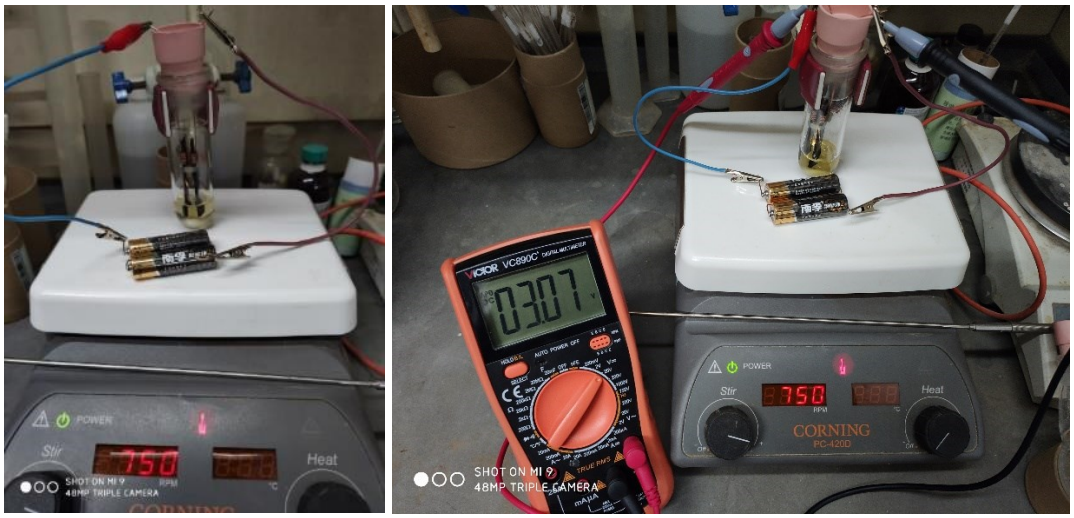
A 200 mL bottle with a stir bar was charged with quinoxalin-2(1*H*)-one (0.96 g, 6.0 mmol, 1.0 equiv.), *N*-hydroxyphthalimide ester (3.28 g, 12.0 mmol, 2.0 equiv.), $^{n}Bu_4NBF_4$ (3.95 g, 12.0 mmol, 2.0 equiv.) and DMA (50 mL). The bottle equipped with two graphite sheet electrodes (300 mm × 500 mm × 3 mm) were inserted into the mixture. The reaction mixture was electrolyzed under a constant current of 15 mA for 40 h. After the reaction, the electrodes were removed and rinsed with EtOAc. Brine was then added; the resulting mixture was extracted with EtOAc. The combined organic layer was dried over anhydrous Na_2SO_4 and concentrated *in vacuo*. The crude material was purified by column chromatography (petroleum ether/ethyl acetate = 10:1) to furnish the desired product in 88% yield as a white solid.

6. One-pot synthesis



An oven-dried 10 mL undivided bottle was charged with cyclohexanecarboxylic acid (128.2 mg, 1.0 mmol, 1.0 equiv.), 4-dimethylaminepyridine (24.4 mg, 0.2 mmol, 0.2 equiv.), *N*, *N*'-diisopropylcarbodiimide (0.2 mL, 1.2 mmol, 1.2 equiv.), NHPI (179.4 mg, 1.1 mmol, 1.1 equiv.) and DCM (1 mL). After stirred for 30 min, quinoxalin-2(1*H*)-one (80.0 mg, 0.5 mmol, 1.0 equiv.), $^{n}Bu_4NBF_4$ (329 mg, 1.0 mmol, 2.0 equiv.) and DMA (5 mL) was added into the undivided bottle. Two graphite sheet electrodes (10 mm × 10 mm × 3 mm) were inserted into the mixture, and the reaction mixture was electrolyzed under a constant current of 5 mA for 12 h. After the reaction, brine was then added and extracted with EtOAc. The combined organic layer was dried over anhydrous Na_2SO_4 and concentrated *in vacuo*. The crude material was purified by column chromatography (petroleum ether/ethyl acetate = 10:1) to furnish the desired product in 87% yield as a white solid.

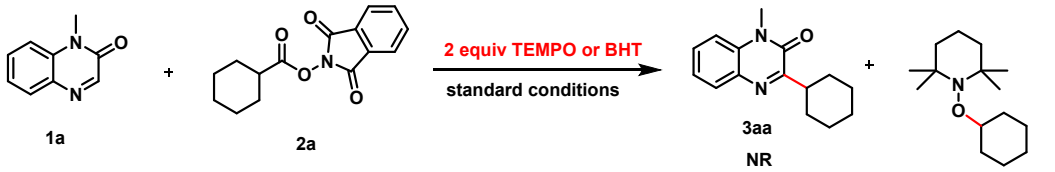
7. Use of 3-V battery as a readily available power source



An oven-dried 10 mL undivided bottle was charged with quinoxalin-2(1*H*)-one (80.0 mg, 0.5 mmol, 1.0 equiv.), *N*-hydroxyphthalimide ester (273.1 mg, 1.0 mmol, 2.0 equiv.), $^{n}Bu_4NBF_4$ (329 mg, 1.0 mmol, 2.0 equiv.) and DMA (5 mL). Two graphite sheet electrodes (10 mm × 10 mm × 3 mm) were inserted into the mixture. Two 1.5 v NanFu batteries were connected by copper wires in series and were used as the power source. After the reaction, the resulting mixture was was quenched with brine and extracted with EtOAc. The combined organic layer was dried over anhydrous Na_2SO_4 and concentrated *in vacuo*. The crude material was purified by column chromatography (petroleum ether/ethyl acetate = 10:1) to furnish the desired product in 90%

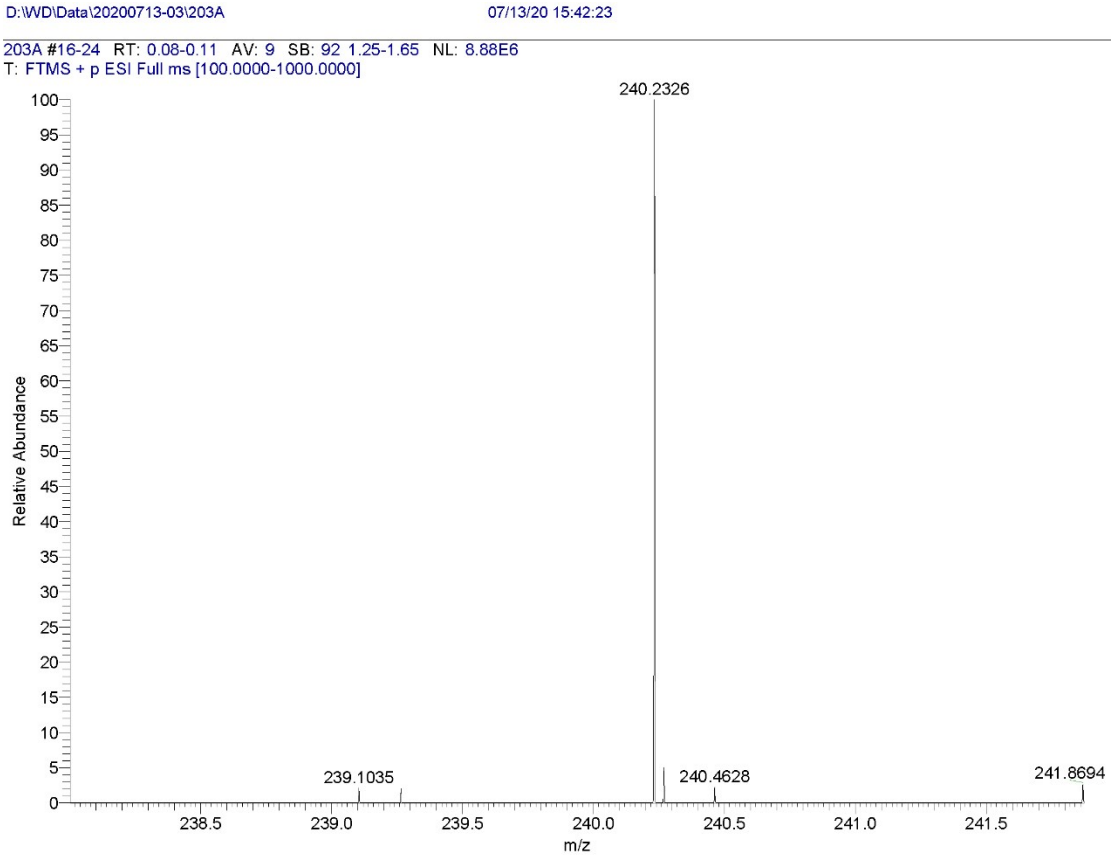
yield as a white solid.

8. Mechanistic Studies



Detected by HRMS
$[M+H]^+$: 240.2327
found: 240.2326

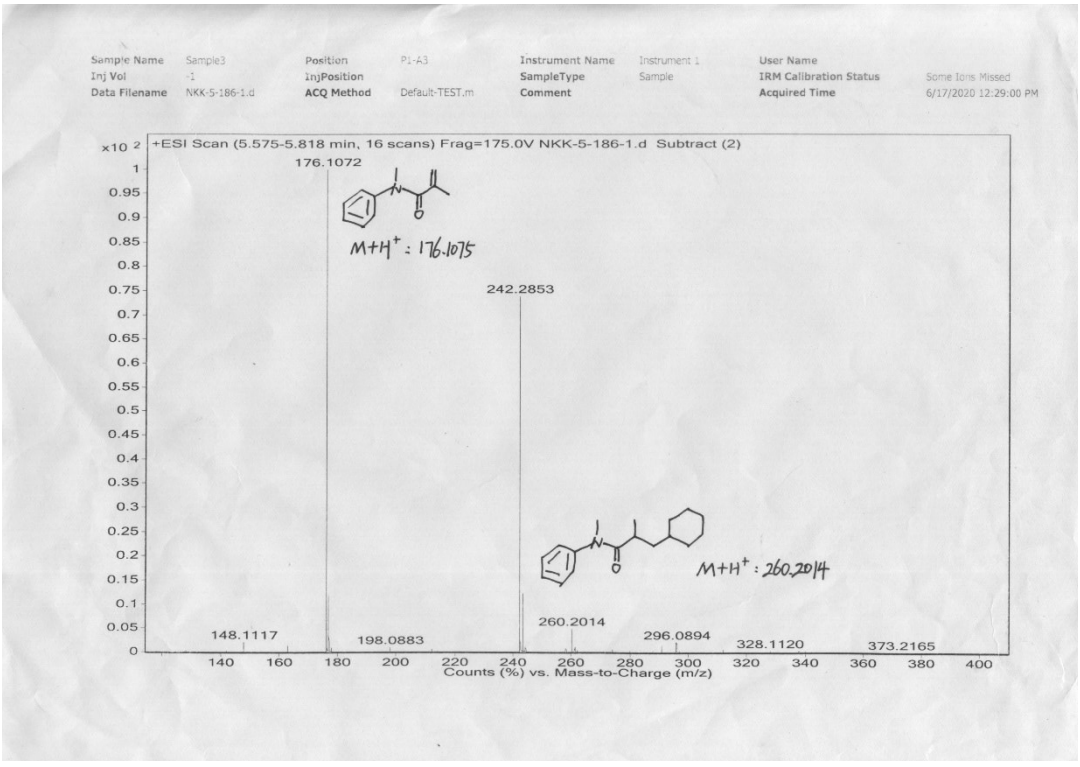
To a 10 mL oven-dried undivided bottle was added quinoxalin-2(1*H*)-one (80.0 mg, 0.5 mmol, 1.0 equiv.), *N*-hydroxyphthalimide ester (273.1 mg, 1.0 m mol, 2.0 equiv.), TEMPO (156.3 mg, 1.0 mmol, 2.0 equiv.), $^{n}Bu_4NBF_4$ (329 mg, 1.0 mmol, 2.0 equiv.) and DMA (5 mL). The reaction mixture was stirred and electrolyzed at a constant current of 5.0 mA under room temperature for 12 h. The reaction was completely suppressed. The radical trapping product 1-(cyclohexyloxy)-2,2,6,6-tetramethylpiperidine can be observed by HR-MS (positive mode ESI). A similar procedure was conducted with BHT (220.4 mg, 1.0 mmol, 2.0 equiv.). The reaction was also completely suppressed.

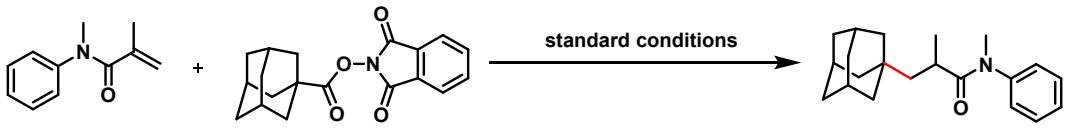


1r **2a** **3ra**

Detected by HRMS
$[M+H]^+$: 260.2014
found: 260.2014

To a 10 mL oven-dried undivided bottle was added *N*-phenylmethacrylamide **1r** (87.6 mg, 0.5 mmol, 1.0 equiv.), *N*-hydroxyphthalimide ester **2a** (273.1 mg, 1.0 mmol, 2.0 equiv.), $^{n}Bu_4NBF_4$ (329 mg, 1.0 mmol, 2.0 equiv.) and DMA (5 mL). The reaction mixture was stirred and electrolyzed at a constant current of 5.0 mA under room temperature for 12 h. The product **3ra** can be observed by HR-MS (positive mode ESI).

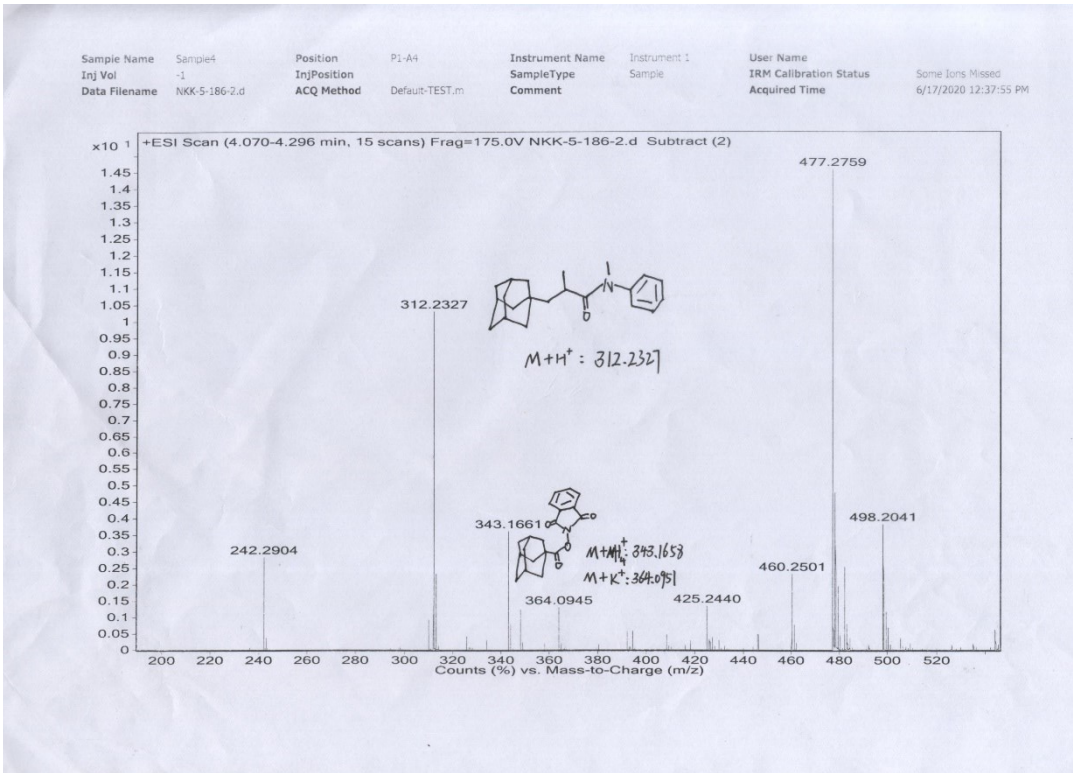




1r **2o** **3ro**

Detected by HRMS
$[M+H]^+$: 312.2327
found: 312.2327

To a 10 mL oven-dried undivided bottle was added N-methyl-N-phenyl methacrylamide **1r** (87.6 mg, 0.5 mmol, 1.0 equiv.), N-hydroxyphthalimide ester **2o** (273.1 mg, 1.0 mmol, 2.0 equiv.), $^{n}Bu_4NBF_4$ (329 mg, 1.0 mmol, 2.0 equiv.) and DMA (5 mL). The reaction mixture was stirred and electrolyzed at a constant current of 5.0 mA under room temperature for 12 h. The product **3ro** can be observed by HR-MS (positive mode ESI).



9. Cyclic voltammetry (CV) tests

The potential was calibrated versus an aqueous SCE by the addition of ferrocene as an internal standard taking $E_{(Fc/Fc+)}^{0}$ = 0.424 V vs SCE.[18] $E^{1a/1a-}$ = -1.67 V vs SCE; $E^{2a/2a-}$ = -1.23 vs SCE.

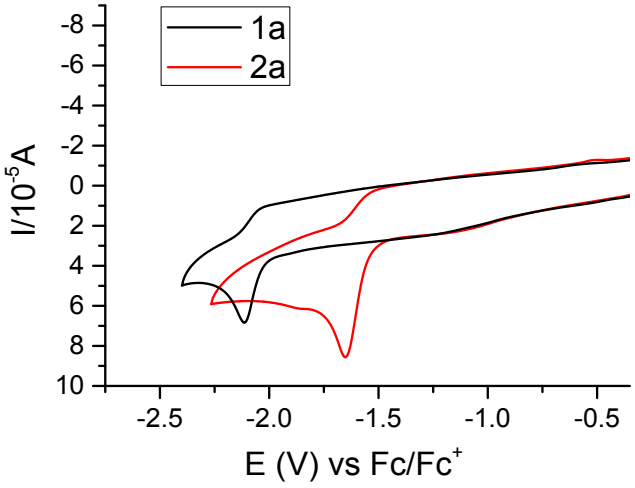
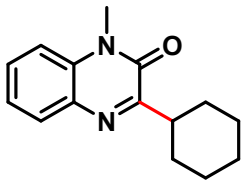


Figure 1. Cyclic voltammograms of compounds 1a and 2a in 0.1 M $^{n}Bu_4NPF_6$/MeCN at a scan rate of 100 mV/s.

10. Characterization Data for Electrolysis Products

3-cyclohexyl-1-methylquinoxalin-2(1*H*)-one(3aa)



Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, 20% EtOAc in petroleum ether) to give **3aa** as a white solid (111.3 mg, 92% yield).

M. p. = 103 – 104 °C;

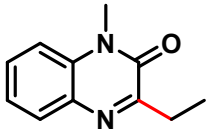
Rf = 0.50 (Petroleum ether /EtOAc = 10:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 (d, J =8.0 Hz, 1H), 7.49 (t, J = 4.0 Hz, 1H), 7.35 – 7.23 (m, 2H), 3.69 (s, 3H), 3.34 (t, J = 12.0 Hz, 1H), 1.96 (d, J = 12.0 Hz, 2H), 1.86 (d, J = 12.0 Hz, 2H), 1.76 (d, J = 8.0 Hz, 1H), 1.40 – 1.65 (m, 4H), 1.23 – 1.37 (m, 1H) ;

^{13}C NMR (101 MHz, $CDCl_3$): δ 177.4, 164.2, 154.5, 134.9, 132.8, 132.8, 129.7, 129.3, 123.4, 113.4, 40.7, 30.5, 29.0, 26.3, 26.1;

HRMS (ESI): calc'd for $C_{15}H_{18}N_2O$ ([M+H]$^+$) 243.1497, found 243.1492.

3-ethyl-1-methylquinoxalin-2(1*H*)-one(3ab)



Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, 10% EtOAc in petroleum ether) to give **3ab** as a white solid (62.0 mg, 66% yield).

M. p. = 96 – 98°C;

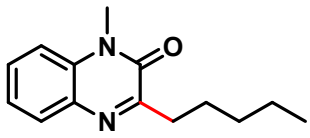
Rf = 0.30 (Petroleum ether /EtOAc = 5:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.84 (d, J = 8.0 Hz, 1H), 7.52 (t, J = 8.0 Hz, 1H), 7.39 – 7.24 (m, 2H), 3.70 (s, 3H), 2.97 (q, J = 8.0, 4.0 Hz, 2H), 1.34 (t, J = 4.0 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 161.9, 154.8, 133.1, 132.7, 129.6, 129.5, 123.5, 113.5, 28.9, 27.5, 10.8.

HRMS (ESI): calc'd for $C_{11}H_{12}N_2O$ ([M+H]$^+$) 189.1028, found 189.1022.

1-methyl-3-pentylquinoxalin-2(1*H*)-one(3ac)



Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, 20% EtOAc in petroleum ether) to give **3ac** as a white solid (80.9 mg, 70% yield).

M. p. = 75 – 76 °C;

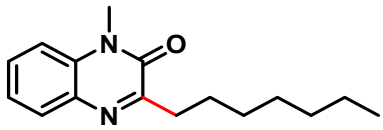
Rf = 0.50 (Petroleum ether /EtOAc = 5:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 (d, J = 8.0 Hz, 1H), 7.52 (t, J = 8.0 Hz, 1H), 7.38 – 7.25 (m, 2H), 3.70 (s, 3H), 2.94 (t, J = 8.0 Hz, 2H), 1.85 – 1.73 (m, 2H), 1.47 – 1.34 (m, 5H), 0.92 (t, J = 4.0 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 161.4, 154.9, 133.1, 132.7, 129.6, 129.5, 123.5, 113.5, 34.4, 31.8, 29.0, 26.6, 22.5, 14.1.

HRMS (ESI): calc'd for $C_{14}H_{18}N_2O$ ($[M+H]^+$) 231.1497, found 231.1493.

3-heptyl-1-methylquinoxalin-2(1*H*)-one(3ad)



Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, 10% EtOAc in petroleum ether) to give **3ad** as a yellow oil (92.0 mg, 71% yield).

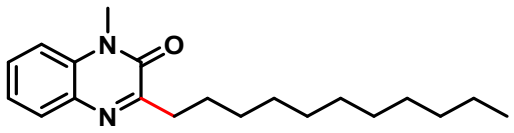
Rf = 0.50 (Petroleum ether /EtOAc = 5:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 (d, J = 8.0 Hz, 1H), 7.55 – 7.48 (m, 1H), 7.35 –7.27 (m,, 2H), 3.70 (s, 3H), 2.94 (t, J = 8.0 Hz, 2H), 1.85 – 1.75 (m, 2H), 1.48 – 1.34 (m, 4H), 1.33 – 1.28 (m, 4H), 0.88 (t, J = 8.0 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 161.4, 154.9, 133.1, 132.8, 129.6, 129.5, 123.5, 113.6, 34.5, 31.8, 29.6, 29.2, 29.1, 26.9, 22.7, 14.1.

HRMS (ESI): calc'd for $C_{16}H_{22}N_2O$ ($[M+H]^+$) 259.1810, found 259.1805.

1-methyl-3-undecylquinoxalin-2(1*H*)-one(3ae)



Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, 10% EtOAc in petroleum ether) to give **3ae** as a white solid (84.5 mg, 54% yield).

M. p. = 58 – 60 °C;

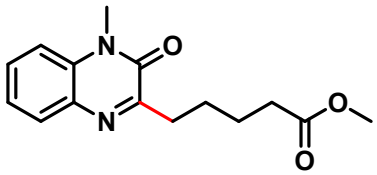
Rf = 0.40 (Petroleum ether /EtOAc = 5:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 (d, J = 8.0 Hz, 1H), 7.52 (t, J = 8.0 Hz, 1H), 7.38 – 7.23 (m, 2H), 3.70 (s, 3H), 2.94 (t, J = 8.0 Hz, 2H), 1.84 – 1.72 (m, 2H), 1.49 – 1.39 (m, 2H), 1.26 (s, 15H), 0.88 (t, J = 4.0 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 161.4, 154.9, 133.1, 132.7, 129.6, 129.5, 123.5, 113.6, 34.4, 31.9, 29.7, 29.6, 29.6, 29.5, 29.4, 29.0, 26.9, 22.7, 14.2.

HRMS (ESI): calc'd for $C_{20}H_{30}N_2O$ ([M+H]$^+$) 315.2436, found 315.2431.

1-methyl-5-(4-methyl-3-oxo-3,4-dihydroquinoxalin-2-yl)-pentanoate(3af)



Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, 20% EtOAc in petroleum ether) to give **3af** as a white solid (96.6 mg, 70% yield).

M. p. = 103 – 105 °C;

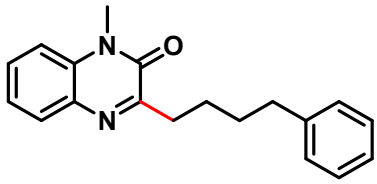
Rf = 0.60 (Petroleum ether /EtOAc = 1:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82 (d, *J* = 7.2 Hz, 1H), 7.57 – 7.49 (m, 1H), 7.38 – 7.26 (m, 3H), 3.70 (s, 3H), 3.67 (s, 3H), 2.97 (s, 3H), 2.40 (s, 3H), 1.96 – 1.74 (m, 6H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 174.1, 160.5, 154.9, 134.3, 133.1, 129.7, 129.6, 123.6, 113.6, 51.5, 33.9, 33.8, 29.1, 26.1, 24.8.

HRMS (ESI): calc'd for $C_{15}H_{18}N_2O_3$ ([M+H]$^+$) 275.1369, found 275.1389.

1-methyl-3-(4-phenylbutyl)-quinoxalin-2(1*H*)-one(3ag)



Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, 20% EtOAc in petroleum ether) to give **3ag** as a yellow oil (119.6 mg, 82% yield).

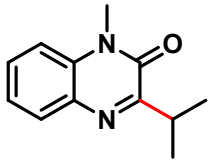
Rf = 0.50 (Petroleum ether /EtOAc = 2:1);

^{1}H NMR (400 MHz, $CDCl_3$): *δ* 7.82 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.55 – 7.48 (m, 1H), 7.36 – 7.31 (m, 1H), 7.31 – 7.23 (m, 3H), 7.22 – 7.13 (m, 3H), 3.70 (s, 3H), 3.05 – 2.94 (m, 2H), 2.74 – 2.65 (m, 2H), 1.90 – 1.73 (m, 4H).

^{13}C NMR (101 MHz, $CDCl_3$): *δ* 161.1, 154.9, 142.6, 133.1, 132.7, 129.6, 129.6, 128.5, 128.3, 125.6, 123.6, 113.6, 35.8, 34.2, 31.4, 29.1, 26.5.

HRMS (ESI): calc'd for $C_{19}H_{20}N_2O$ ([M+H]$^+$) 293.1654, found 293.1648.

2-isopropyl-1-methylquinoxalin-2(1*H*)-one(3ah)



Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, 10% EtOAc in petroleum ether) to give **3ah** as a white solid (76.9 mg, 76% yield).

M. p. = 106 – 107 °C;

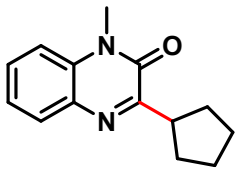
Rf = 0.80 (Petroleum ether /EtOAc = 2:1);

^{1}H NMR (400 MHz, $CDCl_3$): *δ* 7.85 (d, *J* = 8.0 Hz, 1H), 7.51 (t, *J* = 8.0 Hz, 1H), 7.35 - 7.25 (m, 2H), 3.71 (s, 3H), 3.66 - 3.58 (m, 1H), 1.33 (s, 3H), 1.31 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): *δ* 165.1, 154.6, 133.1, 132.8, 129.8, 129.5, 123.4, 113.5, 31.2, 29.1, 20.2.

HRMS (ESI): calc'd for $C_{12}H_{14}N_2O$ ([M+H]$^+$) 203.1184, found 203.1180.

2-cyclopentyl-1-methylquinoxalin-2(1*H*)-one(3ai)



Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, 20% EtOAc in petroleum ether) to give **3ai** as a white solid (79.7 mg, 70% yield).

M. p. = 89 – 91 °C;

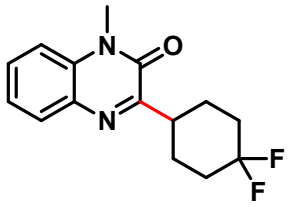
Rf = 0.30 (Petroleum ether /EtOAc = 5:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82 (d, J = 8.0 Hz, 1H), 7.49 (t, J = 8.0 Hz, 1H), 7.39 – 7.20 (m, 2H), 3.70 (s, 4H), 2.06 (s, 2H), 1.93 (s, 2H), 1.82 (s, 2H), 1.72 (s, 2H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 163.8, 155.1, 132.9, 132.7, 129.8, 129.3, 113.4, 42.7, 30.9, 29.1, 25.9.

HRMS (ESI): calc'd for $C_{14}H_{16}N_2O$ ([M+H]$^+$) 229.1341, found 229.1337.

3-(4,4-difluorocyclohexyl)-1-methylquinoxalin-2(1*H*)-one(3aj)



Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, 10% EtOAc in petroleum ether) to give **3aj** as a white solid (118.9 mg, 85% yield).

M. p. = 181 – 182 °C;

Rf = 0.25 (Petroleum ether /EtOAc = 5:1);

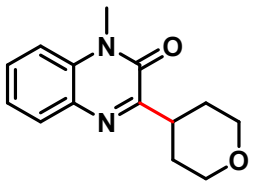
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.84 (d, J = 8.0 Hz, 1H), 7.54 (t, J = 8.0 Hz, 1H), 7.39 – 7.28 (m, 2H), 3.71 (s, 3H), 3.45 – 3.35 (m , 1H), 2.32 – 2.22 (m, 2H), 2.10 – 1.85 (m, 6H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 161.9, 154.4, 132.9, 132.7, 130.0, 129.9, 123.7, 113.6, 38.4, 33.5 (t, J = 88.0 Hz), 29.2, 26.6, 26.5.

^{19}F NMR (377 MHz, $CDCl_3$): δ -91.87 (d, J = 235.3 Hz), -101.16 (d, J = 232.2 Hz).

HRMS (ESI): calc'd for $C_{15}H_{16}F_2N_2O$ ([M+H]$^+$) 279.1309, found 279.1305.

1-methyl-3-(tetrahydro-2*H*-pyran-4-yl)-quinoxalin-2(1*H*)-one(3ak)



Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, 20% EtOAc in petroleum ether) to give **3ak** as a white solid (90.5 mg, 81% yield).

M. p. = 175 – 176 °C;

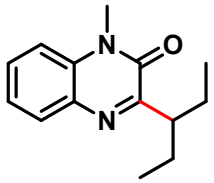
Rf = 0.40 (Petroleum ether /EtOAc = 1:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.86 (dd, J = 8.0, 1.2 Hz, 1H), 7.57 – 7.50 (m, 1H), 7.37 – 7.27 (m, 2H), 4.15 – 4.05 (m, 2H), 3.71 (s, 3H), 3.67 – 3.59 (m, 2H), 3.58 – 3.55 (m, 1H), 2.00 – 1.87 (m, 4H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 162.0, 154.5, 132.9, 132.8, 130.0, 129.8, 123.6, 113.5, 67.9, 38.1, 30.1, 29.1.

HRMS (ESI): calc'd for $C_{14}H_{16}N_2O_2$ ($[M+H]^+$) 245.1290, found 245.1286.

1-methyl-3-(pentan-3-yl)-quinoxalin-2(1*H*)-one(3al)



Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, 20% EtOAc in petroleum ether) to give **3al** as a yellow oil (86.4 mg, 75% yield).

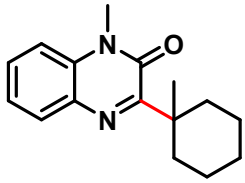
Rf = 0.70 (Petroleum ether /EtOAc = 5:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 (dd, J = 8.0, 1.4 Hz, 1H), 7.56 – 7.46 (m, 1H), 7.38 – 7.21 (m, 2H), 3.71 (s, 2H), 3.42 – 3.29 (m, 1H), 1.95 – 1.79 (m, 2H), 1.77 – 1.62 (m, 3H), 0.88 (t, J = 7.4 Hz, 5H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 163.9, 155.1, 132.9, 132.8, 129.9, 129.5, 123.4, 113.5, 44.7, 29.2, 25.8, 12.0.

HRMS (ESI): calc'd for $C_{14}H_{18}N_2O$ ($[M+H]^+$) 231.1497, found 231.1493.

1-methyl-3-(1-methylcyclohexyl)-quinoxalin-2(1*H*)-one(3am)



Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, 20% EtOAc in petroleum ether) to give **3am** as a white solid (67.2 mg, 52% yield).

M. p. = 70 – 71°C;

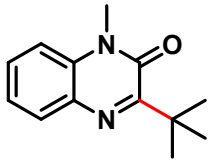
Rf = 0.50 (Petroleum ether /EtOAc = 5:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.84 (dd, J = 8.0, 1.2 Hz, 1H), 7.54 – 7.46 (m, 1H),7.33 – 7.26 (m, 2H), 3.66 (s, 3H), 2.53 – 2.37 (m, 2H), 1.70 – 1.46 (m, 8H), 1.43 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 164.8, 153.8, 133.1, 132.3, 130.1, 129.5, 123.1, 113.3, 42.9, 35.7, 28.8, 26.6, 24.5, 22.9.

HRMS (ESI): calc'd for $C_{16}H_{20=}N_2O$ ($[M+H]^+$) 257.1654, found 257.1647.

3-(*tert*-butyl)-1-methylquinoxalin-2(1*H*)-one(3an)



Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, 20% EtOAc in petroleum ether) to give **3an** as a white solid (42.7 mg, 40% yield).

M. p. = 71 – 72 °C;

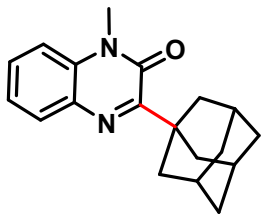
Rf = 0.40 (Petroleum ether /EtOAc = 10:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 (dd, J = 8.0, 1.2 Hz, 1H), 7.49 (ddd, J = 8.4, 8.0, 1.6 Hz, 1H), 7.33 – 7.20 (m, 2H), 3.67 (s, 3H), 1.49 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 165.3, 153.7, 133.3, 132.2, 130.1, 129.5, 123.2, 113.2, 39.5, 28.7, 27.9.

HRMS (ESI): calc'd for $C_{13}H_{16}N_2O$ ([M+H]$^+$) 217.1341, found 217.1337.

3-((1*s*, 3*s*)-adamantan-1-yl)-1-methylquinoxalin-2(1*H*)-one(3ao)



Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, 20% EtOAc in petroleum ether) to give **3ao** as a white solid (70.8 mg, 57% yield).

M. p. = 186 – 187 °C;

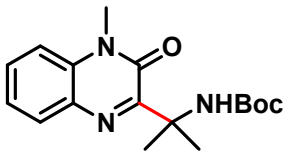
Rf = 0.50 (Petroleum ether /EtOAc = 5:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 (dd, J = 8.0, 1.2 Hz, 1H), 7.54 – 7.44 (m, 1H), 7.36 – 7.21 (m, 2H), 3.66 (s, 3H), 2.24 (d, J = 2.4 Hz, 6H), 2.11 (s, 3H), 1.86 – 1.77 (m, 6H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 164.8, 153.7, 133.0, 132.5, 132.4, 130.1, 129.5, 123.2, 113.3, 42.0, 38.8, 37.0, 28.7, 28.6.

HRMS (ESI): calc'd for $C_{19}H_{22}N_2O$ ([M+H]$^+$) 295.1810, found 295.1806.

***tert*-butyl(*R*)-(2-methyl-1-(4-methyl-3-oxo-3,4-dihydroquinoxalin-2-yl)-propyl)-carbamate(3ap)**



Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, 20% EtOAc in petroleum ether) to give **3ap** as a white solid (118.8 mg, 72% yield).

M. p. = 106 – 107 °C;

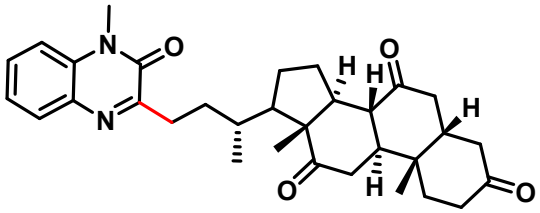
Rf = 0.80 (Petroleum ether /EtOAc = 1:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.87 (dd, J = 8.0, 1.1 Hz, 1H), 7.59 – 7.50 (m, 1H), 7.38 – 7.27 (m, 2H), 6.17 (s, 1H), 3.68 (s, 3H), 1.83 (s, 6H), 1.43 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 160.3, 154.9, 153.0, 133.6, 131.5, 130.3, 130.9, 123.5, 113.5, 78.9, 57.1, 28.9, 28.4, 25.1.

HRMS (ESI): calc'd for $C_{17}H_{24}N_3O_3$ ([M+H]$^+$) 318.1818, found 318.1806.

(5*S*,8*R*,9*S*,10*S*,13*R*,14*S*)-10,13-dimethyl-17-((*R*)-4-(4-methyl-3-oxo-3,4-dihydroquinoxalin-2-yl) butan-2-yl) dodecahydro-3*H*-cyclopenta[a]phenanthrene-3,7,12(2*H*, 4*H*)-trione(3aq)



Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, 30% EtOAc in petroleum ether) to give **3aq** as a white solid (110.3 mg, 43% yield).

M. p. >200 °C;

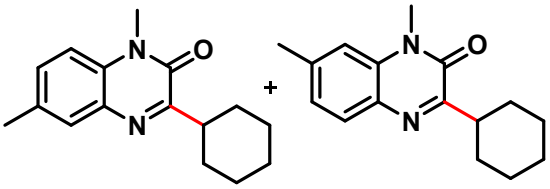
Rf = 0.30 (Petroleum ether /EtOAc = 1:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 (d, J = 8.0 Hz, 1H), 7.52 (t, J = 7.2 Hz, 1H), 7.36 –7.29 (m, 2H), 3.70 (s, 3H), 3.09 – 2.99 (m, 1H), 2.94 – 2.87 (m, 4H), 2.35 – 2.22 (m, 6H), 2.21 – 2.07 (m, 6H), 2.05 – 1.96 (m, 4H), 1.89 – 1.85 (m, 1H), 1.67 – 1.55 (m, 2H), 1.41 (s, 3H), 1.10 (s, 3H), 0.99 (d, J = 6.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 212.0, 209.2, 208.8, 161.5, 154.8, 133.1, 132.7, 129.5, 129.5, 123.5, 113.6, 56.9, 51.7, 49.0, 46.8, 45.8, 45.5, 45.0, 42.8, 38.6, 36.7, 36.2, 36.0, 35.2, 32.3, 31.7, 29.0, 27.7, 25.2, 23.4, 21.9, 18.9, 11.9.

HRMS (ESI): calc'd for $C_{32}H_{40}N_2O_4$ ($[M+H]^+$) 517.3066, found 517.3059.

3-cyclohexyl-1,6-dimethylquinoxalin-2(1H)-one(3ba) and 3-cyclohexyl-1,7-dimethylquinoxalin-2(1H)-one (3ba')



R^3=6-Me 3ba **R^3=7-Me 3ba'**

Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, 20% EtOAc in petroleum ether) to give **3ba** as a white solid (89.3 mg, 70% yield).

M. p. = 98 – 100 °C;

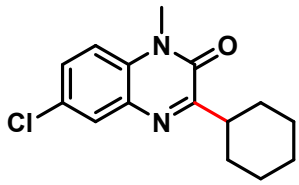
Rf = 0.40 (Petroleum ether /EtOAc = 5:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.70 (d, J = 8.1 Hz, 1H), 7.65 (s, 1H), 7.31 (d, J = 8.2 Hz, 1H), 7.27 (s, 1H), 7.15 (dd, J = 11.6, 8.8 Hz, 2H), 7.06 (s, 1H), 3.67 (s, 6H), 3.38 – 3.25 (m, 2H), 2.50 (s, 3H), 2.44 (s, 3H), 1.95 (d, J = 11.9 Hz, 4H), 1.86 (d, J = 12.4 Hz, 4H), 1.76 (d, J = 12.5 Hz, 2H), 1.62 – 1.37 (m, 8H), 1.37 – 1.24 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 164.2, 163.0, 154.7, 154.5, 140.0, 133.2, 132.8, 132.7, 131.1, 130.6, 130.5, 129.7, 129.4, 124.6, 113.6, 113.2, 40.7, 30.6, 30.5, 29.0, 29.0, 26.3, 26.3, 26.2, 22.0, 20.6.

HRMS (ESI): calc'd for $C_{16}H_{20}N_2O$ ($[M+H]^+$) 257.1654, found 257.1649.

6-chloro-3-cyclohexyl-1-methylquinoxalin-2(1*H*)-one(3ca)



Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, 20% EtOAc in petroleum ether) to give **3ca** as a white solid (85.8 mg, 64% yield).

M. p. = 125 – 126 °C;

Rf = 0.50 (Petroleum ether /EtOAc = 5:1);

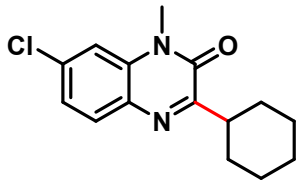
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.74 (d, J = 8.8 Hz, 1H), 7.31 – 7.24 (m, 2H), 3.66 (s, 3H), 3.31 (t, J = 10.8 Hz, 1H), 1.94 (d, J = 11.6 Hz, 2H), 1.86 (d, J = 11.6 Hz, 2H), 1.81 – 1.68 (m, 1H), 1.62 – 1.40 (m, 4H), 1.36 – 1.24 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 164.4, 154.3, 135.2, 133.7, 131.4, 130.8, 123.8, 113.5, 40.8, 30.5,

29.2, 26.3, 26.1.

HRMS (ESI): calc'd for $C_{15}H_{17}ClN_2O$ ($[M+H]^+$) 277.1108, found 277.1103.

7-chloro-3-cyclohexyl-1-methylquinoxalin-2(1*H*)-one(3da)



Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, 20% EtOAc in petroleum ether) to give **3da** as a white solid (61.1 mg, 46% yield).

M. p. = 180 – 182 °C;

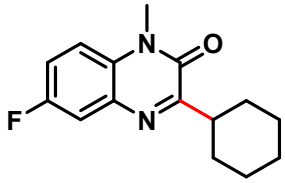
Rf = 0.80 (Petroleum ether /EtOAc = 5:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 (s, 1H), 7.45 (d, J = 8.4 Hz, 1H), 7.21 (d, J = 8.8 Hz, 1H), 3.68 (s, 3H), 3.32 (t, J = 10.4 Hz, 1H), 1.94 (d, J = 11.2 Hz, 2H), 1.86 (d, J = 11.2 Hz, 2H), 1.80-1.72 (m, 1H), 1.60-1.40 (m, 4H), 1.34-1.23 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 165.7, 154.2, 133.4, 131.6, 129.3, 129.2, 128.7, 114.6, 40.8, 30.5, 29.3, 26.3, 26.1.

HRMS (ESI): calc'd for $C_{15}H_{18}N_2O$ ($[M+H]^+$) 277.1108, found 277.1103.

3-cyclohexyl-6-fluoro-1-methylquinoxalin-2(1*H*)-one(3ea)



Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, 20% EtOAc in petroleum ether) to give **3ea** as a white solid (68.4 mg, 53% yield).

M. p. = 112 – 113 °C;

Rf = 0.40 (Petroleum ether /EtOAc = 5:1);

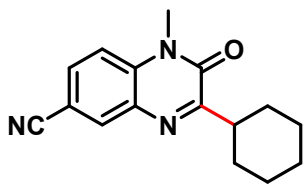
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.56 – 7.49 (m, 1H), 7.29 – 7.20 (m, 2H), 3.69 (s, 3H), 3.38 – 3.28 (m, 1H), 1.94 (d, J = 12.3 Hz, 2H), 1.89 – 1.83 (m, 2H), 1.77 (d, J = 13.1 Hz, 1H), 1.60 – 1.39 (m, 5H), 1.35 – 1.23 (m, 1H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 165.8, 159.8, 157.4, 154.2, 133.5 (d, J = 11.3 Hz), 129.5 (d, J = 2.1 Hz), 117.0 (d, J = 23.8 Hz), 115.2 (d, J = 22.4 Hz), 114.5 (d, J = 8.8 Hz), 40.8, 30.5, 29.3, 26.2, 26.1.

^{19}F NMR (377 MHz, $CDCl_3$): δ -117.45 – -121.07 (m).

HRMS (ESI): calc'd for $C_{15}H_{17}FN_2O$ ($[M+H]^+$) 261.1403, found 261.1398.

1-cyclohexyl-1-methyl-2-oxo-1,2-dihydroquinoxaline-6-carbonitrile(3fa)



Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, 20% EtOAc in petroleum ether) to give **3fa** as a white solid (100.0 mg, 75% yield).

M. p. = 140 – 141 °C;

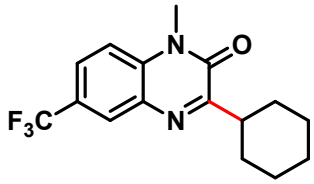
Rf = 0.40 (Petroleum ether /EtOAc = 5:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.91 (d, J = 7.6 Hz, 1H), 7.58 (s, 2H), 3.70 (s, 3H), 3.39 – 3.30 (m, 1H), 1.95 (d, J = 12.0 Hz, 2H), 1.87 (d, J = 12.0 Hz, 2H), 1.80 – 1.71 (m, 1H), 1.61 – 1.39 (m, 4H), 1.35 – 1.24 (m, 1H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 167.9, 154.0, 135.1, 133.3, 130.7, 126.4, 118.3, 117.7, 112.4, 41.1, 30.4, 29.3, 26.2, 26.0.

HRMS (ESI): calc'd for $C_{16}H_{18}N_3O$ ($[M+H]^+$) 268.1450, found 268.1445.

1-cyclohexyl-1-methyl-6-(trifluoromethyl)-quinoxalin-2(1*H*)-one(3ga)



Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, 20% EtOAc in petroleum ether) to give **3ga** as a white solid (85.7 mg, 55% yield).

M. p. = 98 – 101 °C;

Rf = 0.50 (Petroleum ether /EtOAc = 5:1);

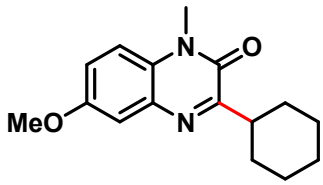
^{1}H NMR (400 MHz, CDCl3): δ 8.13 (s, 1H), 7.72 (d, J = 8.4 Hz, 1H), 7.38 (d, J = 8.4 Hz, 1H), 3.72 (s, 3H), 3.34 (t, J = 10.6 Hz, 1H), 1.95 (d, J = 11.4 Hz, 2H), 1.88 (d, J = 11.4 Hz, 2H), 1.78 (d, J = 12.0 Hz, 1H), 1.62 – 1.42 (m, 4H), 1.38 – 1.23 (m, 1H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 256.0, 243.5, 166.0, 154.3, 135.2, 132.2, 127.2 (q, J = 3.4 Hz), 125.7 (q, J = 7.0, 3.6 Hz), 125.5 (t, J = 31.8 Hz), 122.5, 114.1, 40.8, 30.5, 29.3, 26.2, 26.1.

^{19}F NMR (377 MHz, $CDCl_3$): δ -61.89 (s).

HRMS (ESI): calc'd for $C_{16}H_{17}F_3N_2O$ ($[M+H]^+$) 311.1371, found 311.1368.

1-cyclohexyl-6-methoxy-1-methylquinoxalin-2(1*H*)-one(3ha)



Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, 10% EtOAc in petroleum ether) to give **3ha** as a white solid (73.2 mg, 54% yield).

M. p. = 105 – 106 °C;

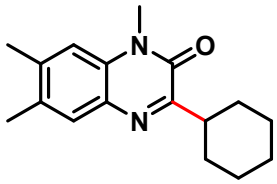
Rf = 0.40 (Petroleum ether /EtOAc = 5:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.74 (d, J = 8.8 Hz, 1H), 7.27 (s, 1H), 6.90 (dd, J = 8.8, 2.4 Hz, 1H), 6.69 (d, J = 2.4 Hz, 1H), 3.91 (s, 3H), 3.65 (s, 3H), 3.34 – 3.23 (m, 1H), 1.94 (d, J = 12.0 Hz, 3H), 1.86 (d, J = 12.0 Hz, 2H), 1.79 – 1.66 (m, 2H), 1.61 – 1.41 (m, 4H), 1.36 – 1.23 (m, 1H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 160.9, 160.6, 154.9, 134.3, 131.0, 127.8, 110.2, 98.0, 55.8, 40.6, 30.60, 29.11, 26.40, 26.22.

HRMS (ESI): calc'd for $C_{16}H_{20}N_2O_2$ ($[M+H]^+$) 273.1603, found 273.1595.

1-cyclohexyl-1, 6, 7-trimethylquinoxalin-2(1*H*)-one(3ia)



Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, 20% EtOAc in petroleum ether) to give **3ia** as a white solid (102.2 mg, 76% yield).

M. p. = 111 – 112 °C;

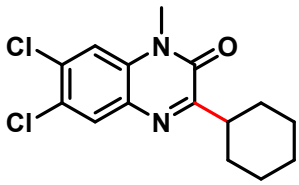
Rf = 0.40 (Petroleum ether /EtOAc = 5:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.59 (s, 1H), 7.03 (s, 1H), 3.66 (s, 3H), 3.36 – 3.23 (m, 1H), 2.40 (s, 3H), 2.33 (s, 3H), 1.94 (d, J = 12.8 Hz, 2H), 1.86 (d, J = 9.6 Hz, 2H), 1.76 (d, J = 12.8 Hz, 1H), 1.62 – 1.40 (m, 4H), 1.36 – 1.21 (m, 1H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 163.0, 154.6, 139.0, 132.2, 131.3, 130.8, 129.9, 114.1, 40.6, 30.6, 29.0, 26.4, 26.2, 20.5, 19.1.

HRMS (ESI): calc'd for $C_{17}H_{22}N_2O$ ($[M+H]^+$) 271.1801, found 271.1803.

6,7-dichloro-3-cyclohexyl-1-methylquinoxalin-2(1*H*)-one(3ja)



Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, 20% EtOAc in petroleum ether) to give **3ja** as a white solid (78.0 mg, 50% yield).

M. p. = 123 – 124 °C;

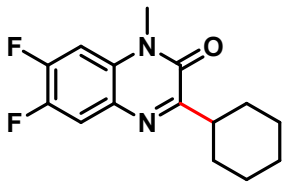
Rf = 0.70 (Petroleum ether /EtOAc = 5:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.91 (s, 1H), 7.35 (s, 1H), 3.65 (s, 3H), 3.30 (t, J = 10.7 Hz, 1H), 1.93 (d, J = 11.0 Hz, 2H), 1.86 (d, J = 11.9 Hz, 2H), 1.80 – 1.67 (m, 2H), 1.58 – 1.38 (m, 5H), 1.35 – 1.22 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 165.9, 153.9, 133.3, 132.3, 132.0, 130.6, 127.1, 115.0, 40.9, 30.5, 29.3, 26.2, 26.1.

HRMS (ESI): calc'd for $C_{15}H_{16}Cl_2N_2O$ ($[M+H]^+$) 311.0718, found 311.0712.

1-cyclohexyl-6, 7-difluoro-1-methylquinoxalin-2(1*H*)-one(3ka)



Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, 10% EtOAc in petroleum ether) to give **3ka** as a white solid (59.0 mg, 43% yield).

M. p. = 176 – 178 °C;

Rf = 0.65 (Petroleum ether /EtOAc = 5:1);

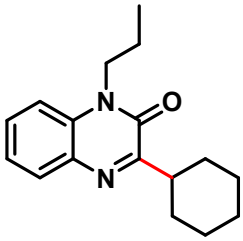
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.64 (q, J = 10.4, 8.3 Hz, 1H), 7.08 (q, J = 11.4, 7.1 Hz, 1H), 3.65 (s, 3H), 3.34 – 3.25 (m, 1H), 1.93 (d, J = 11.8 Hz, 2H), 1.86 (d, J = 12.4 Hz, 2H), 1.76 (d, J = 12.8 Hz, 1H), 1.58 – 1.39 (m, 5H), 1.37 – 1.22 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 164.8 (d, J = 3.4 Hz), 154.1, 150.9 (dd, J = 252.0, 14.2 Hz), 146.5 (dd, J = 246.3, 13.9 Hz), 130.0 (dd, J = 8.7, 1.8 Hz), 129.2 (dd, J = 9.4, 2.8 Hz), 117.4 (dd, J = 17.9, 2.1 Hz), 102.1 (d, J = 23.0 Hz), 40.8, 30.5, 29.6, 26.2, 26.1.

^{19}F NMR (377 MHz, $CDCl_3$): δ -130.90 – -136.79 (m), -141.91 – -144.83 (m).

HRMS (ESI): calc'd for $C_{15}H_{16}F_2N_2O$ ($[M+H]^+$) 279.1309, found 279.1303.

3-cyclohexyl-1-propylquinoxalin-2(1*H*)-one(3la)



Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, 20% EtOAc in petroleum ether) to give **3la** as a white solid (89.9 mg, 67% yield).

M. p. = 103 – 104 °C;

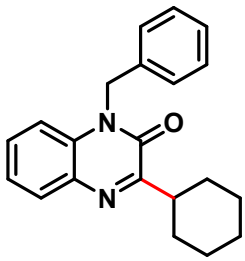
Rf = 0.80 (Petroleum ether /EtOAc = 5:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.84 (dd, J = 8.0, 1.2 Hz, 1H), 7.53 – 7.43 (m, 1H), 7.29 (dd, J = 13.2, 7.5 Hz, 2H), 4.25 – 4.14 (m, 2H), 3.39 – 3.29 (m, 1H), 1.96 (d, J = 11.6 Hz, 2H), 1.90 – 1.84 (m, 2H), 1.83 – 1.74 (m, 4H), 1.63 – 1.41 (m, 4H), 1.36 – 1.24 (m, 1H), 1.05 (t, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 164.3, 154.3, 133.2, 132.0, 130.0, 129.3, 123.2, 113.5, 43.8, 40.7, 30.5, 26.4, 26.2, 20.7, 11.4.

HRMS (ESI): calc'd for $C_{15}H_{18}N_2O$ ($[M+H]^+$) 271.1801, found 271.1800.

1-benzyl-3-cyclohexylquinoxalin-2(1*H*)-one(3ma)



Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, 10% EtOAc in petroleum ether) to give **3ma** as a white solid (89.0 mg, 56% yield)

M. p. = 133 – 134 °C;

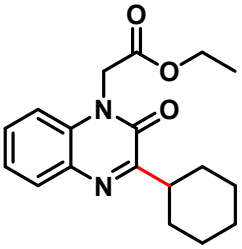
Rf = 0.40 (Petroleum ether /EtOAc = 5:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.84 (d, J = 6.4 Hz, 1H), 7.36 (d, J = 6.4 Hz, 1H), 7.34 – 7.17 (m, 7H), 5.49 (s, 2H), 3.49 – 3.31 (m, 1H), 2.01 (d, J = 10.4 Hz, 2H), 1.88 (d, J = 9.6 Hz, 2H), 1.82 – 1.70 (m, 1H), 1.67 – 1.40 (m, 5H), 1.40 – 1.21 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 164.4, 154.6, 135.5, 133.2, 132.2, 129.9, 129.4, 128.9, 127.6, 126.9, 123.5, 114.3, 45.9, 40.8, 30.6, 26.4, 26.2.

HRMS (ESI): calc'd for $C_{21}H_{22}N_2O$ ($[M+H]^+$) 319.1810, found 319.1803.

ethyl 2-(3-cyclohexyl-2-oxoquinoxalin-1(2*H*)-yl)-acetate(3na)



Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, 20% EtOAc in petroleum ether) to give **3na** as a white solid (135.2 mg, 86% yield).

M. p. = 84 – 85 °C;

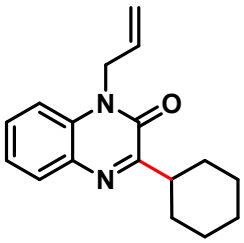
Rf = 0.40 (Petroleum ether /EtOAc = 5:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 (d, J = 8.2 Hz, 1H), 7.46 (t, J = 7.6 Hz, 1H), 7.32 (t, J = 7.6 Hz, 1H), 7.05 (d, J = 8.2 Hz, 1H), 5.01 (s, 2H), 4.25 (q, J = 13.6, 6.8 Hz, 2H), 3.32 (t, J = 11.2 Hz, 1H), 1.97 (d, J = 11.8 Hz, 2H), 1.87 (d, J = 12.0 Hz, 2H), 1.77 (d, J = 15.9 Hz, 1H), 1.64 – 1.38 (m, 5H), 1.37 – 1.30 (m, 1H), 1.27 (t, J = 6.8 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 167.3, 164.0, 154.1, 133.0, 132.0, 130.1, 129.6, 123.7, 112.9, 62.0, 43.6, 40.8, 30.5, 26.3, 26.2, 14.1.

HRMS (ESI): calc'd for $C_{18}H_{22}N_2O_3$ ($[M+H]^+$) 315.1709, found 315.1704.

1-allyl-3-cyclohexylquinoxalin-2(1*H*)-one(3oa)



Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, 20% EtOAc in petroleum ether) to give **3oa** as a white solid (87.3 mg, 65% yield).

M. p. = 91 – 92 °C;

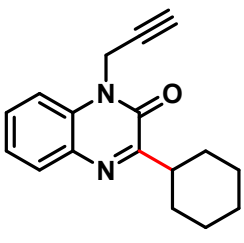
Rf = 0.50 (Petroleum ether /EtOAc = 5:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.84 (d, J = 7.6 Hz, 1H), 7.46 (t, J = 7.6 Hz, 1H), 7.35 – 7.21 (m, 2H), 6.00 – 5.81 (m, 1H), 5.26 (d, J = 10.4 Hz, 1H), 5.17 (d, J = 17.2 Hz, 1H), 4.90 (d, J = 2.8 Hz, 2H), 3.35 (t, J = 11.2 Hz, 1H), 1.97 (d, J = 11.2 Hz, 2H), 1.87 (d, J = 11.2 Hz, 2H), 1.80 – 1.70 (m, 1H), 1.65 – 1.40 (m, 5H), 1.38 – 1.25 (m, 1H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 164.3, 154.0, 133.0, 132.0, 130.8, 129.8, 129.3, 123.4, 118.0, 114.0, 44.5, 40.7, 30.5, 26.3, 26.1.

HRMS (ESI): calc'd for $C_{15}H_{18}N_2O$ ($[M+H]^+$) 269.1654, found 269.1650.

2-cyclohexyl-1-(prop-2-yn-1-yl)-quinoxalin-2(1*H*)-one(3pa)



Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, 10% EtOAc in petroleum ether) to give **3pa** as a white solid (84.0 mg, 63% yield).

M. p. = 148 – 149 °C;

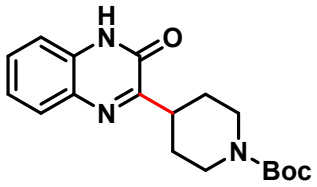
Rf = 0.70 (Petroleum ether /EtOAc = 5:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 (d, J = 7.9 Hz, 1H), 7.53 (t, J = 7.7 Hz, 1H), 7.43 (d, J = 8.1 Hz, 1H), 7.34 (t, J = 7.6 Hz, 1H), 5.05 (d, J = 2.4 Hz, 2H), 3.39 – 3.26 (m, 1H), 2.29 (t, J = 2.4 Hz, 1H), 1.96 (d, J = 11.8 Hz, 2H), 1.86 (d, J = 12.7 Hz, 2H), 1.76 (d, J = 12.7 Hz, 1H), 1.62 – 1.40 (m, 4H), 1.37 – 1.25 (m, 1H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 164.2, 153.5, 133.1, 131.3, 129.9, 129.5, 123.8, 114.0, 73.1, 40.8, 31.5, 30.5, 26.3, 26.1.

HRMS (ESI): calc'd for $C_{17}H_{18}N_2O$ ([M+H]$^+$) 267.1497, found 267.1491.

***tert*-butyl 4-(3-oxo-3,4-dihydroquinoxalin-2-yl)-piperidine-1-carboxylate(3qa)**



Electrolysis was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, 30% EtOAc in petroleum ether) to give **3qa** as a white solid (136.0 mg, 83% yield).

M. p. >200 °C;

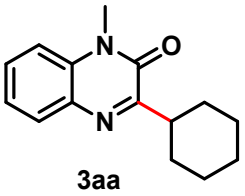
Rf = 0.30 (Petroleum ether /EtOAc = 1:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 11.68 (s, 1H), 7.83 (d, J = 7.9 Hz, 1H), 7.50 (t, J = 7.4 Hz, 1H), 7.38 – 7.29 (m, 2H), 4.28 (s, 2H), 3.49 (t, J = 11.5 Hz, 1H), 2.95 (s, 2H), 2.03 – 1.92 (m, 2H), 1.89 – 1.76 (m, 2H), 1.49 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 162.8, 155.9, 154.9, 132.9, 130.7, 129.9, 129.1, 124.2, 115.5, 79.5, 38.5, 29.4, 28.6, 28.5.

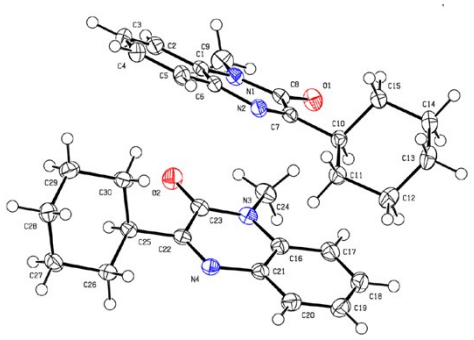
HRMS (ESI): calc'd for $C_{18}H_{23}N_3O_3$ ([M+Na]$^+$) 352.1637, found 352.1630.

11. X-ray Crystallography



3aa

≡



CCDC 2009783

Table 1 Crystal data and structure refinement for r20200612d.

Identification code	r20200612d

Empirical formula	$C_{15}H_{18}N_2O$
Formula weight	242.31
Temperature/K	113.15
Crystal system	triclinic
Space group	P-1
a/Å	10.3988(9)
b/Å	11.9760(9)
c/Å	12.1316(8)
α/°	86.151(6)
β/°	70.363(7)
γ/°	65.045(8)
Volume/Å^3	1285.17(19)
Z	4
ρ_{calc}g/cm^3	1.252
μ/mm^{-1}	0.080
F(000)	520.0
Crystal size/mm^3	0.2 × 0.18 × 0.16
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	3.578 to 52.734
Index ranges	-12 ≤ h ≤ 12, -14 ≤ k ≤ 14, -15 ≤ l ≤ 15
Reflections collected	13407
Independent reflections	5209 [R_{int} = 0.0265, R_{sigma} = 0.0300]
Data/restraints/parameters	5209/0/327
Goodness-of-fit on F^2	1.027
Final R indexes [I>=2σ (I)]	R_1 = 0.0433, wR_2 = 0.1099
Final R indexes [all data]	R_1 = 0.0521, wR_2 = 0.1162
Largest diff. peak/hole / e Å^{-3}	0.63/-0.23

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for r20200612d. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
O1	8948.8(10)	1484.4(9)	5996.9(8)	29.3(2)
N1	6988.4(12)	3406.3(10)	6499.7(9)	23.1(2)
N2	5896.1(12)	2620.5(10)	8704.5(9)	22.2(2)
C1	5732.8(14)	4255.7(12)	7366.8(12)	22.6(3)
C2	4985.0(16)	5490.4(13)	7174.2(13)	29.5(3)
C3	3768.0(16)	6292.2(13)	8077.1(15)	34.2(3)
C4	3271.6(16)	5897.3(13)	9179.3(14)	32.3(3)
C5	3994.0(15)	4678.1(13)	9370.1(12)	27.7(3)
C6	5223.3(14)	3845.3(12)	8472.9(11)	21.7(3)

C7	7067.9(14)	1851.3(12)	7891.1(11)	20.2(3)
C8	7762.9(14)	2213.1(12)	6721.2(11)	21.6(3)
C9	7555.1(17)	3803.5(14)	5335.3(12)	33.0(3)
C10	7850.9(14)	529.5(12)	8126.9(11)	21.9(3)
C11	6794.7(15)	87.4(13)	9062.1(13)	28.1(3)
C12	7651.2(17)	-1245.2(13)	9303.1(14)	33.8(3)
C13	8984.2(16)	-1393.1(13)	9653.6(12)	30.5(3)
C14	10032.0(15)	-951.0(13)	8726.4(12)	29.1(3)
C15	9186.1(14)	382.8(12)	8495.0(12)	24.2(3)
O2	4849.5(11)	3002.3(9)	5089.2(8)	30.8(2)
N3	6153.9(12)	967.6(10)	5294.8(9)	22.3(2)
N4	3511.1(12)	1067.2(10)	7034.9(9)	21.7(2)
C16	6198.0(14)	-67.8(12)	5908.6(11)	22.1(3)
C17	7527.3(15)	-1156.2(13)	5686.6(12)	27.0(3)
C18	7501.5(17)	-2152.3(13)	6325.4(13)	31.9(3)
C19	6180.7(17)	-2092.0(13)	7186.7(13)	31.9(3)
C20	4875.1(16)	-1021.9(13)	7405.1(12)	27.2(3)
C21	4859.9(14)	5.2(12)	6775.5(11)	21.9(3)
C22	3508.3(14)	2030.9(12)	6486.9(11)	20.4(3)
C23	4874.0(14)	2062.5(12)	5573.3(11)	22.1(3)
C24	7483.7(15)	885.7(14)	4294.7(12)	28.3(3)
C25	2092.1(14)	3215.7(12)	6762.1(11)	21.2(3)
C26	651.0(14)	3030.4(12)	7337.9(11)	23.8(3)
C27	-745.7(15)	4266.6(13)	7609.2(12)	28.0(3)
C28	-669.2(15)	5180.3(13)	8377.5(13)	29.3(3)
C29	769.0(15)	5358.5(13)	7816.1(14)	31.7(3)
C30	2164.1(15)	4125.7(12)	7550.6(12)	27.5(3)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for r20200612d. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1	23.8(5)	34.6(6)	23.1(5)	-0.8(4)	-5.5(4)	-8.1(4)
N1	22.5(5)	28.3(6)	22.4(5)	8.2(5)	-9.0(4)	-14.2(5)
N2	19.9(5)	22.9(6)	24.0(5)	3.7(4)	-8.8(4)	-8.3(4)
C1	18.7(6)	24.4(7)	29.9(7)	5.9(5)	-11.6(5)	-11.8(5)
C2	27.4(7)	27.6(7)	41.1(8)	14.3(6)	-16.9(6)	-16.1(6)
C3	27.6(7)	21.1(7)	57.5(10)	9.8(7)	-20.8(7)	-9.7(6)
C4	22.5(7)	24.8(7)	45.8(9)	-1.8(6)	-11.8(6)	-5.8(6)
C5	20.3(6)	27.8(7)	32.0(7)	2.3(6)	-8.3(6)	-8.0(6)
C6	18.4(6)	21.7(6)	27.2(7)	4.8(5)	-10.7(5)	-8.7(5)

C7	19.5(6)	22.6(6)	22.0(6)	3.0(5)	-10.1(5)	-9.9(5)
C8	19.8(6)	27.2(7)	21.8(6)	2.4(5)	-9.8(5)	-11.7(5)
C9	36.7(8)	40.4(8)	25.9(7)	13.9(6)	-10.1(6)	-22.0(7)
C10	22.9(6)	20.6(6)	21.5(6)	1.5(5)	-10.2(5)	-6.7(5)
C11	26.1(7)	25.2(7)	36.0(7)	9.0(6)	-13.7(6)	-12.1(6)
C12	38.0(8)	24.9(7)	39.8(8)	9.7(6)	-15.3(7)	-14.0(6)
C13	34.0(7)	23.3(7)	25.9(7)	4.0(5)	-12.1(6)	-3.4(6)
C14	25.0(7)	28.1(7)	27.8(7)	0.4(6)	-12.0(6)	-2.8(6)
C15	21.6(6)	24.9(7)	24.8(6)	1.5(5)	-9.5(5)	-7.2(5)
O2	28.8(5)	30.9(5)	30.4(5)	11.0(4)	-6.1(4)	-14.6(4)
N3	20.6(5)	27.7(6)	18.1(5)	0.2(4)	-3.7(4)	-11.7(5)
N4	22.7(5)	23.5(6)	20.3(5)	0.9(4)	-6.9(4)	-11.3(5)
C16	25.1(6)	24.5(7)	19.7(6)	-1.1(5)	-9.9(5)	-11.1(5)
C17	24.5(7)	29.6(7)	25.8(7)	-4.6(6)	-9.2(5)	-9.0(6)
C18	30.8(7)	24.7(7)	38.9(8)	-2.6(6)	-17.0(6)	-5.9(6)
C19	39.4(8)	24.4(7)	38.8(8)	7.2(6)	-20.9(7)	-14.6(6)
C20	30.8(7)	27.9(7)	27.6(7)	4.8(6)	-11.7(6)	-15.7(6)
C21	23.9(6)	23.4(7)	21.0(6)	-0.1(5)	-9.4(5)	-10.8(5)
C22	21.8(6)	24.5(7)	17.8(6)	0.2(5)	-7.1(5)	-11.9(5)
C23	22.3(6)	27.2(7)	18.7(6)	1.7(5)	-7.2(5)	-11.9(5)
C24	21.2(6)	38.3(8)	22.7(6)	-0.9(6)	-1.4(5)	-14.5(6)
C25	20.2(6)	23.1(6)	21.1(6)	2.4(5)	-6.7(5)	-10.2(5)
C26	22.6(6)	26.5(7)	24.2(6)	-0.8(5)	-6.5(5)	-12.8(6)
C27	20.2(6)	32.3(7)	31.8(7)	0.7(6)	-8.5(5)	-11.4(6)
C28	21.7(7)	26.1(7)	34.5(7)	-2.1(6)	-5.5(6)	-7.6(6)
C29	26.7(7)	24.3(7)	42.3(8)	-4.3(6)	-7.0(6)	-11.8(6)
C30	22.6(7)	27.5(7)	34.2(7)	-3.3(6)	-8.4(6)	-12.3(6)

Table 4 Bond Lengths for r20200612d.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
O1	C8	1.2333(16)	O2	C23	1.2286(16)
N1	C1	1.3949(17)	N3	C16	1.3947(17)
N1	C8	1.3732(17)	N3	C23	1.3746(17)
N1	C9	1.4683(16)	N3	C24	1.4724(16)
N2	C6	1.3896(16)	N4	C21	1.3913(16)
N2	C7	1.2938(16)	N4	C22	1.2922(16)
C1	C2	1.3968(19)	C16	C17	1.3996(19)
C1	C6	1.4058(18)	C16	C21	1.4057(18)

C2	C3	1.381(2)	C17	C18	1.383(2)
C3	C4	1.391(2)	C18	C19	1.395(2)
C4	C5	1.377(2)	C19	C20	1.375(2)
C5	C6	1.3958(18)	C20	C21	1.4008(18)
C7	C8	1.4888(17)	C22	C23	1.4928(17)
C7	C10	1.5030(17)	C22	C25	1.5045(17)
C10	C11	1.5317(18)	C25	C26	1.5294(17)
C10	C15	1.5357(18)	C25	C30	1.5380(18)
C11	C12	1.5293(19)	C26	C27	1.5295(18)
C12	C13	1.521(2)	C27	C28	1.5273(19)
C13	C14	1.522(2)	C28	C29	1.5202(19)
C14	C15	1.5246(18)	C29	C30	1.5266(19)

Table 5 Bond Angles for r20200612d.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C1	N1	C9	119.72(11)	C16	N3	C24	119.79(11)
C8	N1	C1	121.66(11)	C23	N3	C16	121.59(11)
C8	N1	C9	118.54(11)	C23	N3	C24	118.58(11)
C7	N2	C6	118.70(11)	C22	N4	C21	118.54(11)
N1	C1	C2	122.31(12)	N3	C16	C17	121.84(12)
N1	C1	C6	118.23(11)	N3	C16	C21	118.16(12)
C2	C1	C6	119.46(13)	C17	C16	C21	120.00(12)
C3	C2	C1	119.51(13)	C18	C17	C16	119.19(13)
C2	C3	C4	121.36(13)	C17	C18	C19	121.36(13)
C5	C4	C3	119.37(14)	C20	C19	C18	119.42(13)
C4	C5	C6	120.55(13)	C19	C20	C21	120.77(13)
N2	C6	C1	121.97(12)	N4	C21	C16	122.27(12)
N2	C6	C5	118.29(12)	N4	C21	C20	118.47(12)
C5	C6	C1	119.74(12)	C20	C21	C16	119.26(12)
N2	C7	C8	123.47(11)	N4	C22	C23	123.48(12)
N2	C7	C10	120.00(11)	N4	C22	C25	120.69(11)
C8	C7	C10	116.46(11)	C23	C22	C25	115.82(11)
O1	C8	N1	122.35(12)	O2	C23	N3	122.16(11)
O1	C8	C7	122.10(12)	O2	C23	C22	122.12(12)
N1	C8	C7	115.55(11)	N3	C23	C22	115.71(11)
C7	C10	C11	112.85(11)	C22	C25	C26	113.40(10)
C7	C10	C15	109.05(10)	C22	C25	C30	109.53(10)
C11	C10	C15	110.37(11)	C26	C25	C30	110.12(10)

C12	C11	C10	110.98(11)	C25	C26	C27	110.93(11)
C13	C12	C11	111.60(12)	C28	C27	C26	111.63(11)
C12	C13	C14	110.93(12)	C29	C28	C27	110.92(11)
C13	C14	C15	111.23(11)	C28	C29	C30	111.28(11)
C14	C15	C10	110.97(11)	C29	C30	C25	111.18(11)

Table 6 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for r20200612d.

Atom	*x*	*y*	*z*	U(eq)
H2	5311.1	5776.46	6427.64	35
H3	3259.42	7130.75	7942.74	41
H4	2442.08	6463.38	9794.52	39
H5	3652.85	4401.43	10118.59	33
H9A	6765.18	4103.84	4978.39	50
H9B	8439.1	3102.66	4834.81	50
H9C	7839.13	4468.98	5415.6	50
H10	8258.03	-2.11	7378.86	26
H11A	6332.19	633.24	9798.02	34
H11B	5974.31	139.95	8791.24	34
H12A	6956.89	-1490.78	9941.28	41
H12B	8017.57	-1801.39	8589.4	41
H13A	9549.43	-2273.7	9753.61	37
H13B	8611.95	-910.29	10415.09	37
H14A	10486.29	-1490.4	7987.2	35
H14B	10858.65	-1013.79	8994.17	35
H15A	9884.01	633	7865.29	29
H15B	8811.24	932.71	9214.83	29
H17	8436.31	-1210.22	5104.18	32
H18	8401.91	-2892.69	6174.48	38
H19	6182.8	-2784.09	7618.66	38
H20	3973.08	-978.73	7989.79	33
H24A	8331.22	700.36	4570.12	42
H24B	7250.81	1675.21	3939.68	42
H24C	7754.33	227	3708.74	42
H25	2050.57	3590.83	6006.04	25
H26A	682.49	2629.25	8074.92	29
H26B	589.62	2478.07	6805.11	29
H27A	-832.35	4624.95	6863.73	34

H27B	-1657.27	4125.93	8017.1	34
H28A	-1554.5	5985.68	8490.99	35
H28B	-702.86	4868.39	9158.63	35
H29A	824.23	5910.77	8353.01	38
H29B	746.85	5759.5	7077.87	38
H30A	2239.57	3762.42	8296.37	33
H30B	3077.63	4266.89	7153.57	33

Experimental

Single crystals of $C_{15}H_{18}N_2O$ **[r20200612d]** were **[]**. A suitable crystal was selected and **[]** on a **'Rigaku Saturn 70 CCD'** diffractometer. The crystal was kept at 113.15 K during data collection. Using Olex2 [1], the structure was solved with the ShelXT [2] structure solution program using Intrinsic Phasing and refined with the ShelXL [3] refinement package using Least Squares minimisation.

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H. (2009), J. Appl. Cryst. 42, 339-341.
2. Sheldrick, G.M. (2015). Acta Cryst. A71, 3-8.
3. Sheldrick, G.M. (2015). Acta Cryst. C71, 3-8.

Crystal structure determination of [r20200612d]

Crystal Data for $C_{15}H_{18}N_2O$ (M =242.31 g/mol): triclinic, space group P-1 (no. 2), a = 10.3988(9) Å, b = 11.9760(9) Å, c = 12.1316(8) Å, α = 86.151(6)°, β = 70.363(7)°, γ = 65.045(8)°, V = 1285.17(19) Å^3, Z = 4, T = 113.15 K, μ(MoKα) = 0.080 mm^{-1}, *Dcalc* = 1.252 g/cm^3, 13407 reflections measured (3.578° ≤ 2Θ ≤ 52.734°), 5209 unique (R_{int} = 0.0265, R_{sigma} = 0.0300) which were used in all calculations. The final R_1 was 0.0433 (I > 2σ(I)) and wR_2 was 0.1162 (all data).

Refinement model description

Number of restraints - 0, number of constraints - unknown.

Details:

1. Fixed Uiso

At 1.2 times of:

All C(H) groups, All C(H,H) groups

At 1.5 times of:

All C(H,H,H) groups

2.a Ternary CH refined with riding coordinates:

C10(H10), C25(H25)

2.b Secondary CH2 refined with riding coordinates:

C11(H11A,H11B), C12(H12A,H12B), C13(H13A,H13B), C14(H14A,H14B), C15(H15A, H15B), C26(H26A,H26B), C27(H27A,H27B), C28(H28A,H28B), C29(H29A,H29B), C30(H30A,H30B)

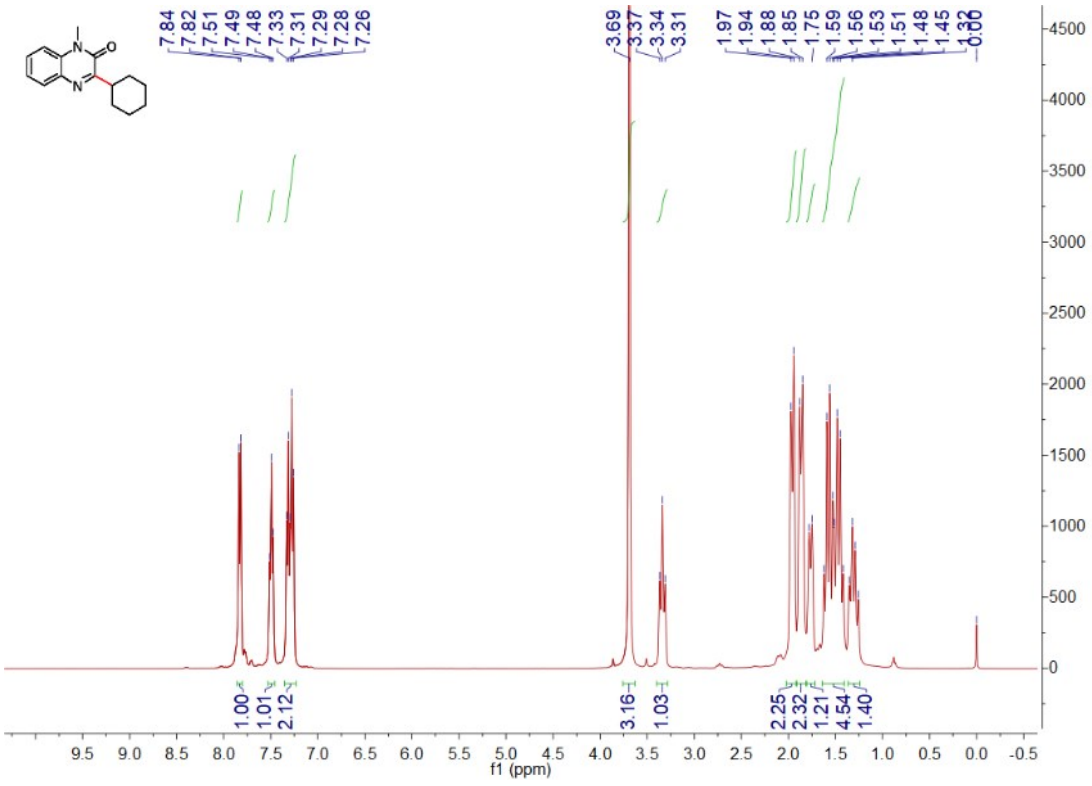
2.c Aromatic/amide H refined with riding coordinates:

C2(H2), C3(H3), C4(H4), C5(H5), C17(H17), C18(H18), C19(H19), C20(H20)

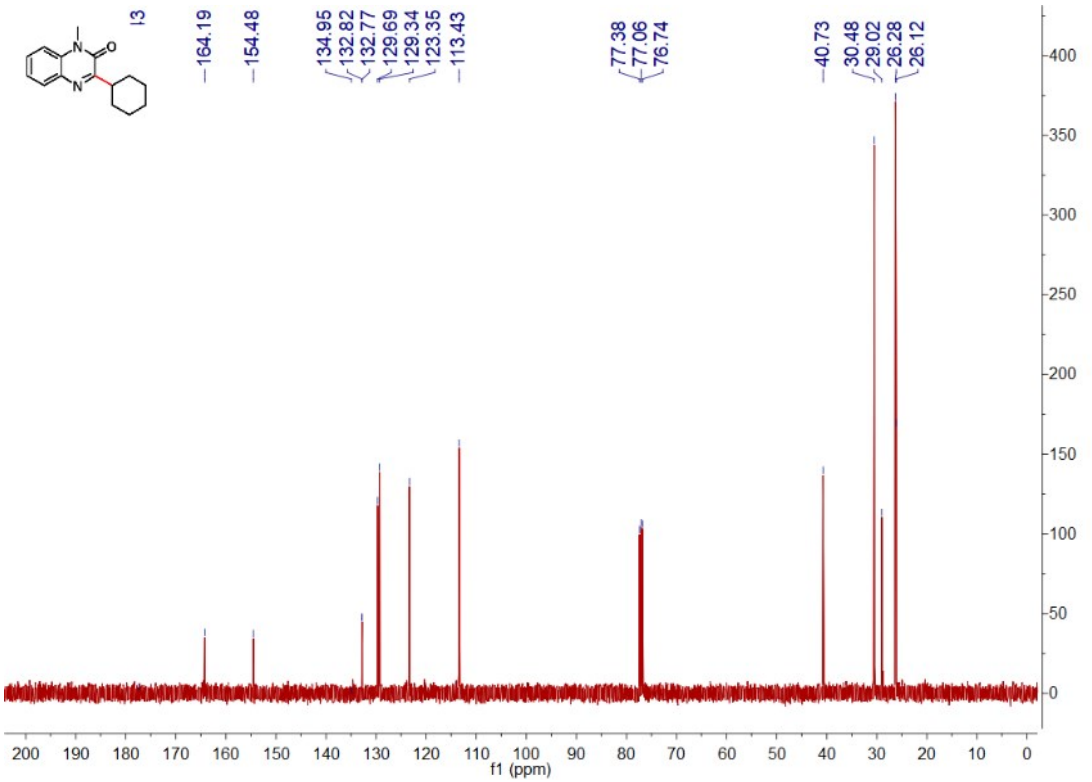
2.d Idealised Me refined as rotating group:
C9(H9A,H9B,H9C), C24(H24A,H24B,H24C)

12. Spectra of prepared compounds

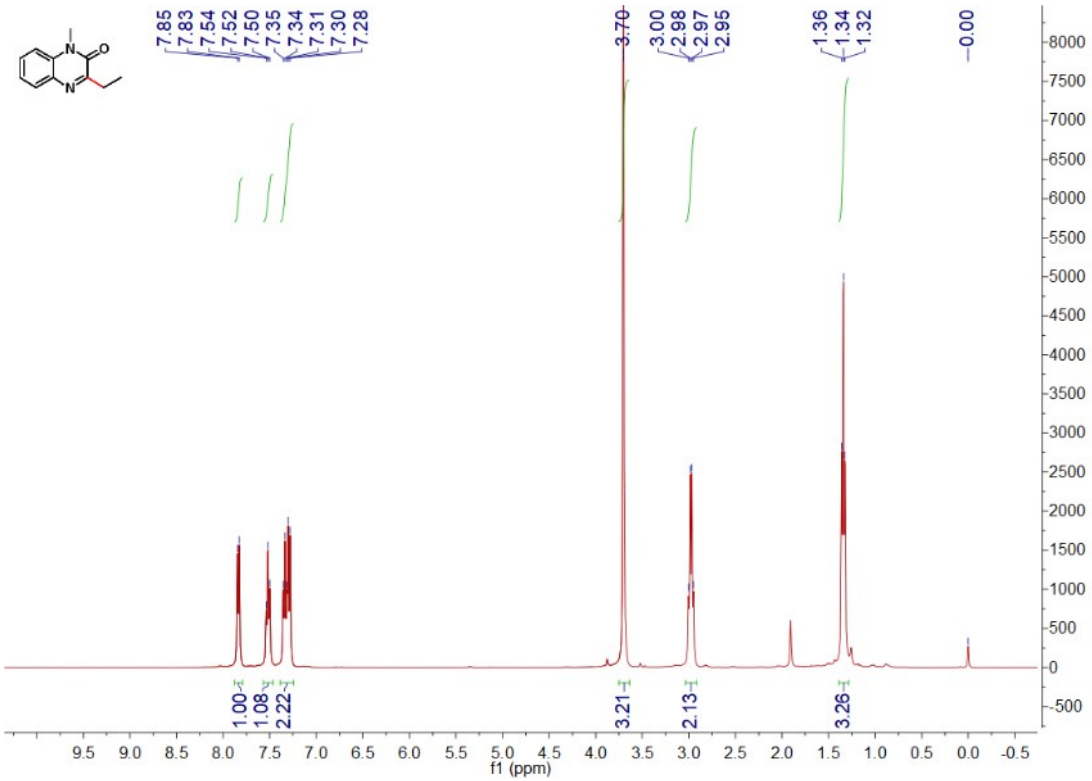
^{1}H NMR spectrum of compound **3aa**



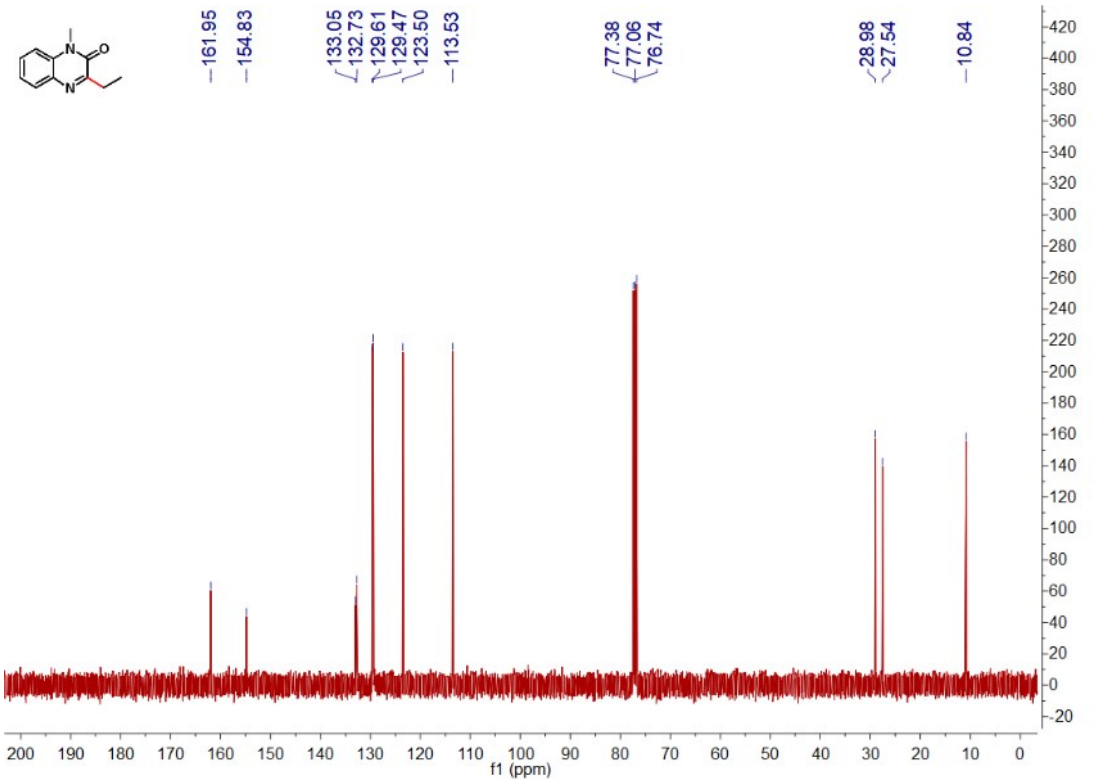
^{13}C NMR spectrum of compound **3aa**



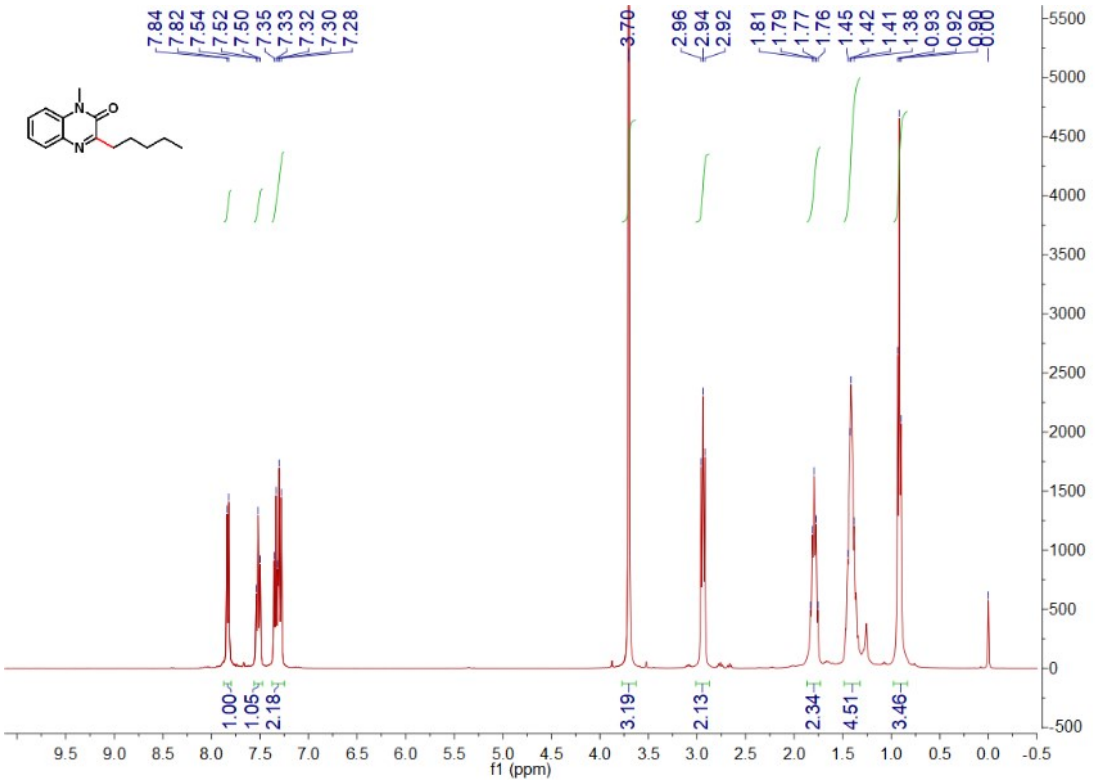
^{1}H NMR spectrum of compound **3ab**



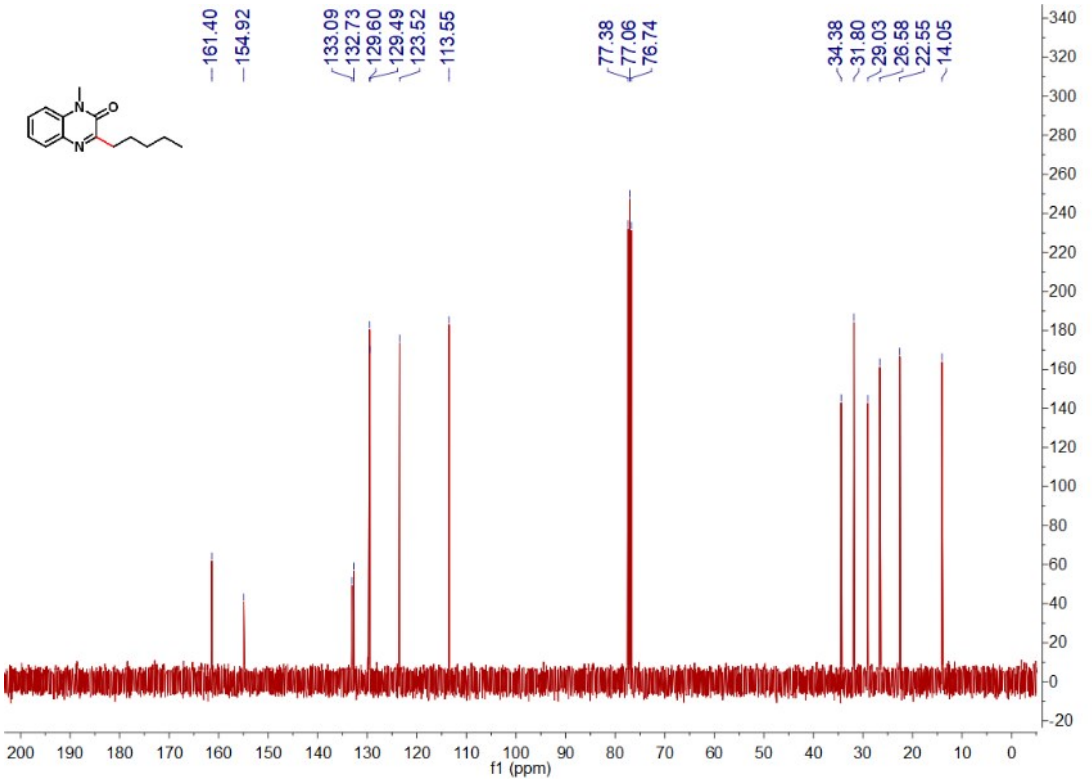
^{13}C NMR spectrum of compound **3ab**



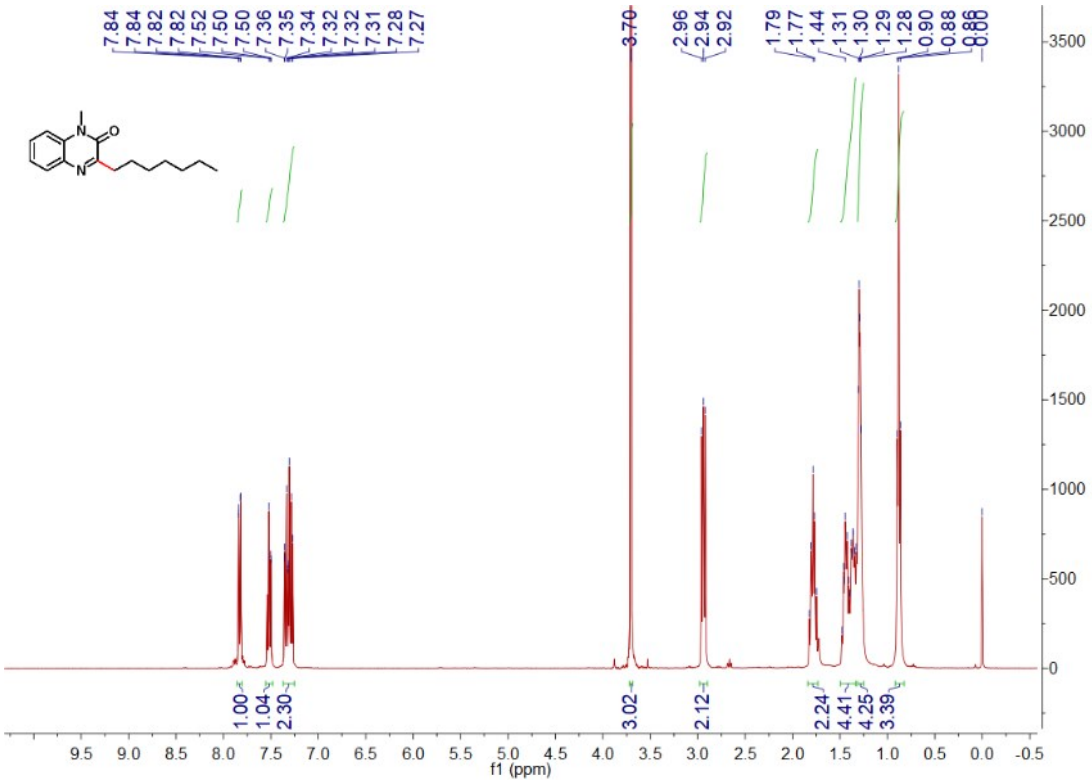
^{1}H NMR spectrum of compound **3ac**



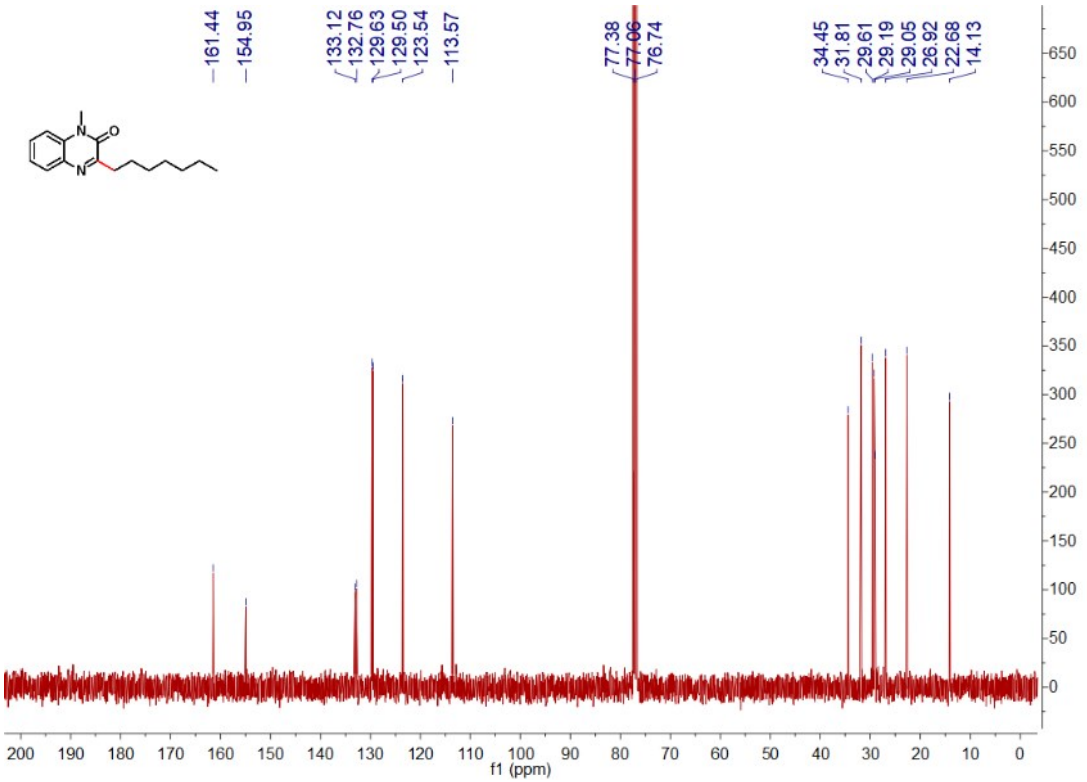
^{13}C NMR spectrum of compound **3ac**



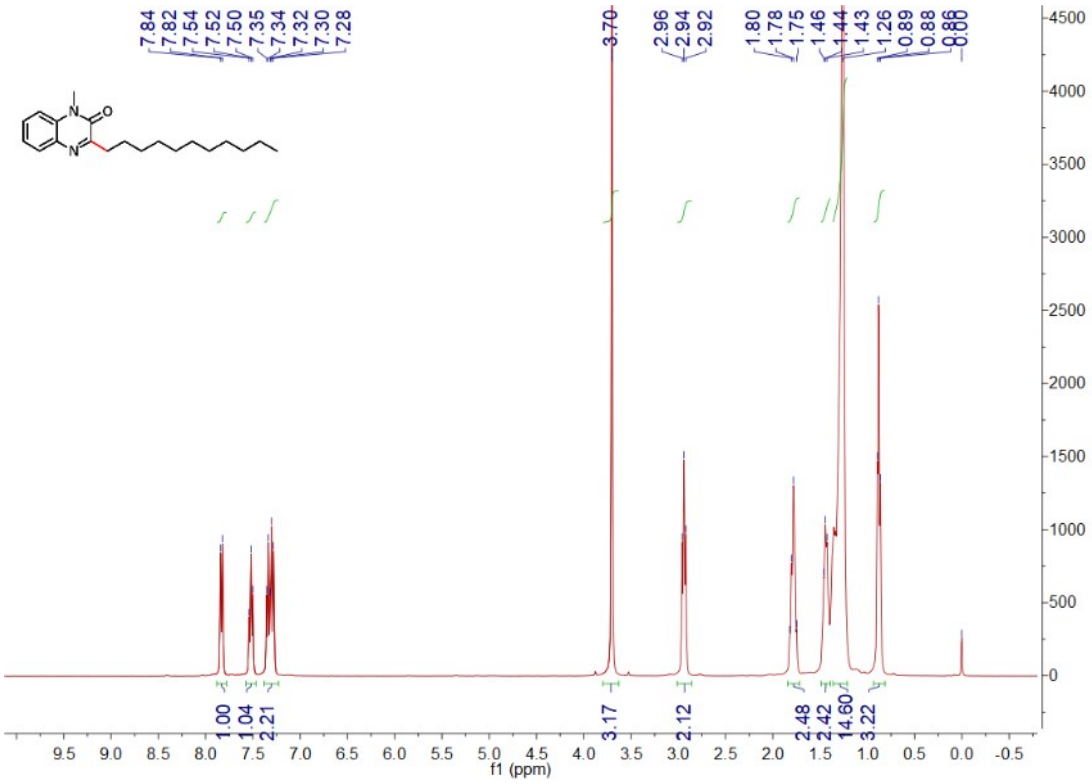
^{1}H NMR spectrum of compound **3ad**



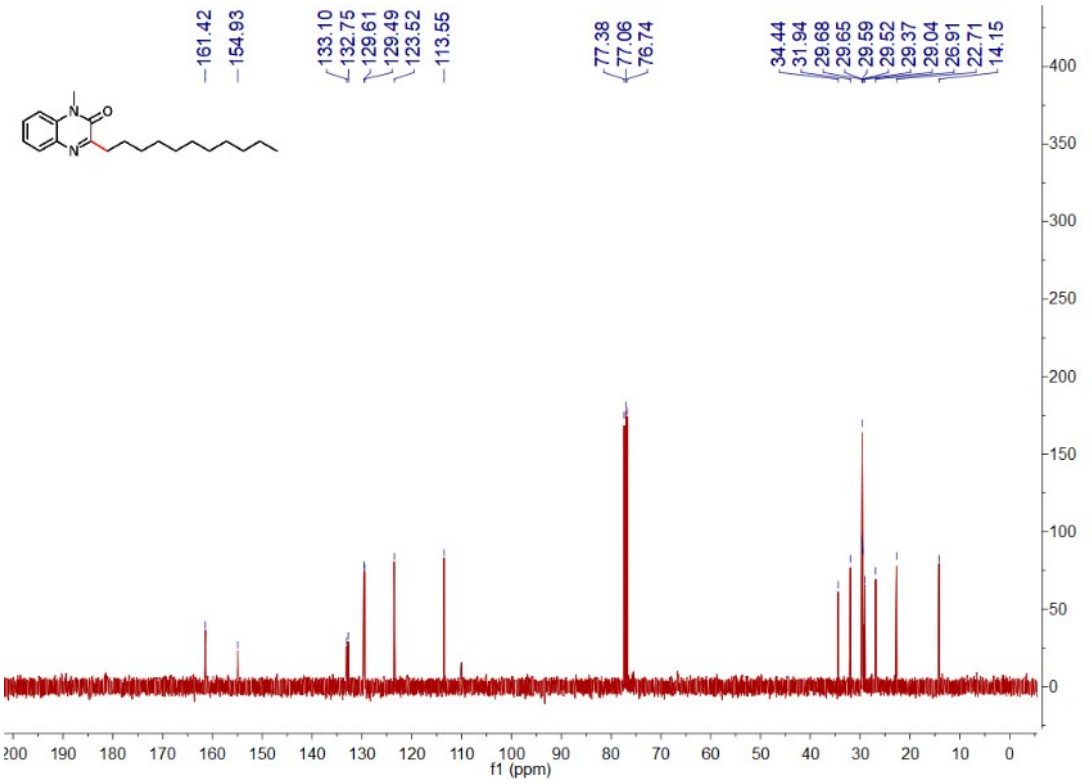
^{13}C NMR spectrum of compound **3ad**



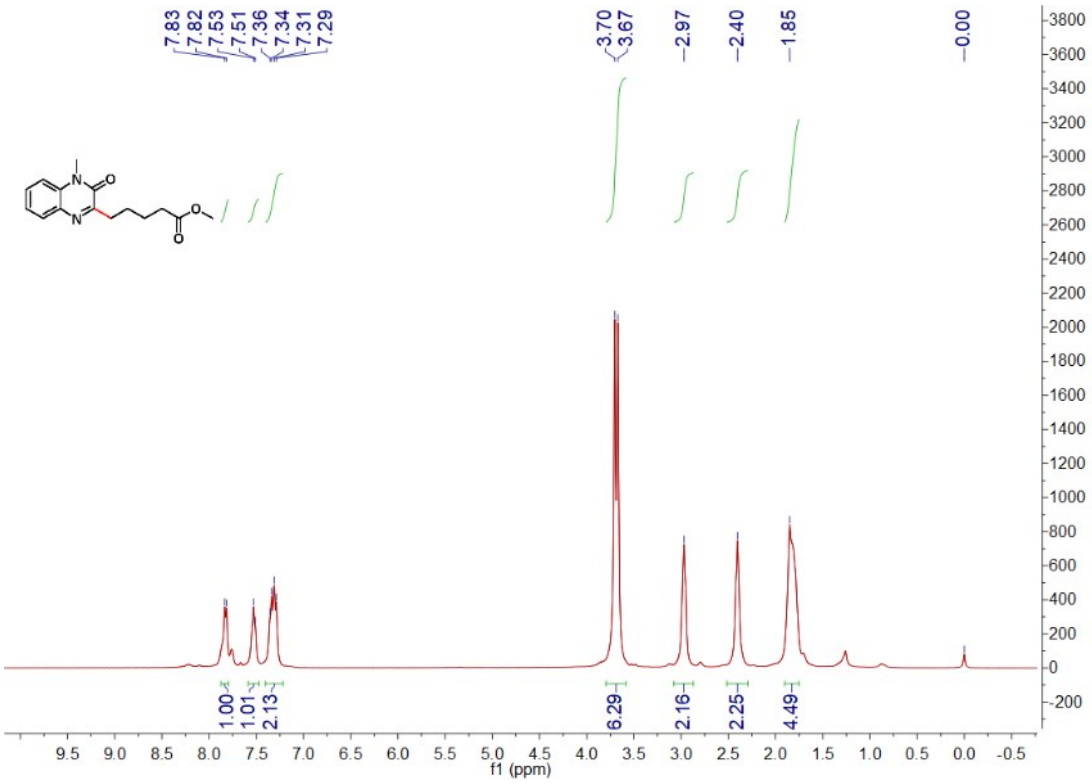
^{1}H NMR spectrum of compound **3ae**



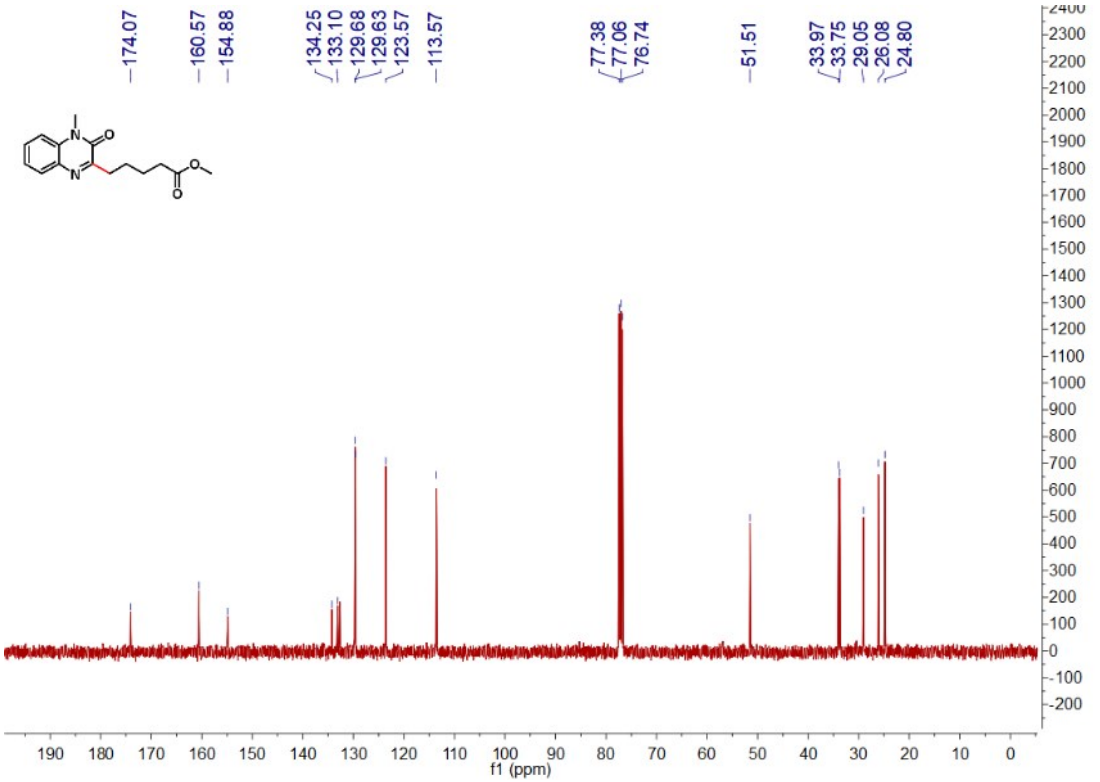
^{13}C NMR spectrum of compound **3ae**



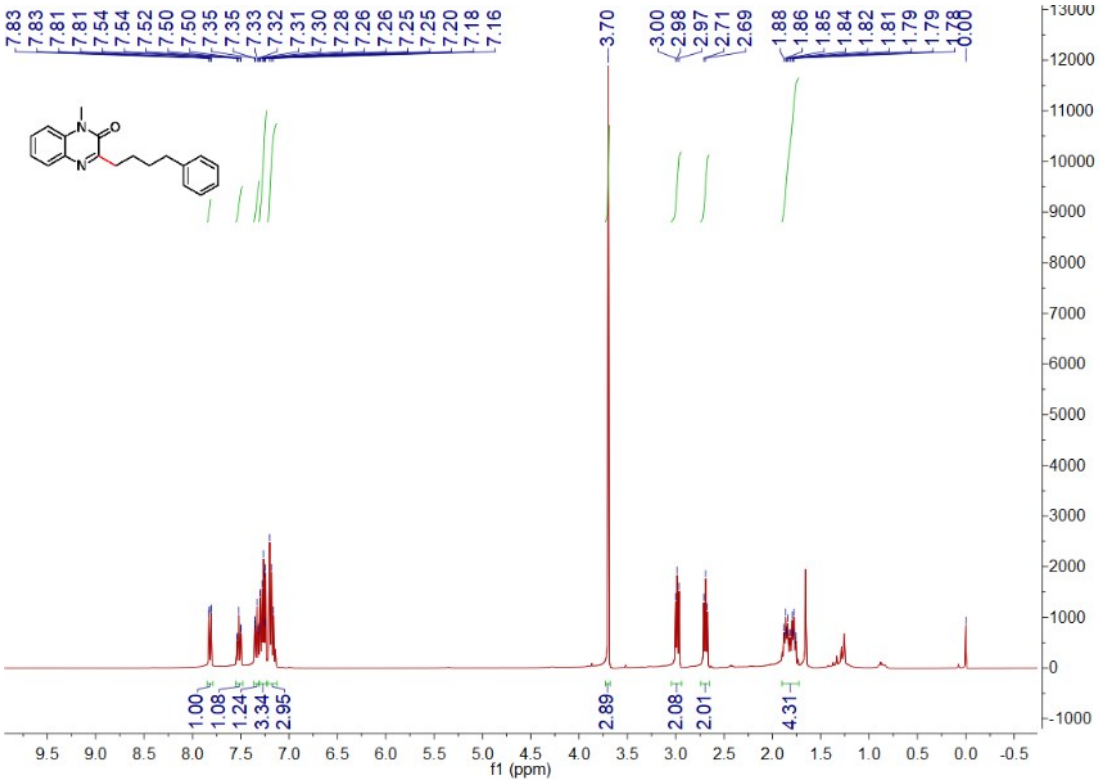
^{1}H NMR spectrum of compound **3af**



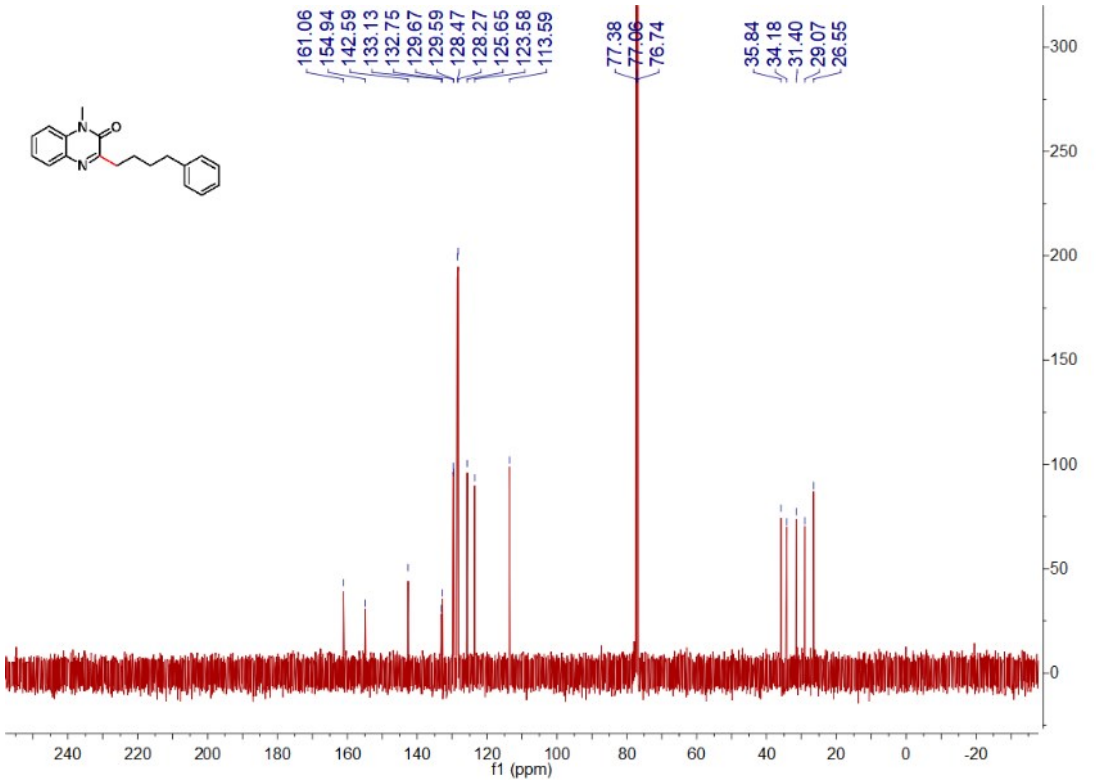
^{13}C NMR spectrum of compound **3af**



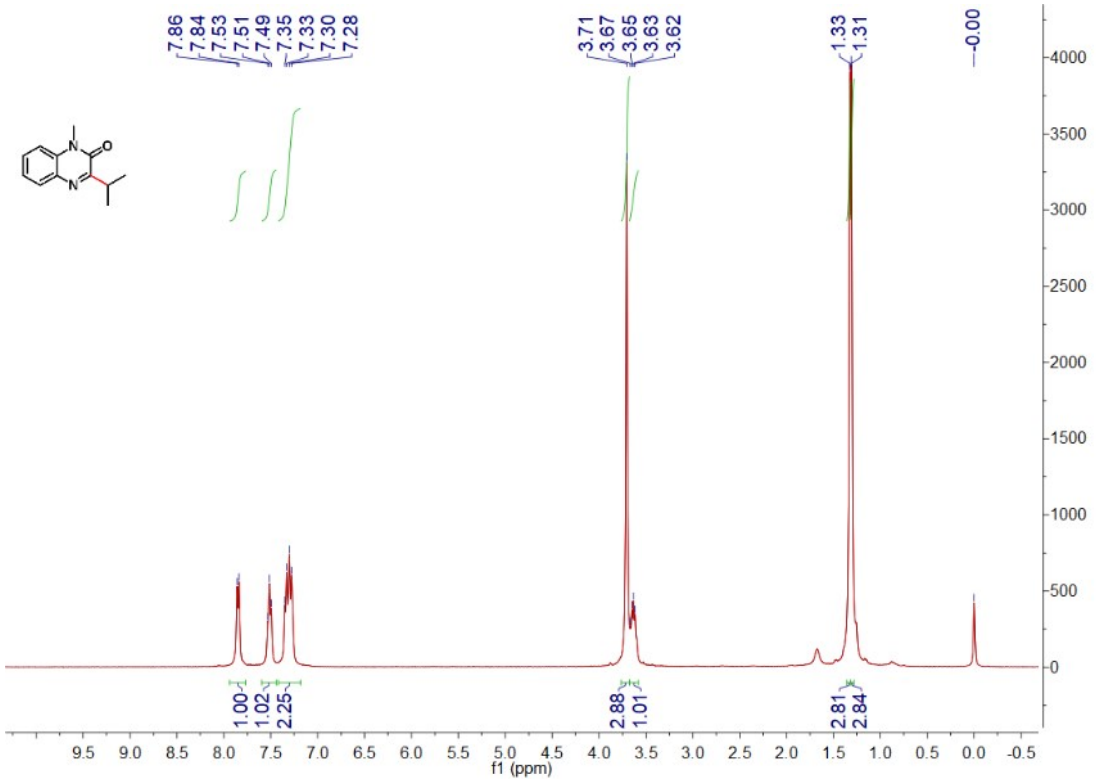
^{1}H NMR spectrum of compound **3ag**



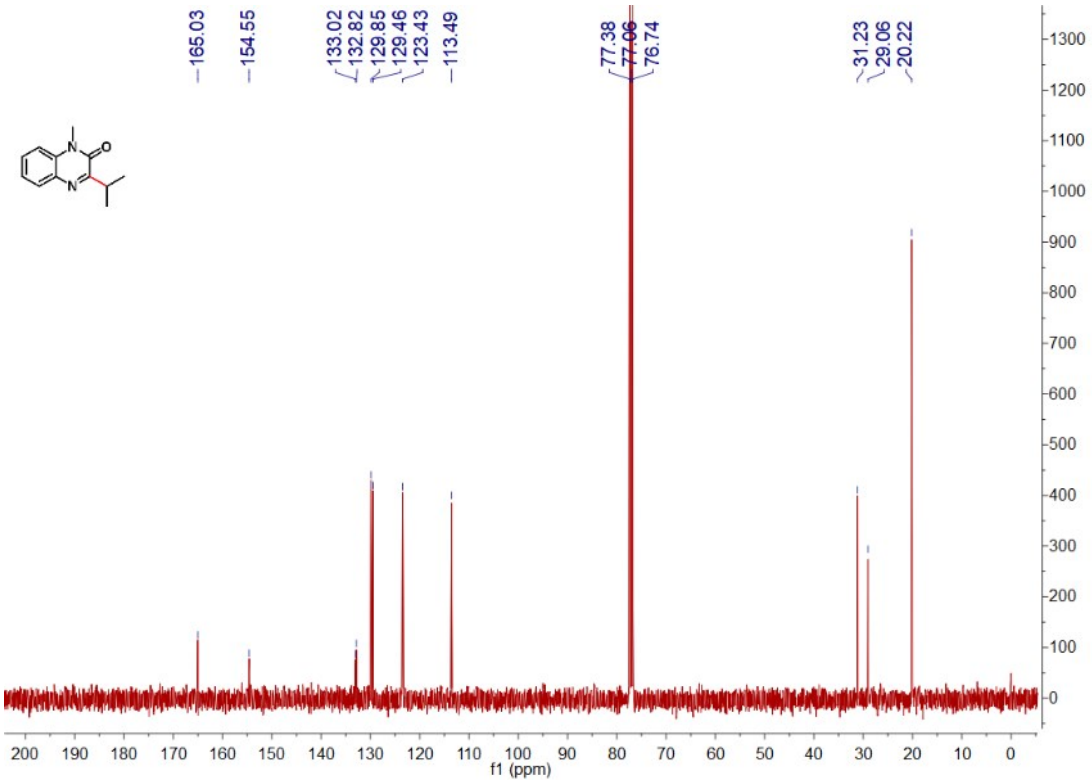
^{13}C NMR spectrum of compound **3ag**



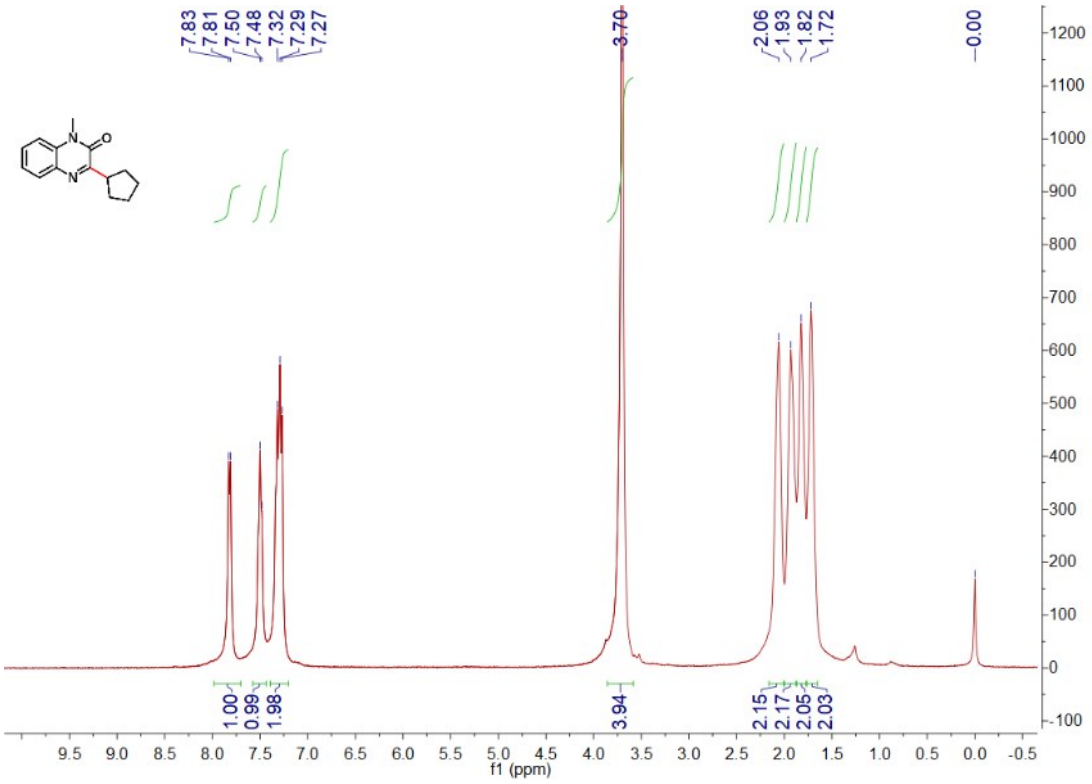
^{1}H NMR spectrum of compound **3ah**



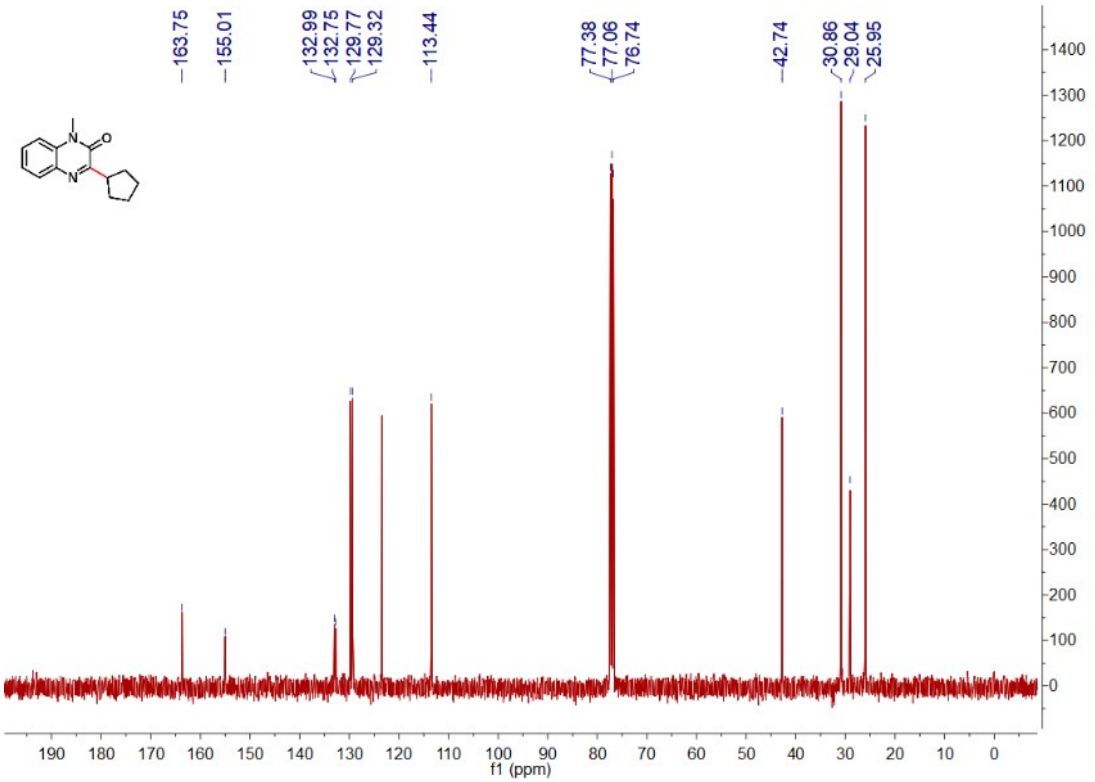
^{13}C NMR spectrum of compound **3ah**



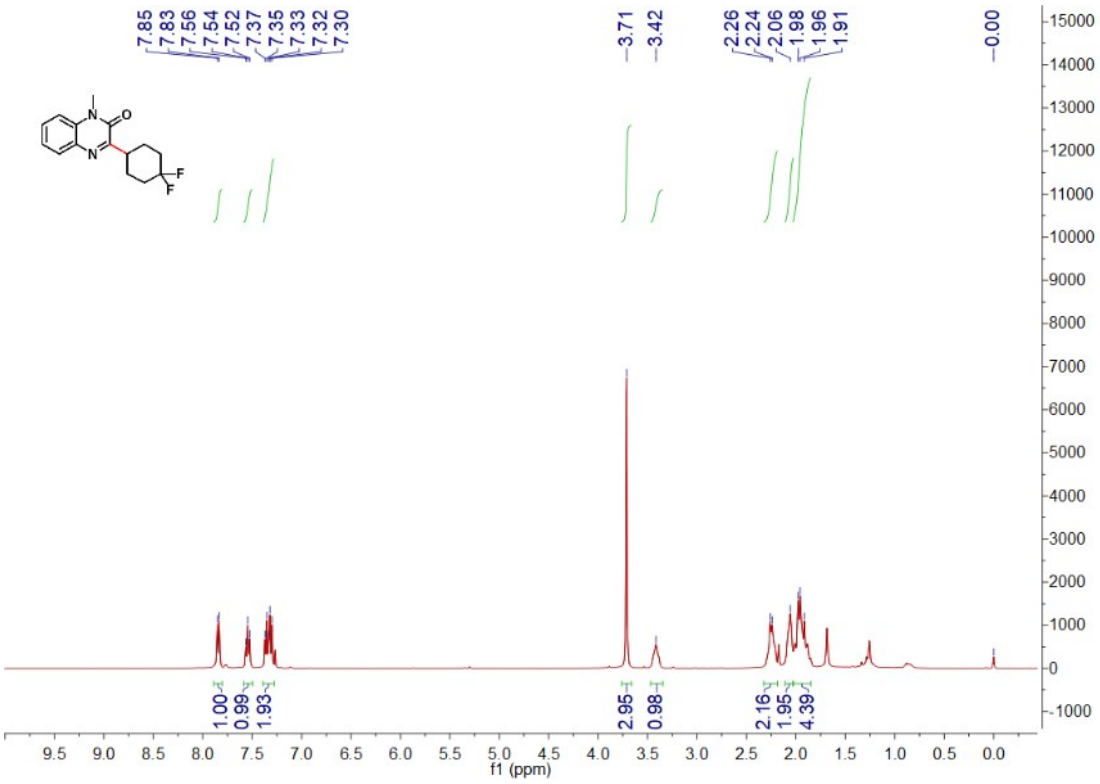
^{1}H NMR spectrum of compound **3ai**



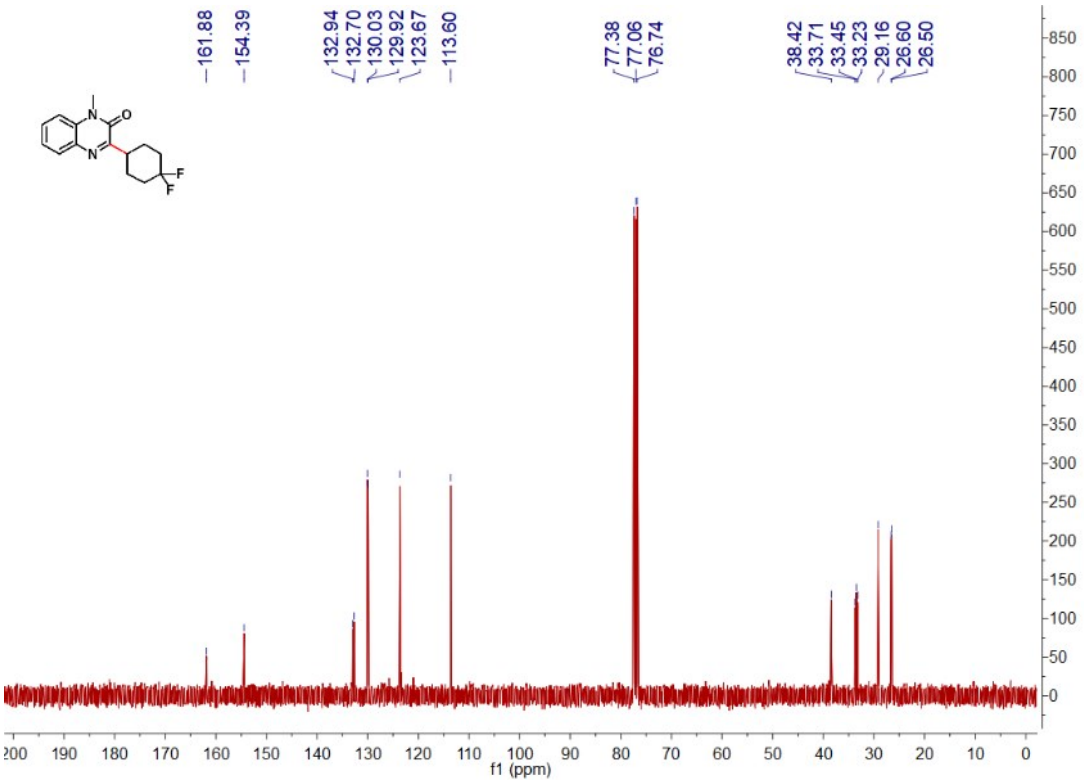
^{13}C NMR spectrum of compound **3ai**



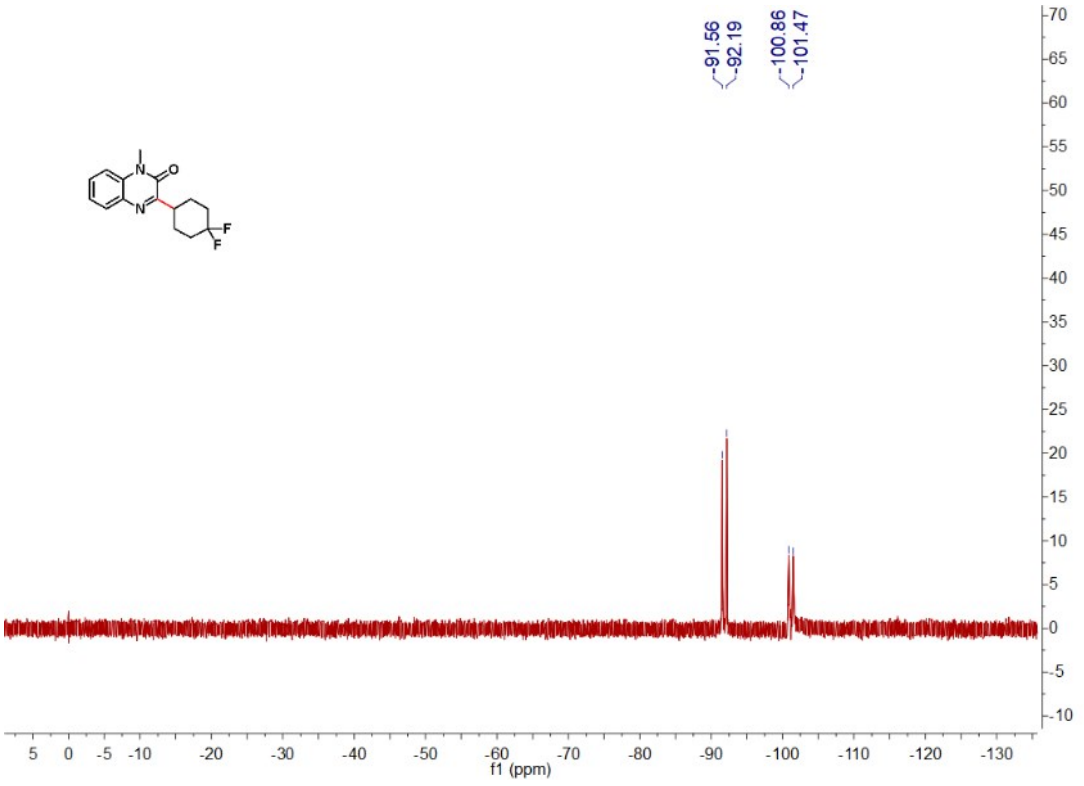
^{1}H NMR spectrum of compound **3aj**



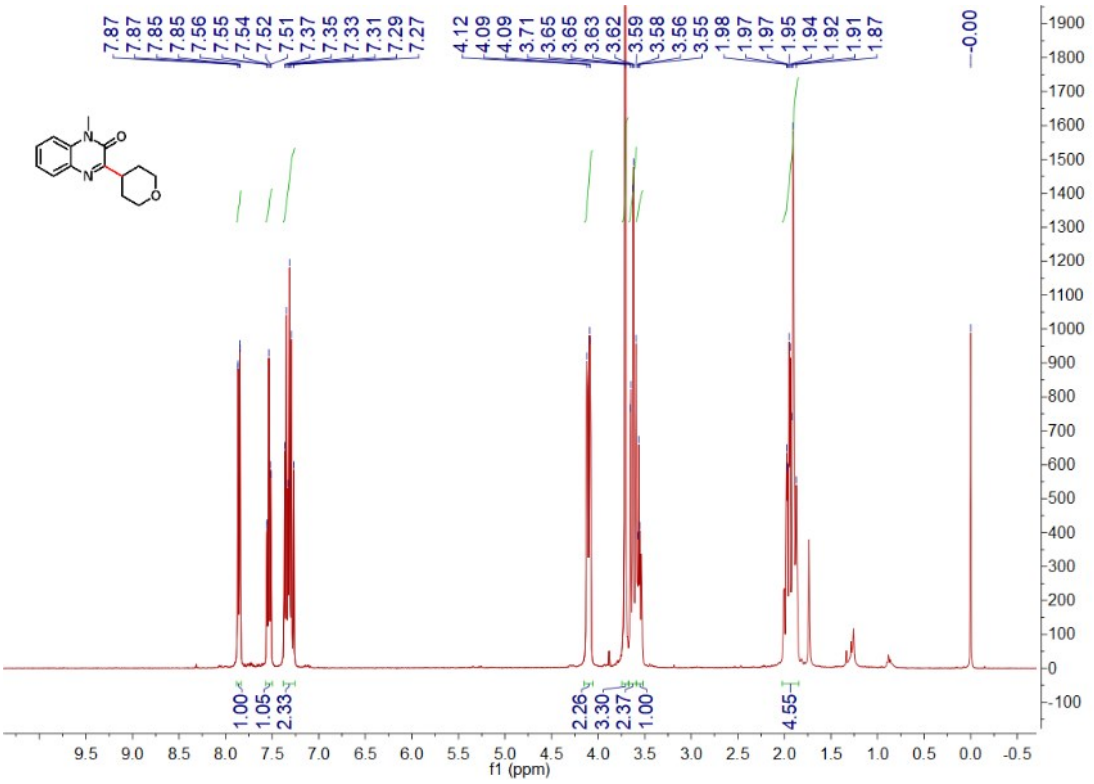
^{13}C NMR spectrum of compound **3aj**



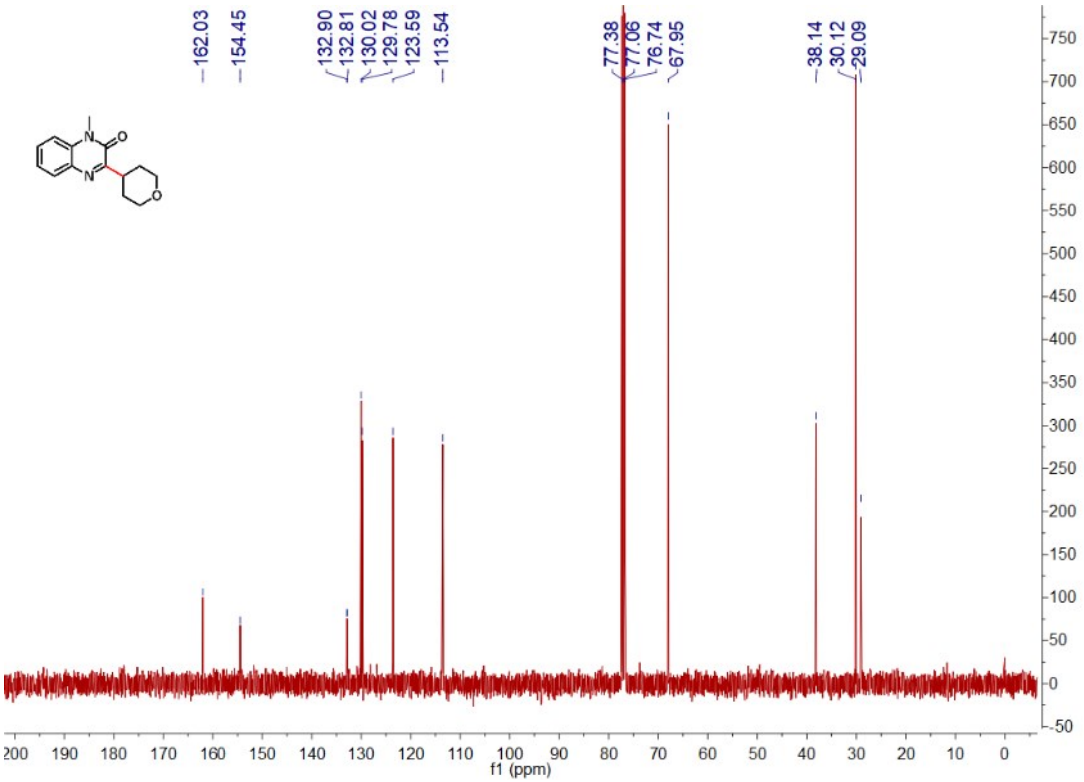
^{19}F NMR spectrum of compound **3aj**



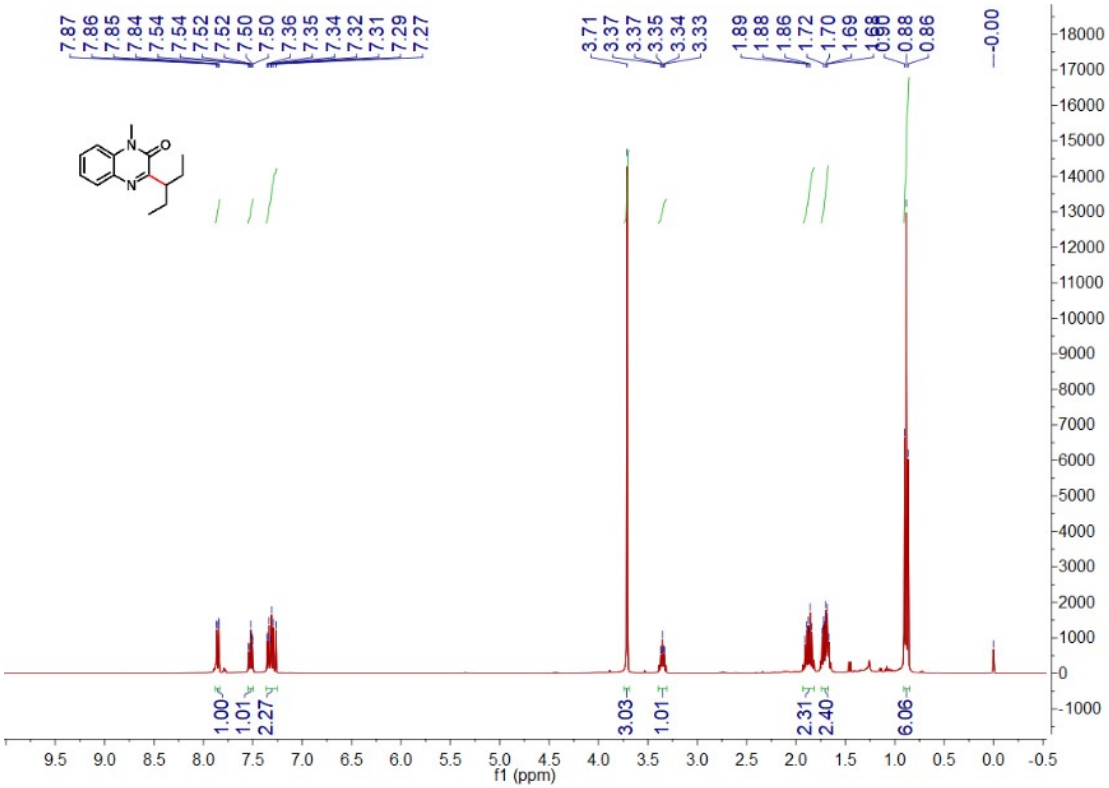
[1]H NMR spectrum of compound **3ak**



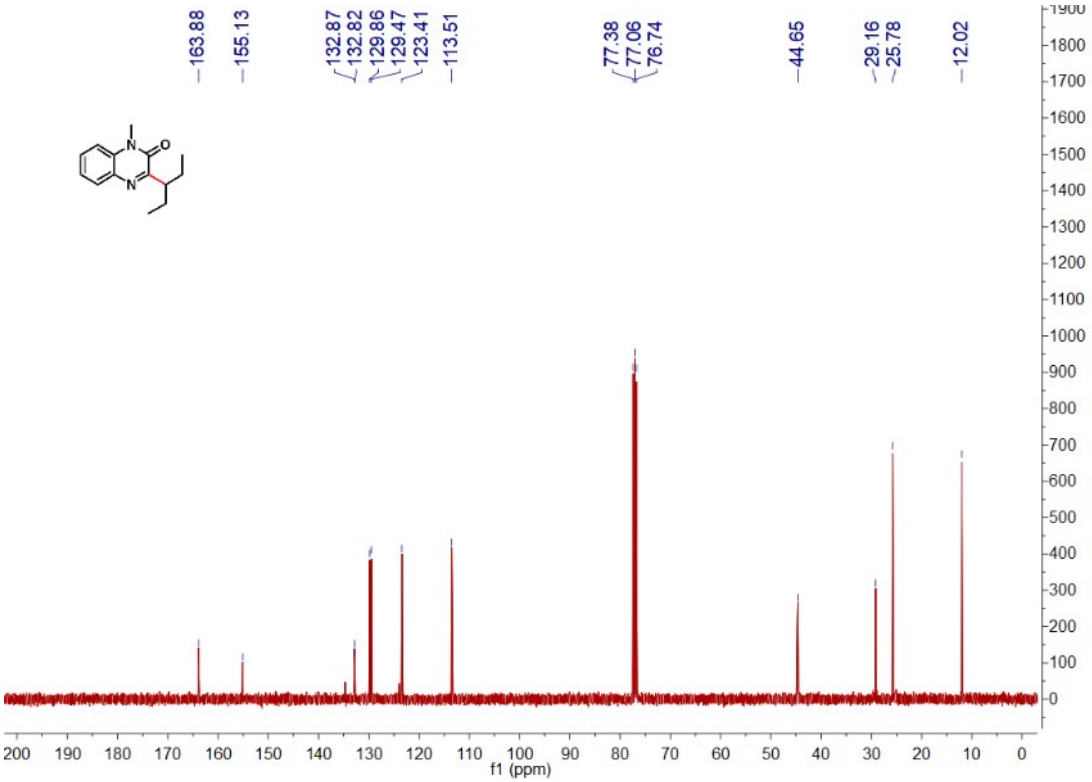
[13]C NMR spectrum of compound **3ak**



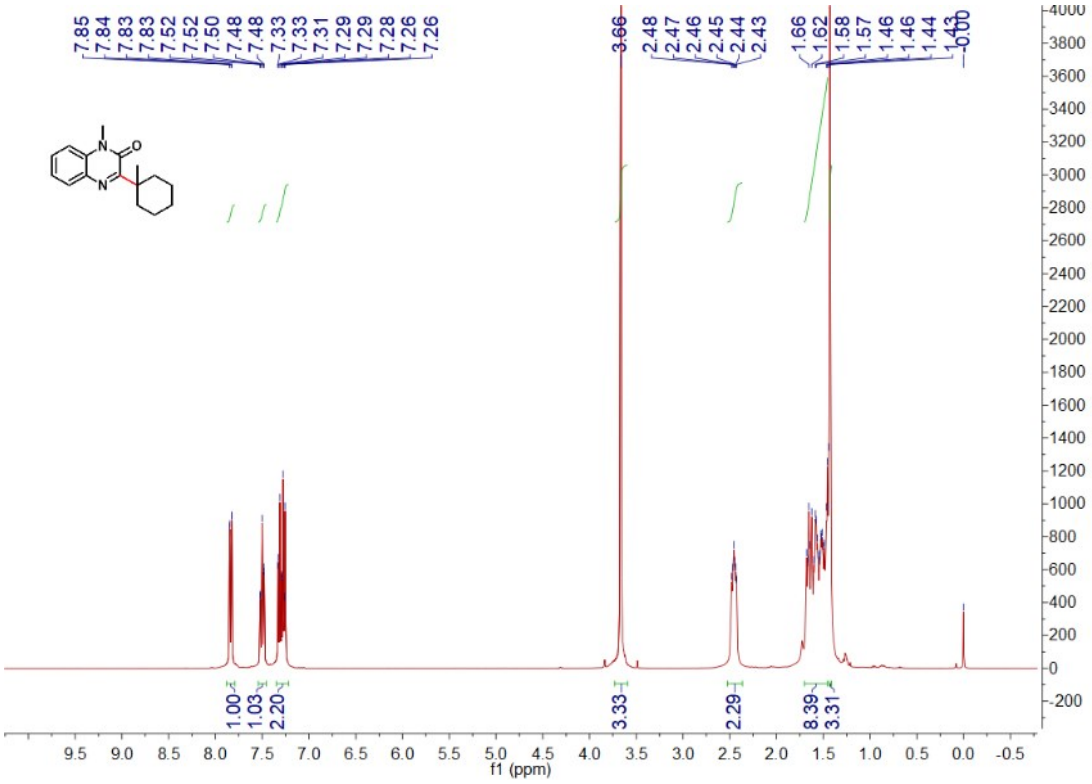
^{1}H NMR spectrum of compound **3al**



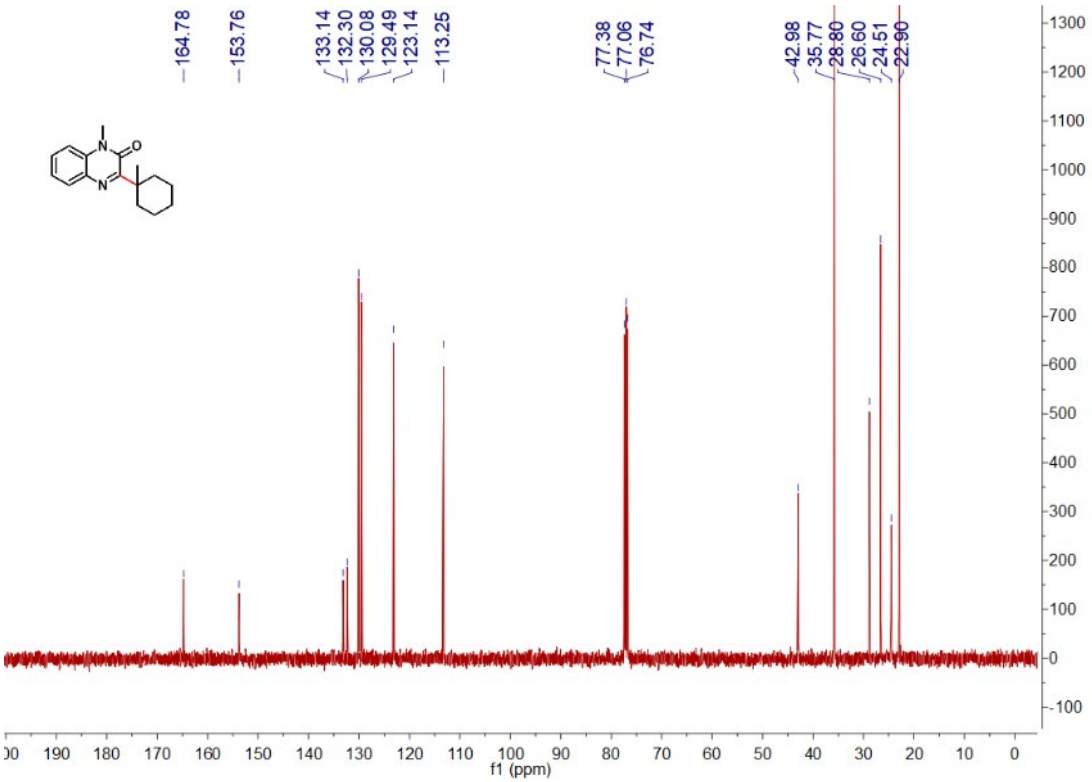
^{13}C NMR spectrum of compound **3al**



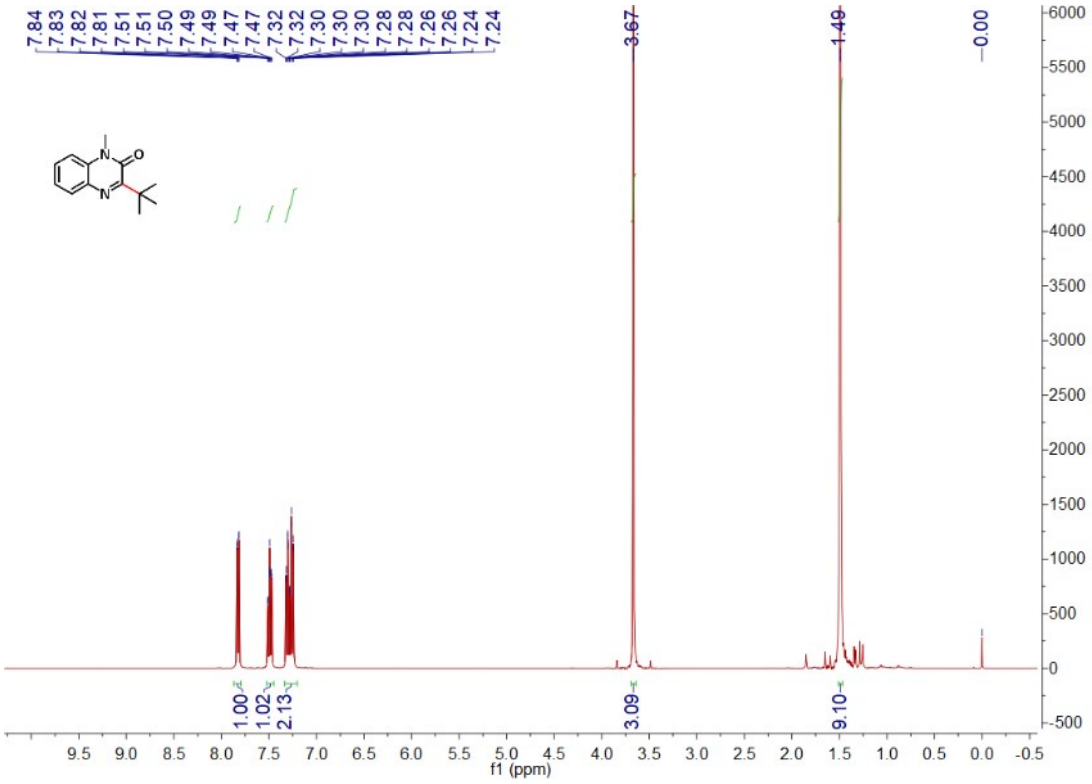
^{1}H NMR spectrum of compound **3am**



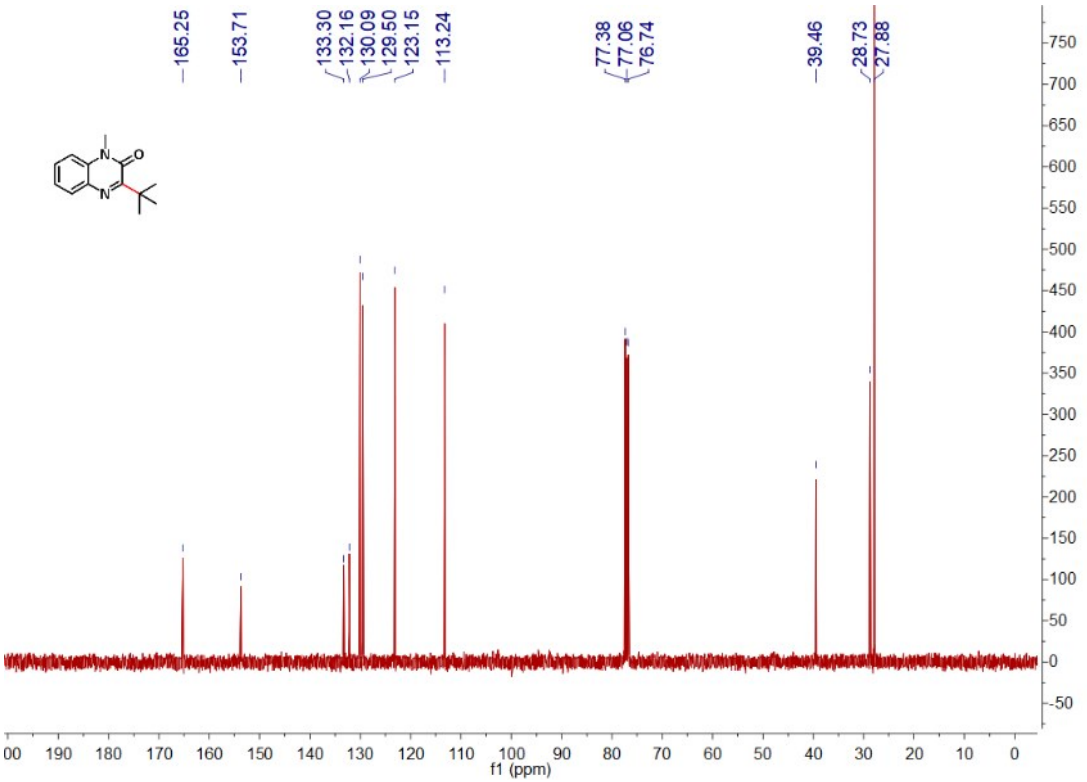
^{13}C NMR spectrum of compound **3am**



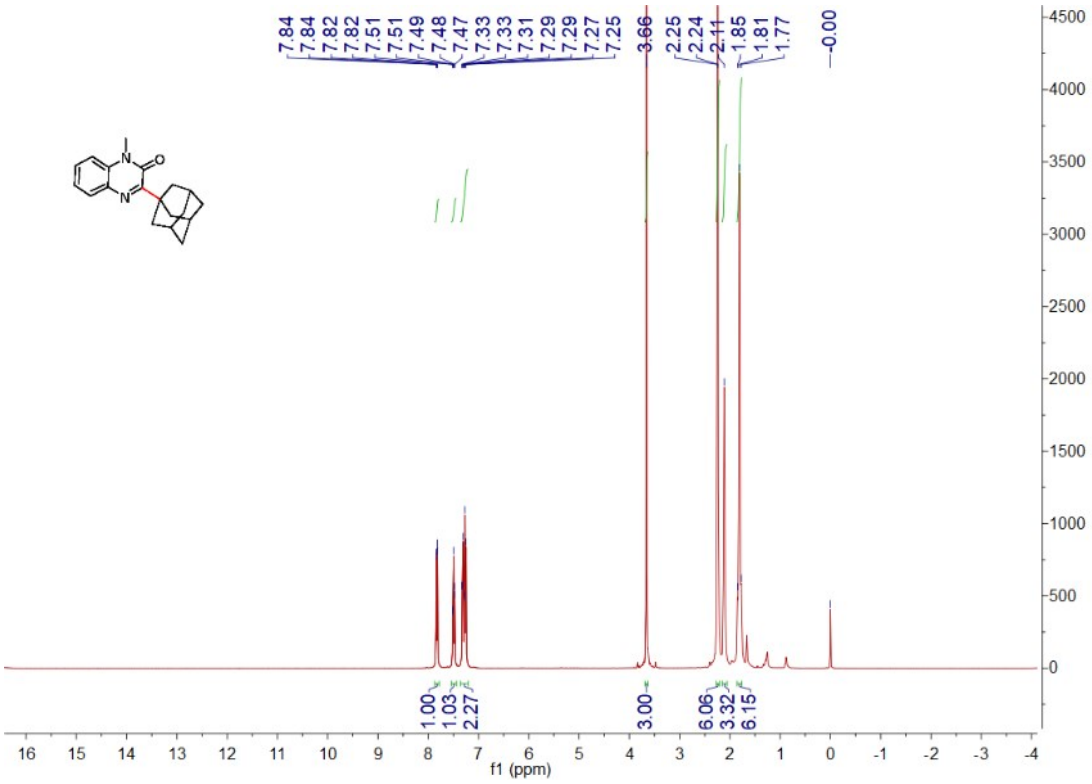
^{1}H NMR spectrum of compound **3an**



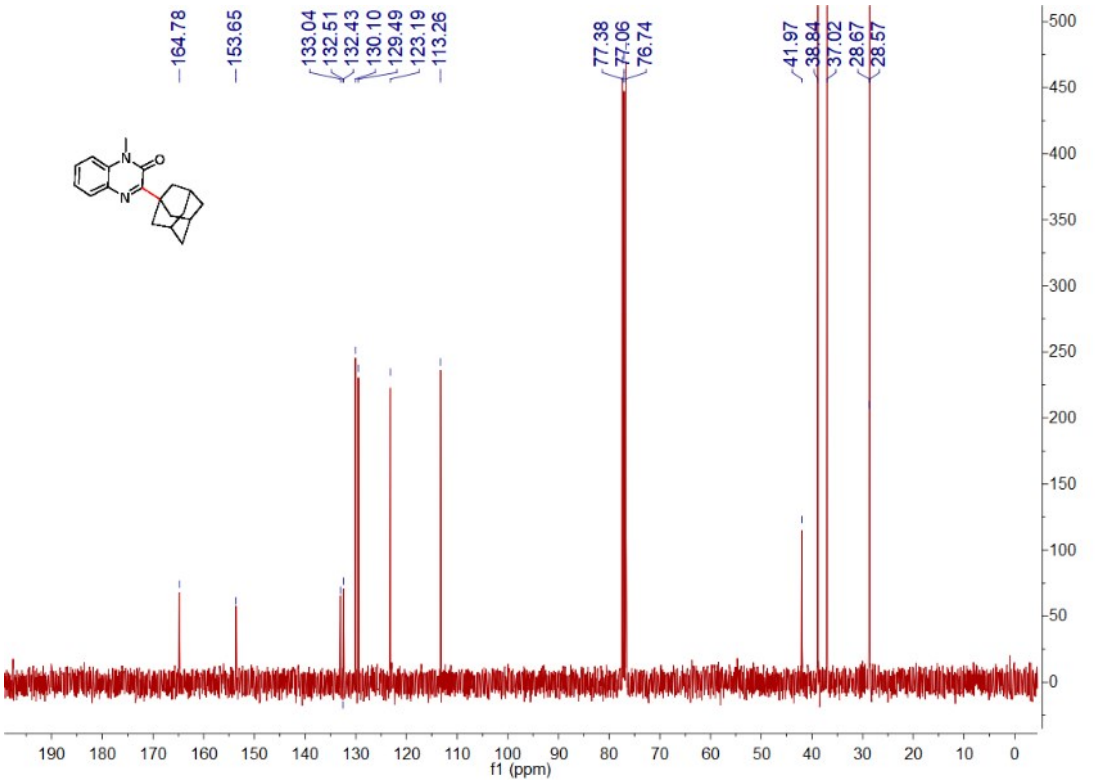
^{13}C NMR spectrum of compound **3an**



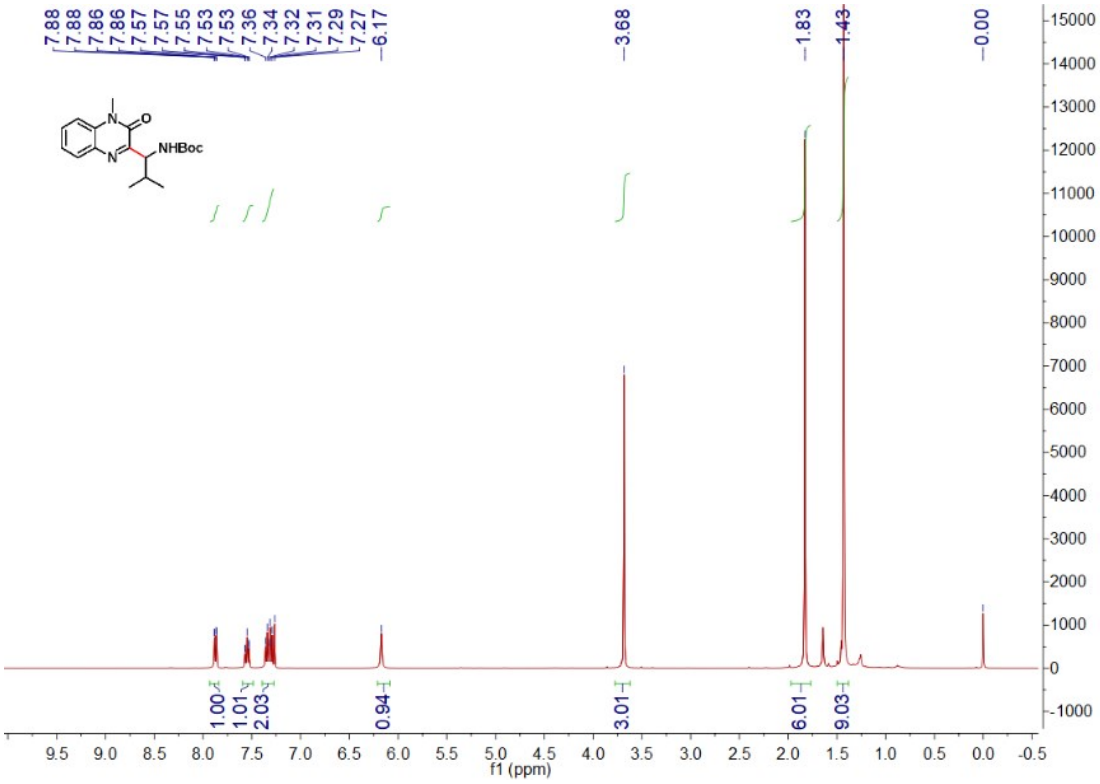
^{1}H NMR spectrum of compound **3ao**



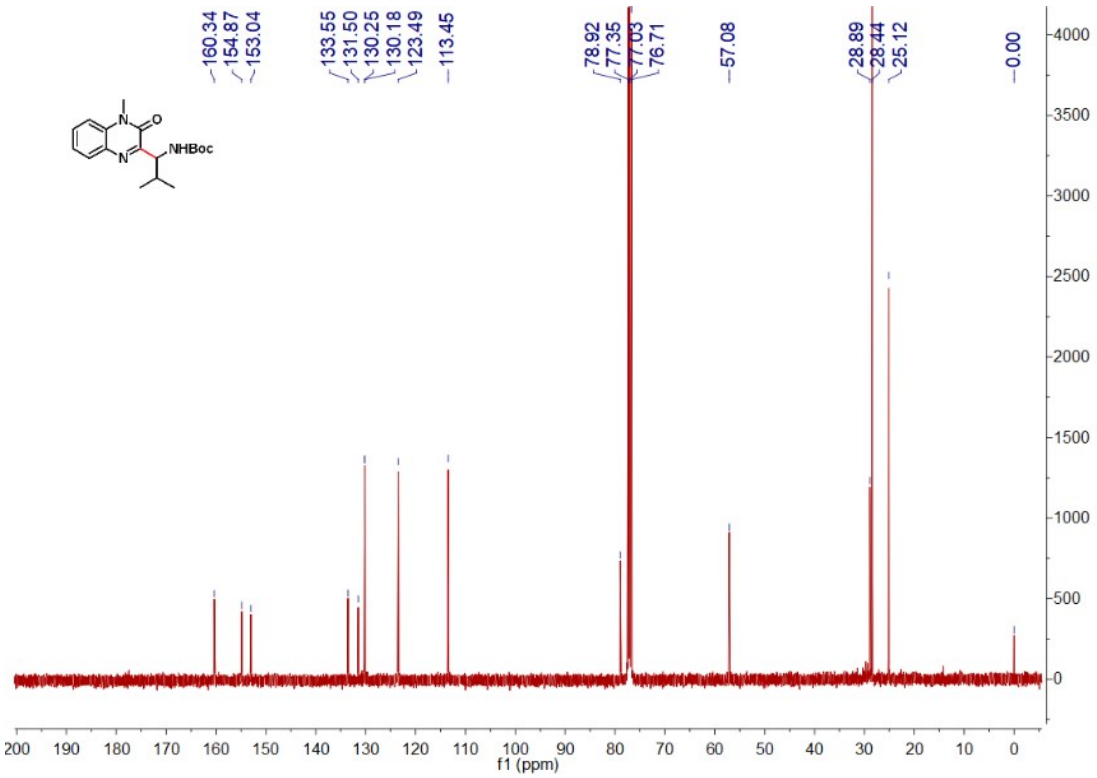
^{13}C NMR spectrum of compound **3ao**



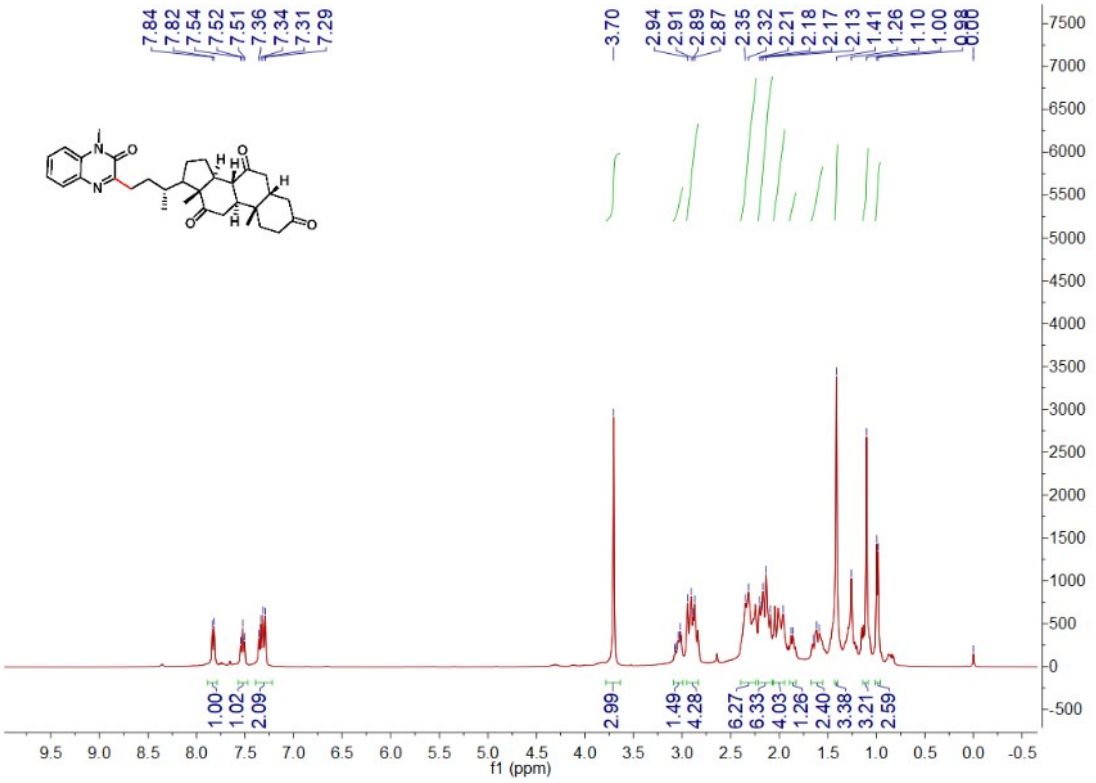
^{1}H NMR spectrum of compound **3ap**



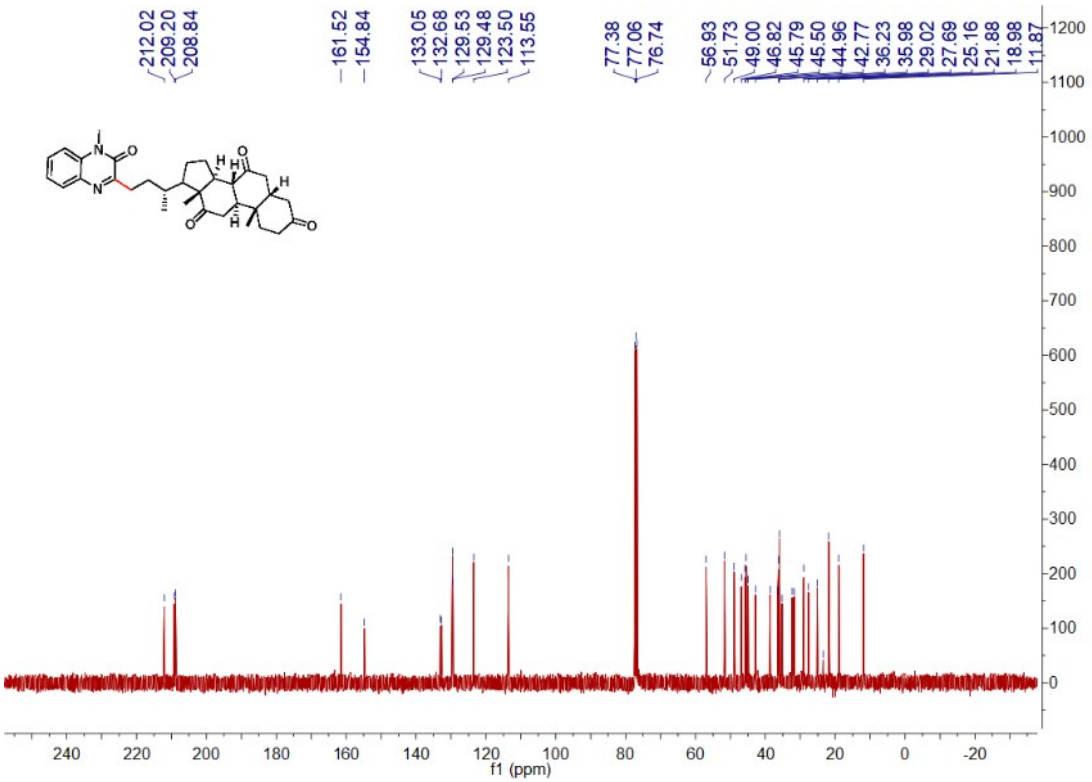
^{13}C NMR spectrum of compound **3ap**



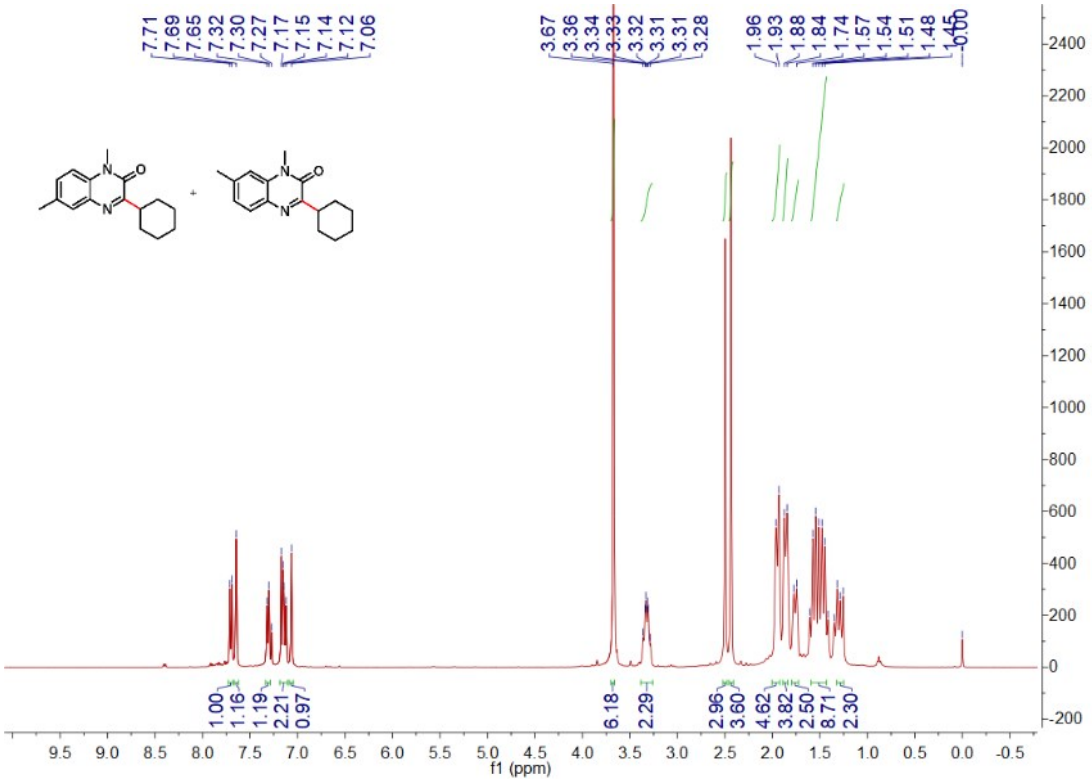
^{1}H NMR spectrum of compound **3aq**



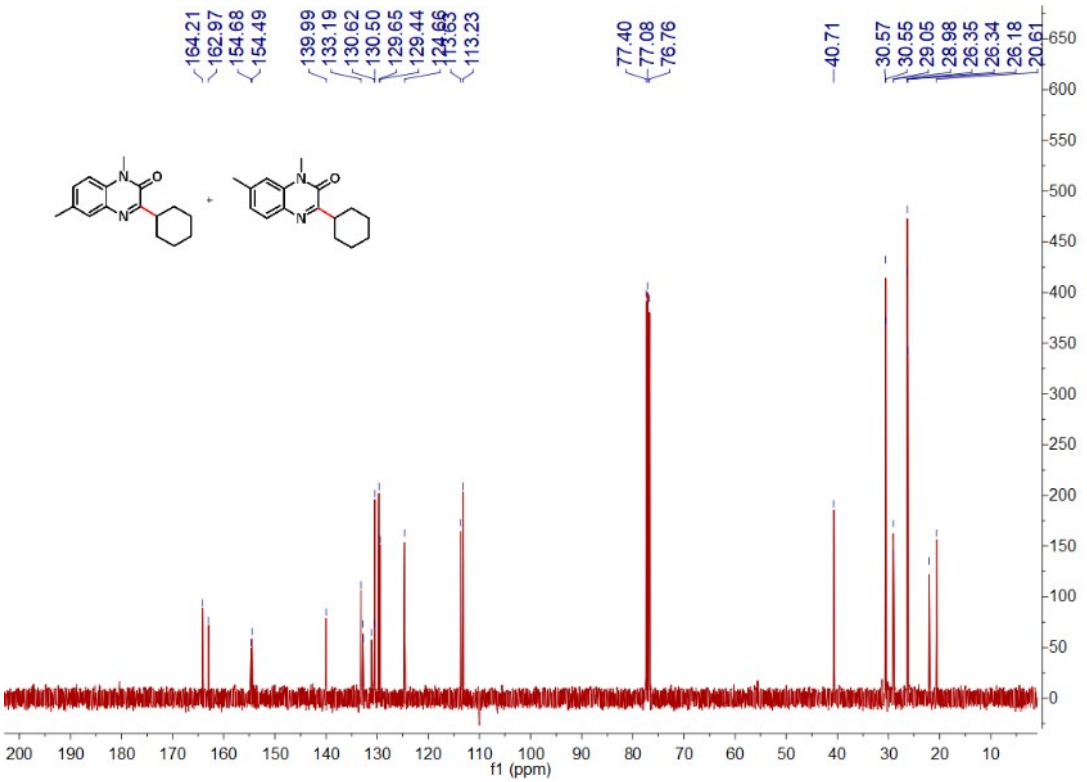
^{13}C NMR spectrum of compound **3aq**



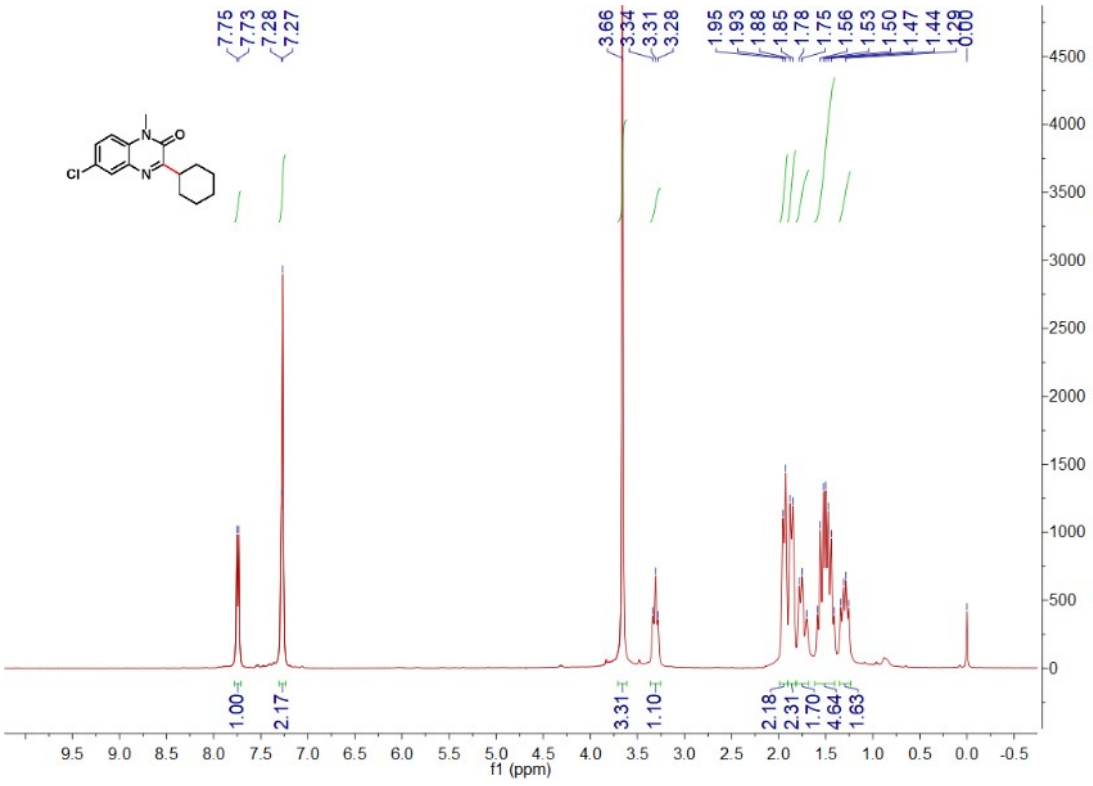
^{1}H NMR spectrum of compound **3ba**



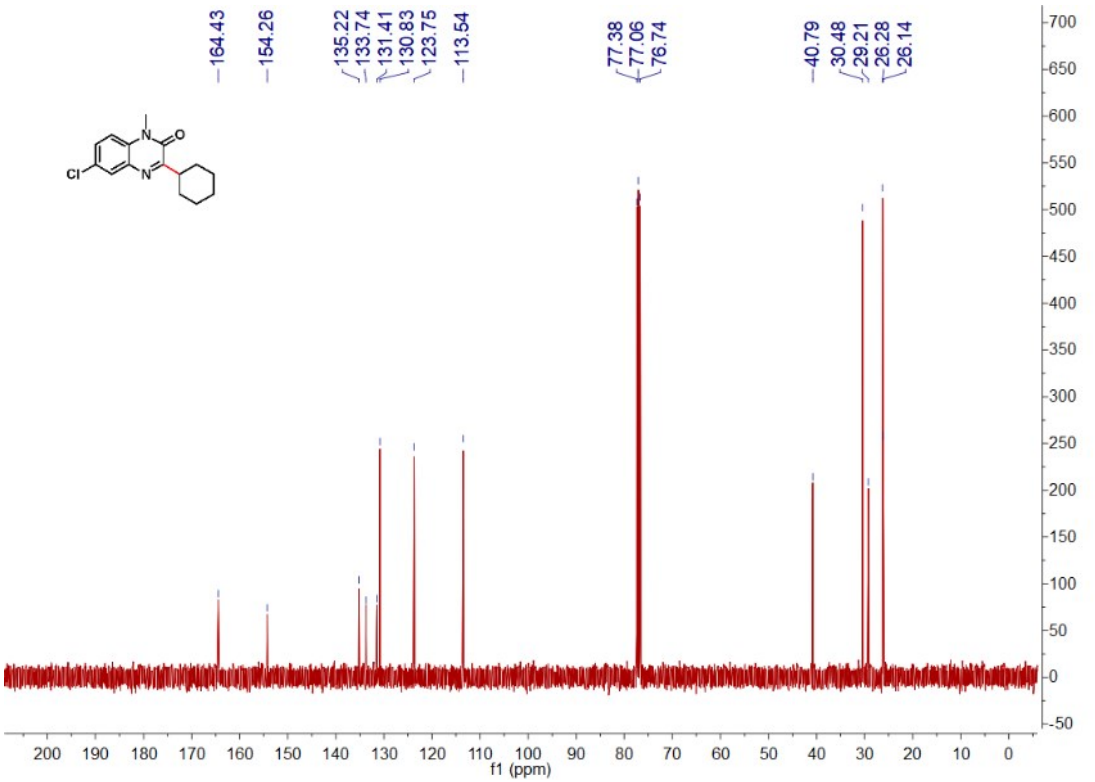
^{13}C NMR spectrum of compound **3ba**



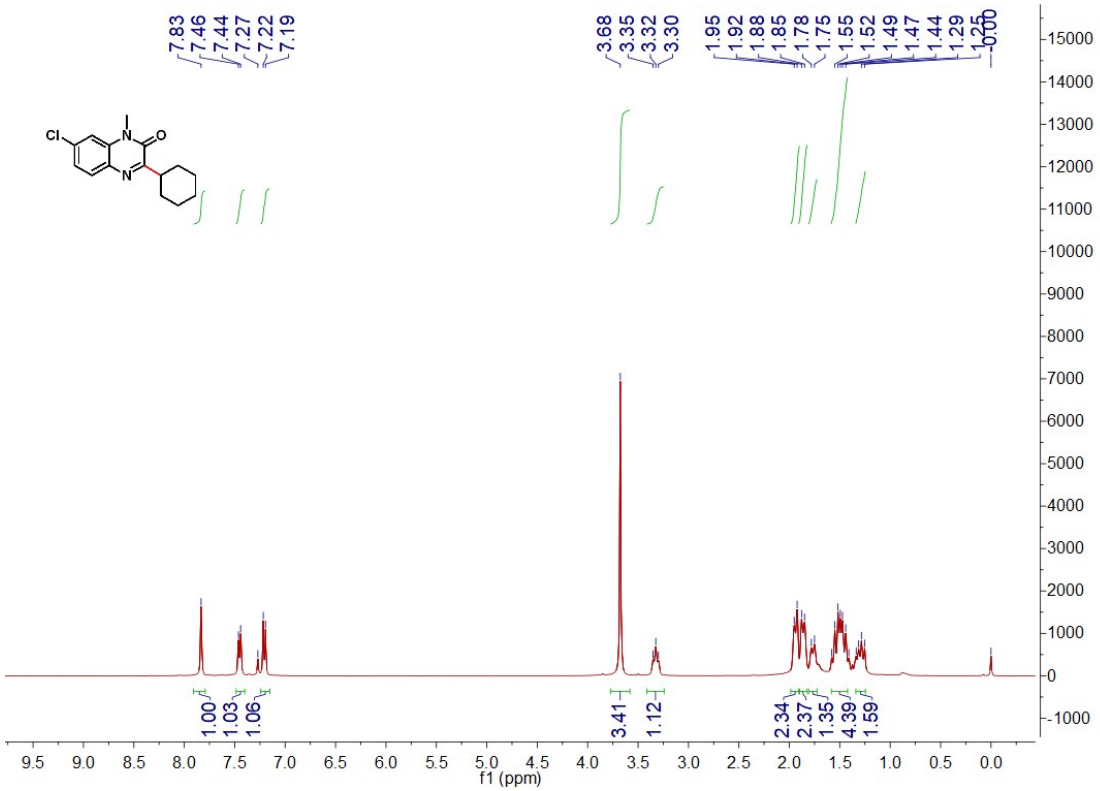
^{1}H NMR spectrum of compound **3ca**



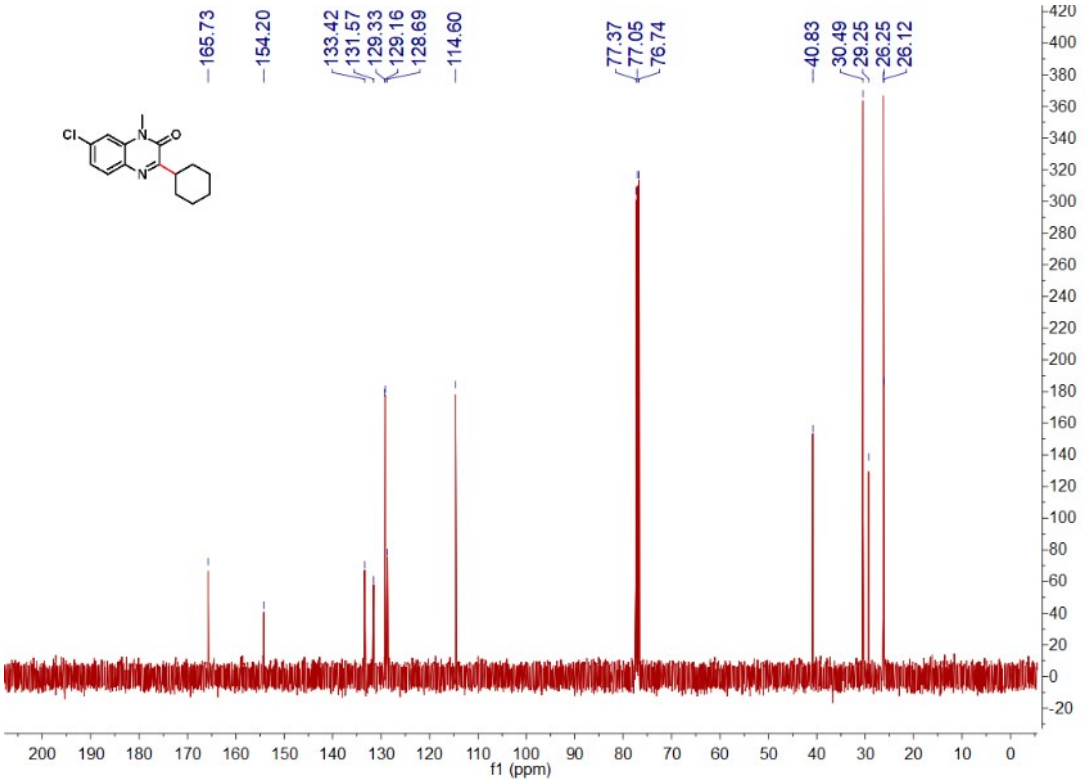
^{13}C NMR spectrum of compound **3ca**



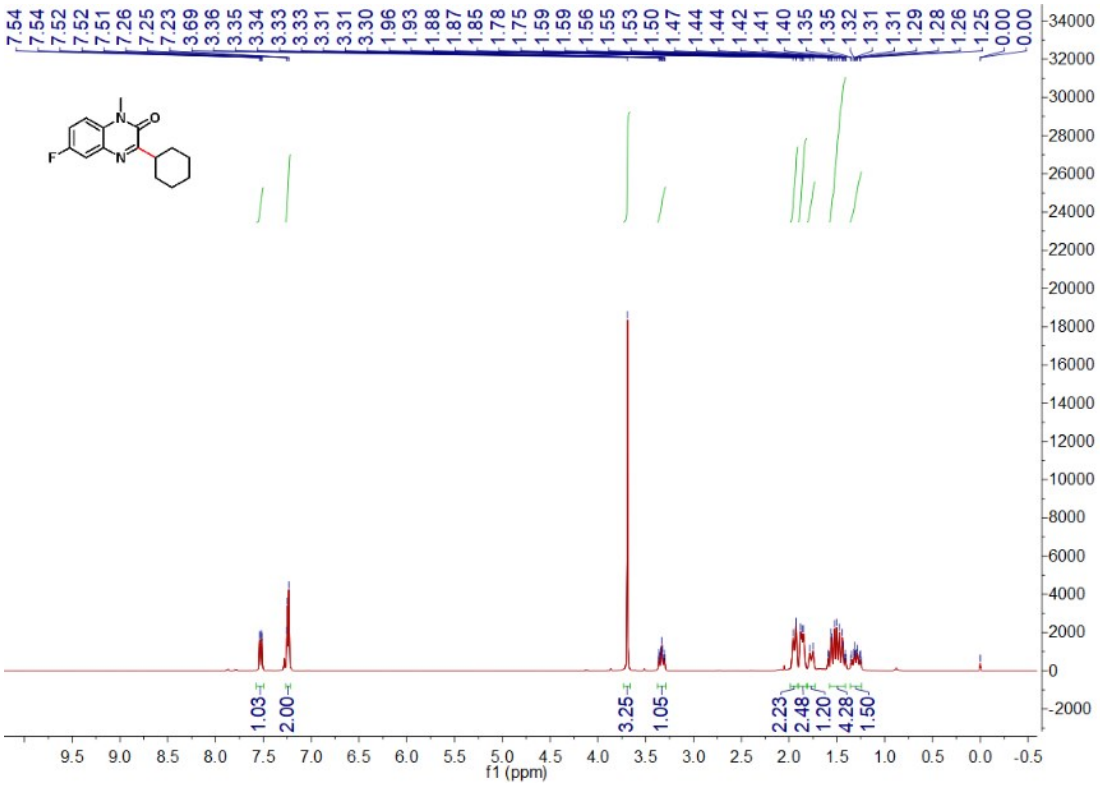
^{1}H NMR spectrum of compound **3da**



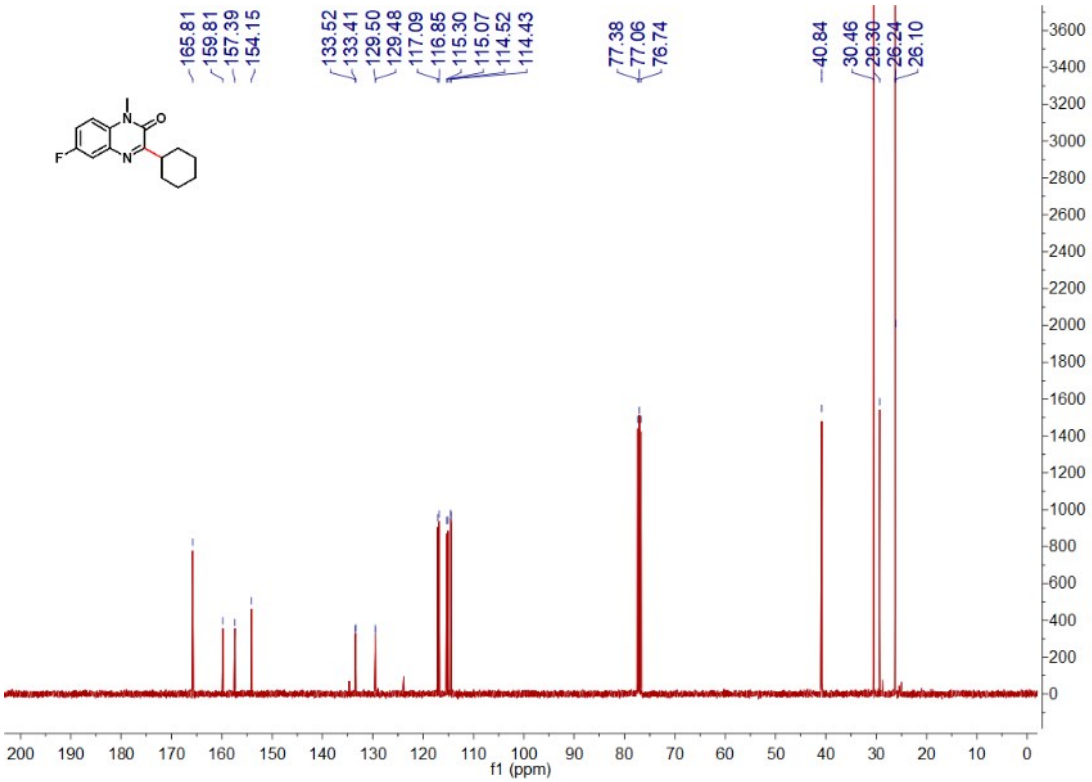
^{13}C NMR spectrum of compound **3da**



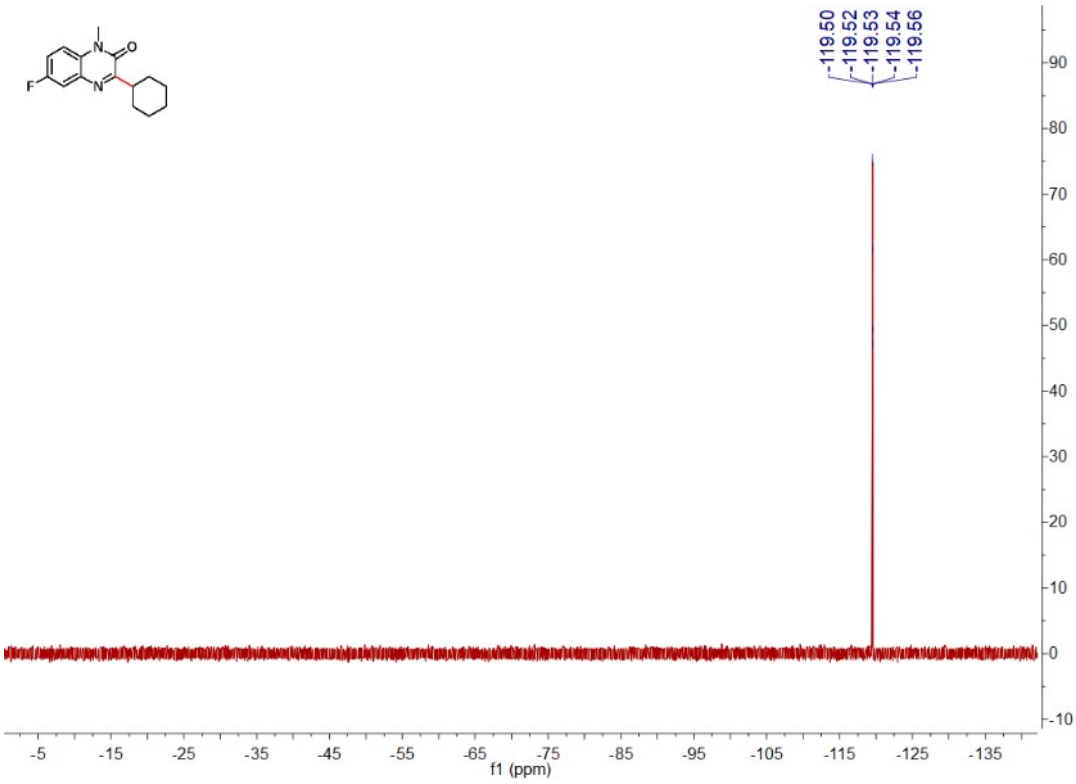
^{1}H NMR spectrum of compound **3ea**



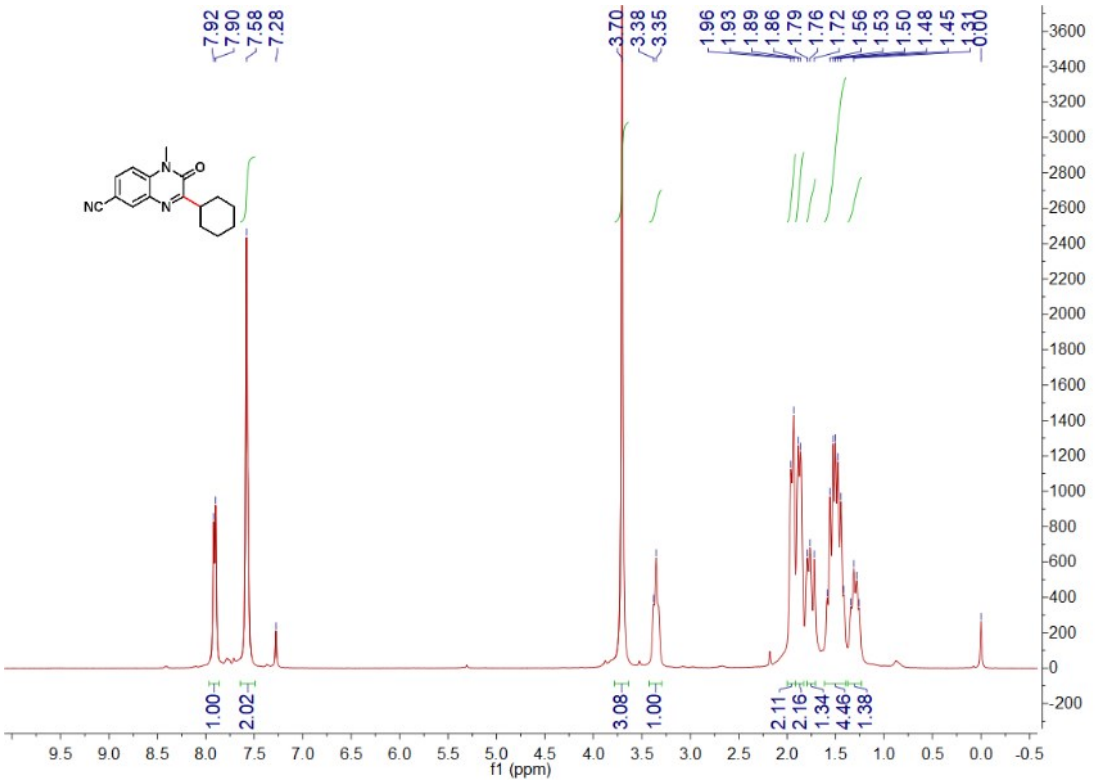
^{13}C NMR spectrum of compound **3ea**



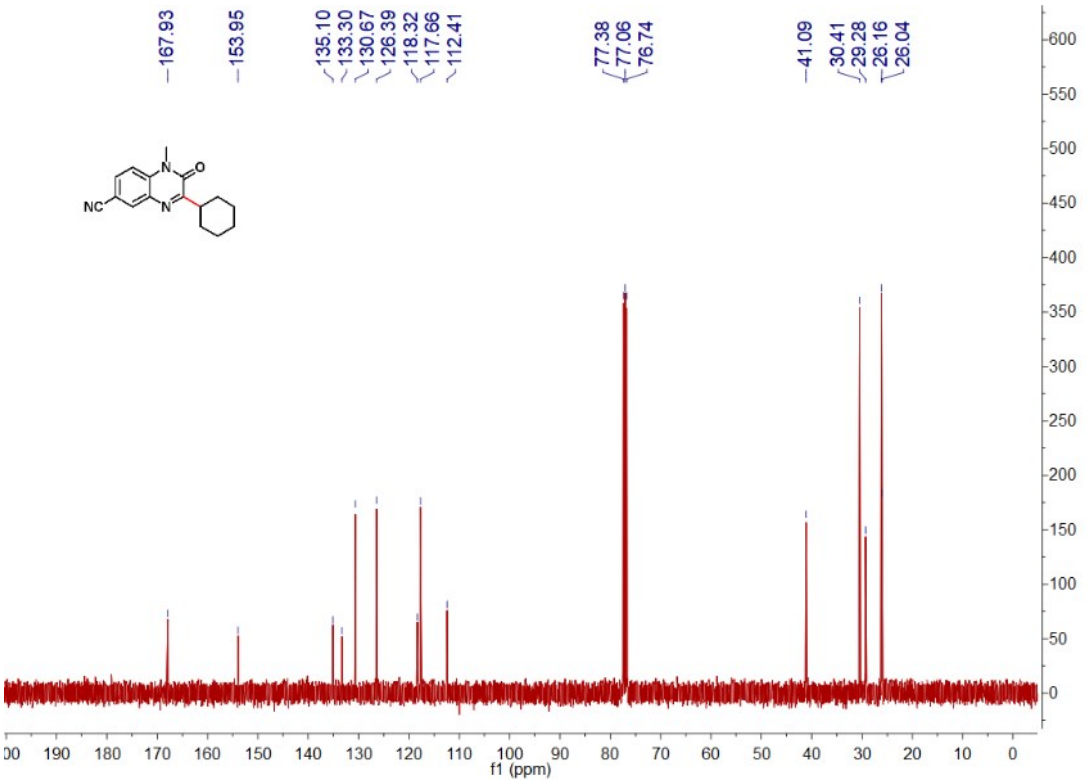
^{19}F NMR spectrum of compound **3ea**



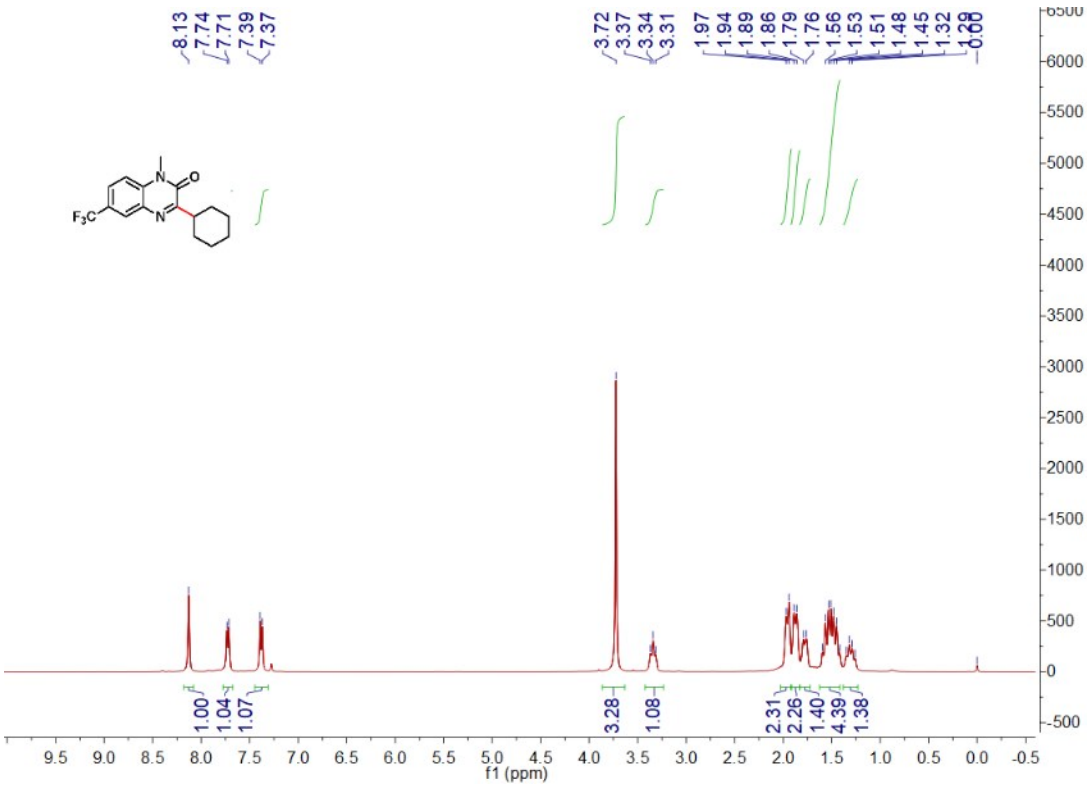
^{1}H NMR spectrum of compound **3fa**



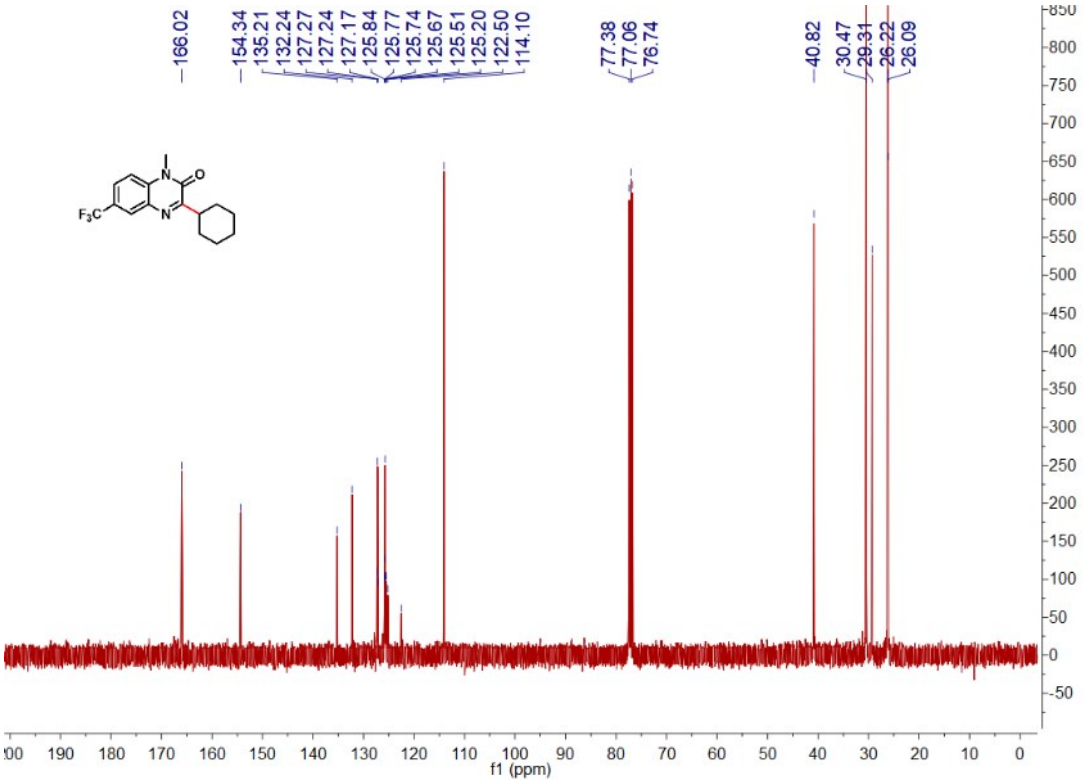
^{13}C NMR spectrum of compound **3fa**



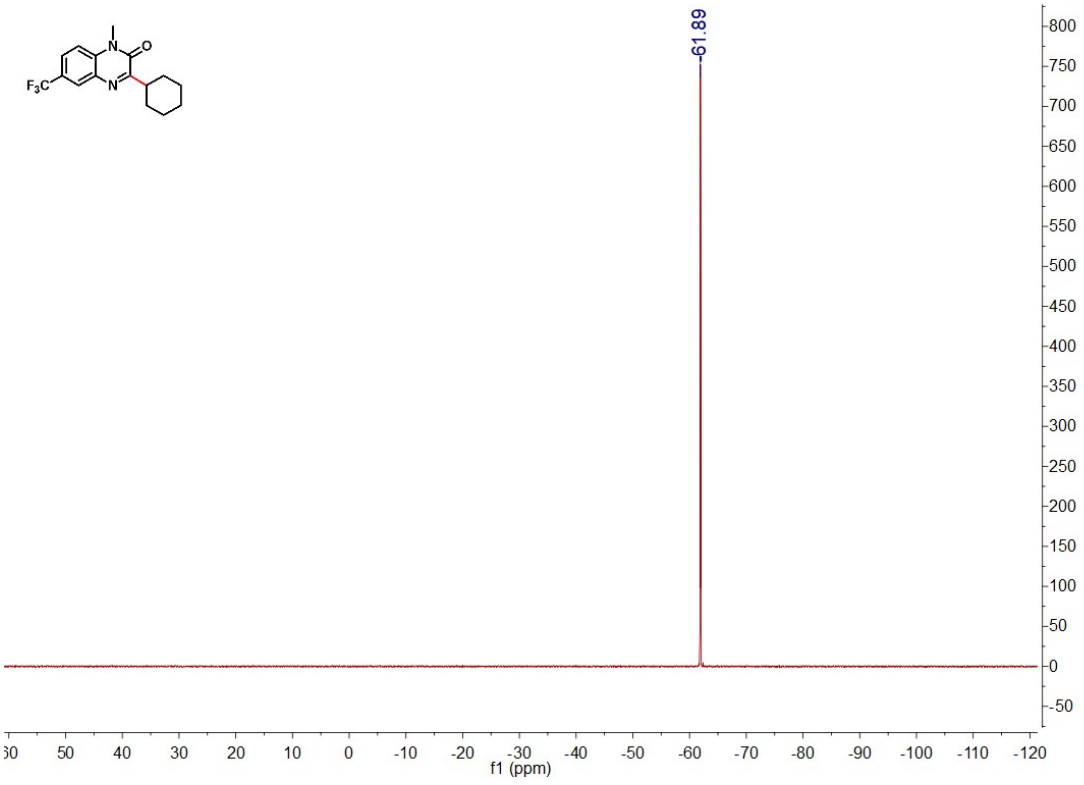
^{1}H NMR spectrum of compound **3ga**



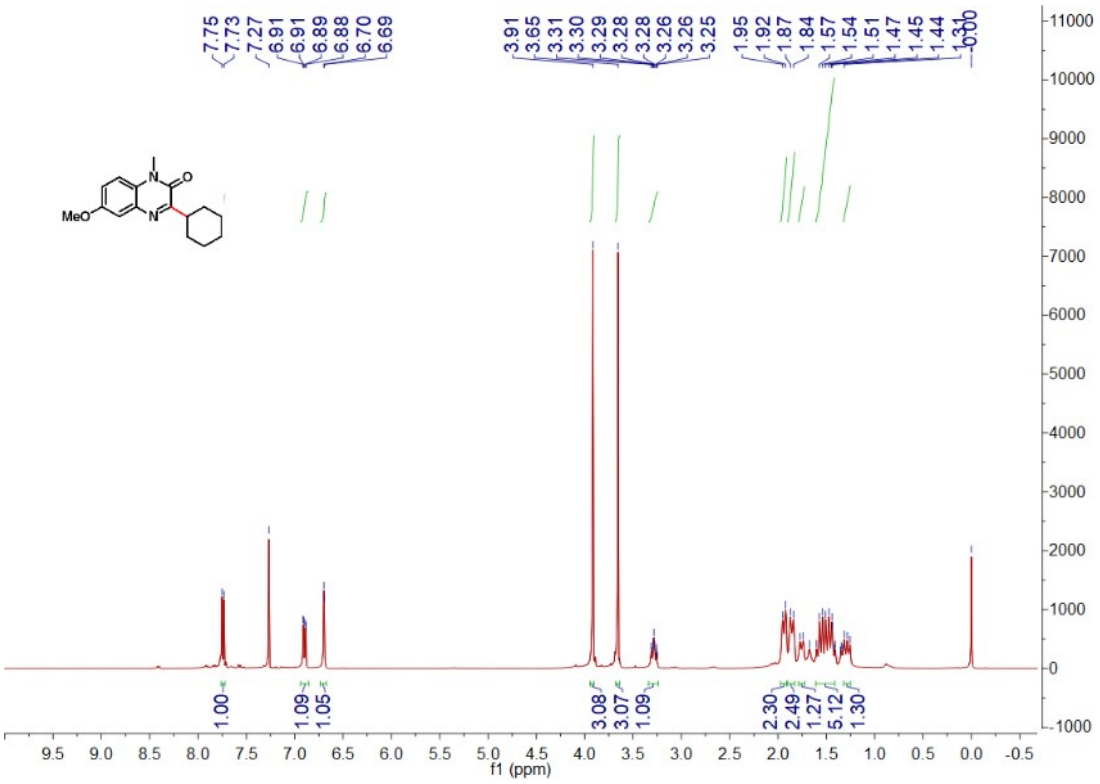
^{13}C NMR spectrum of compound **3ga**



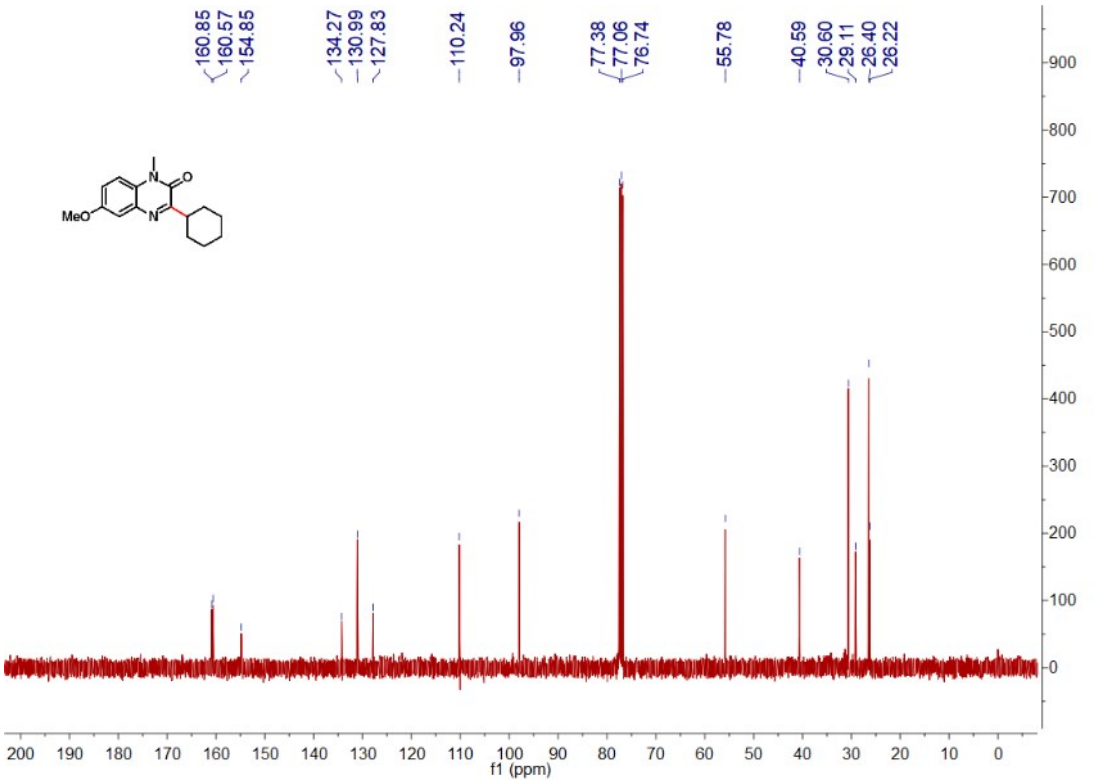
^{19}F NMR spectrum of compound **3ga**



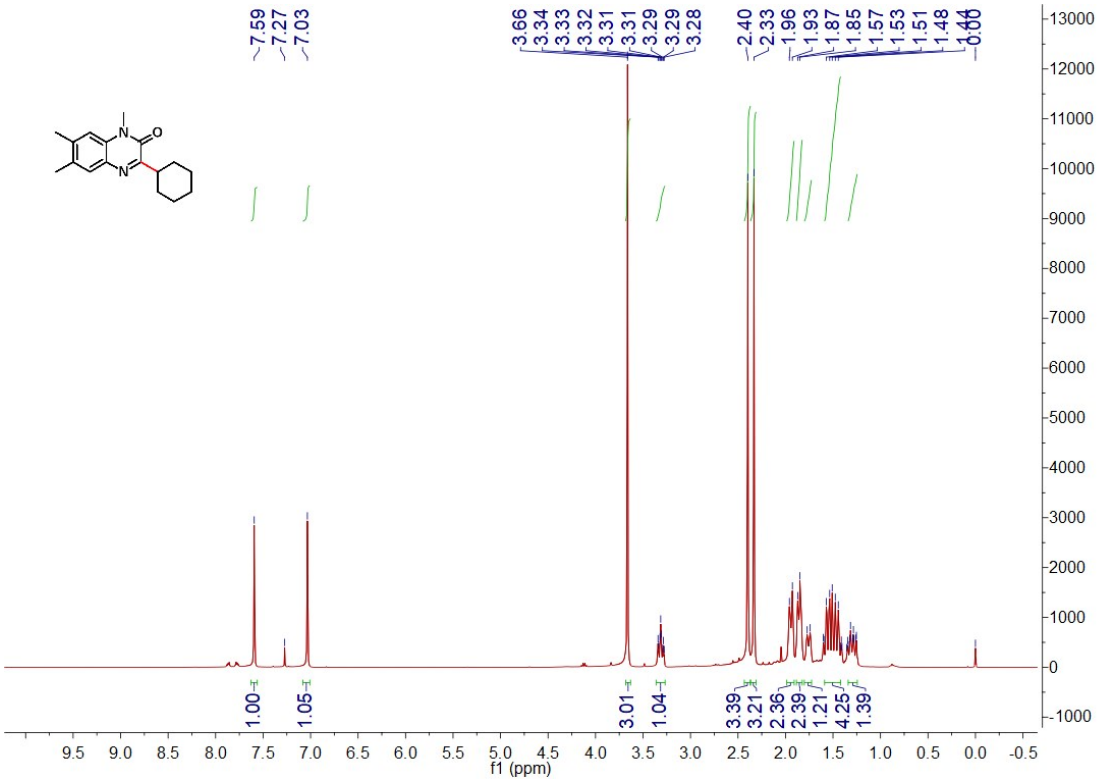
^{1}H NMR spectrum of compound **3ha**



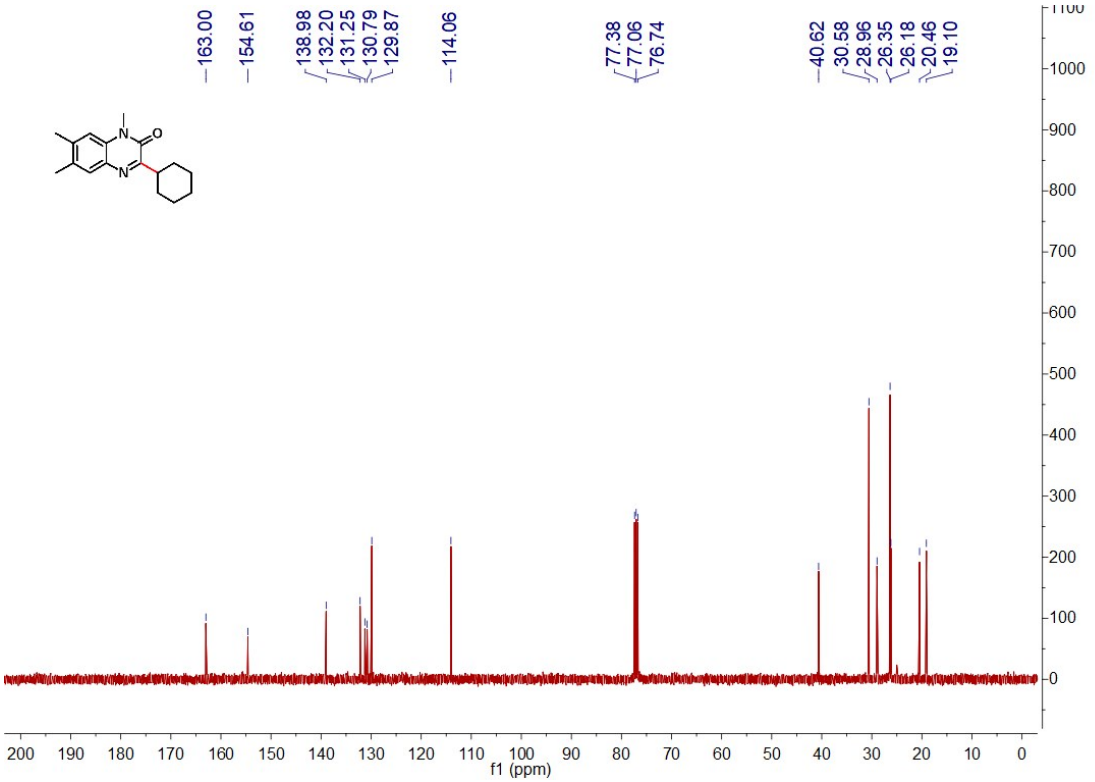
^{13}C NMR spectrum of compound **3ha**



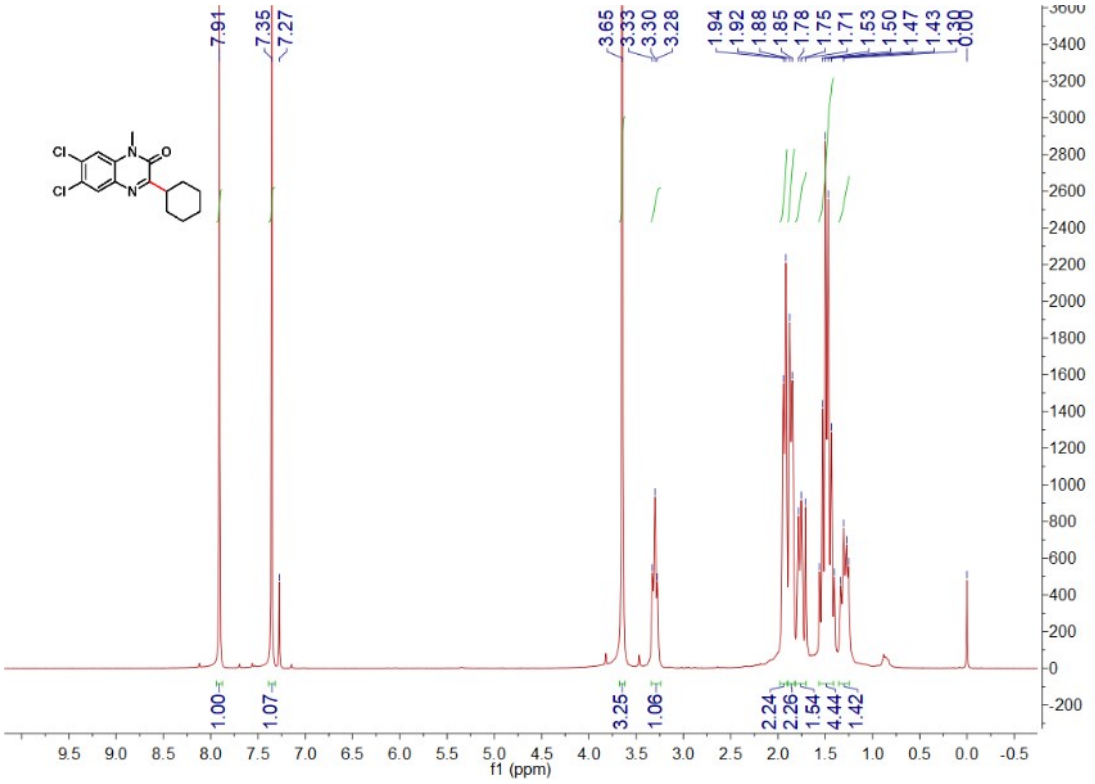
^{1}H NMR spectrum of compound **3ia**



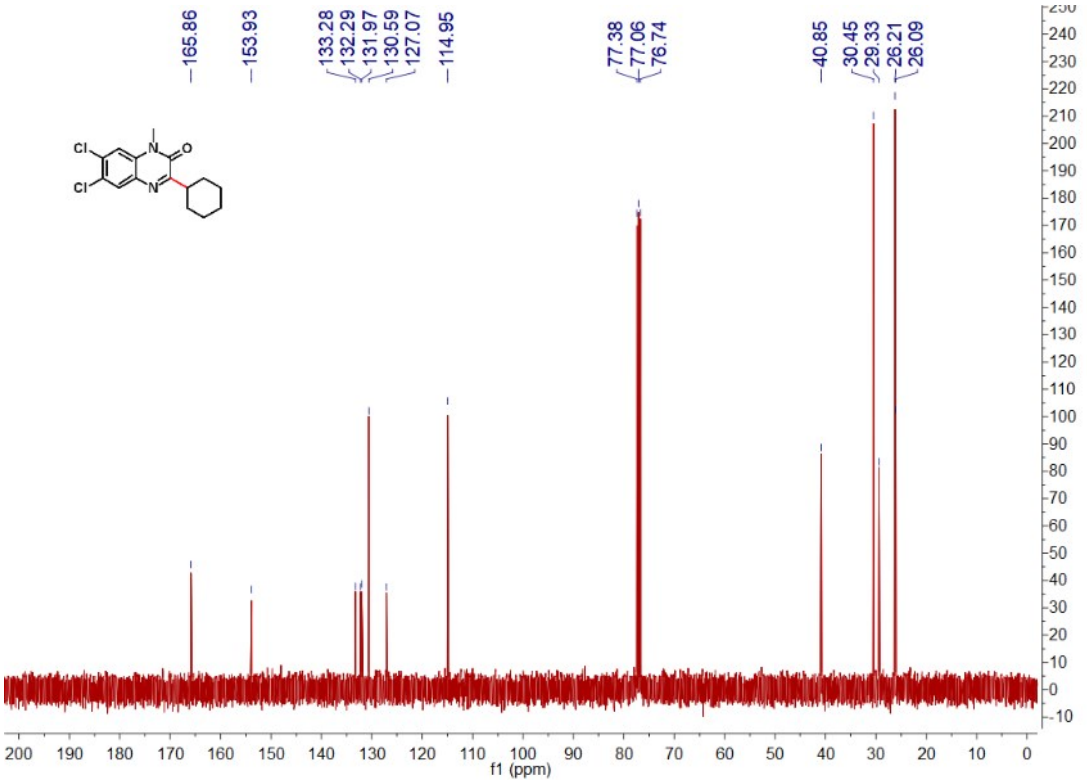
^{13}C NMR spectrum of compound **3ia**



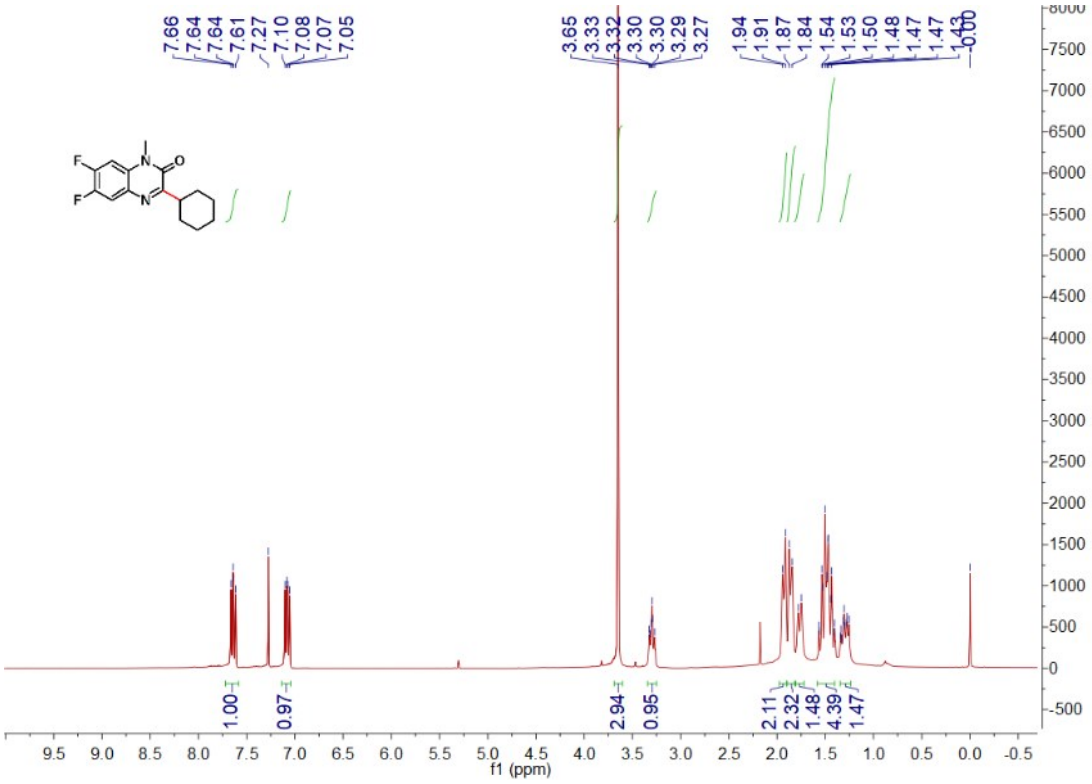
^{1}H NMR spectrum of compound **3ja**



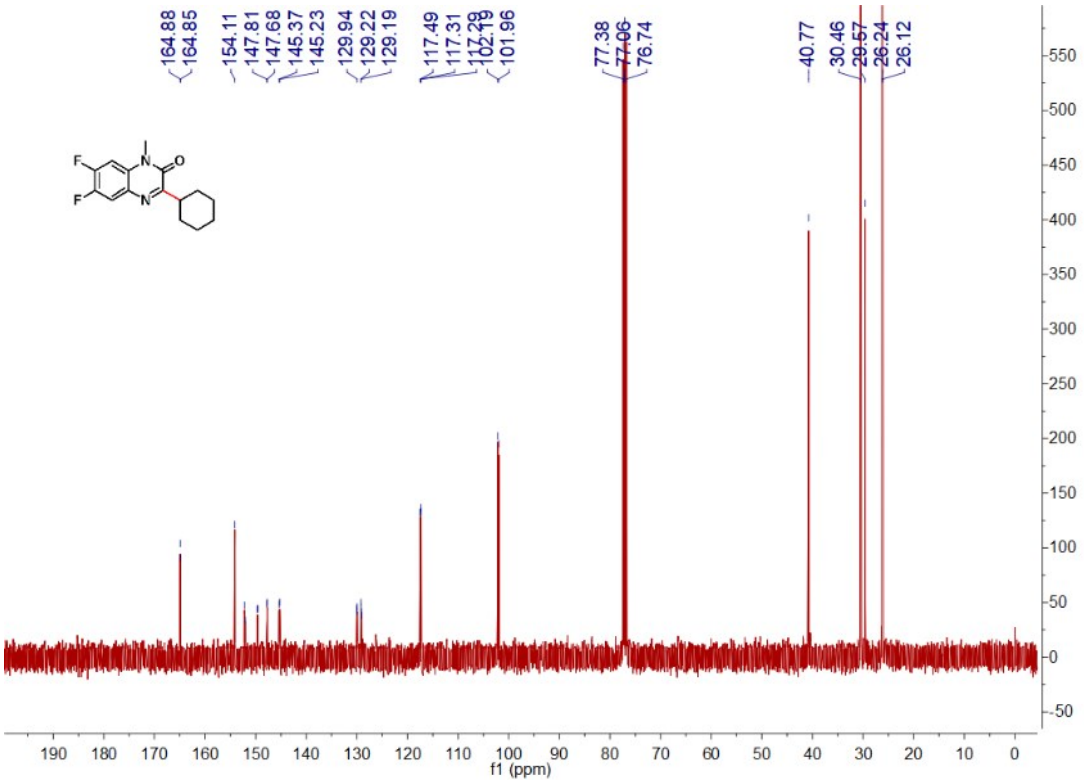
^{13}C NMR spectrum of compound **3ja**



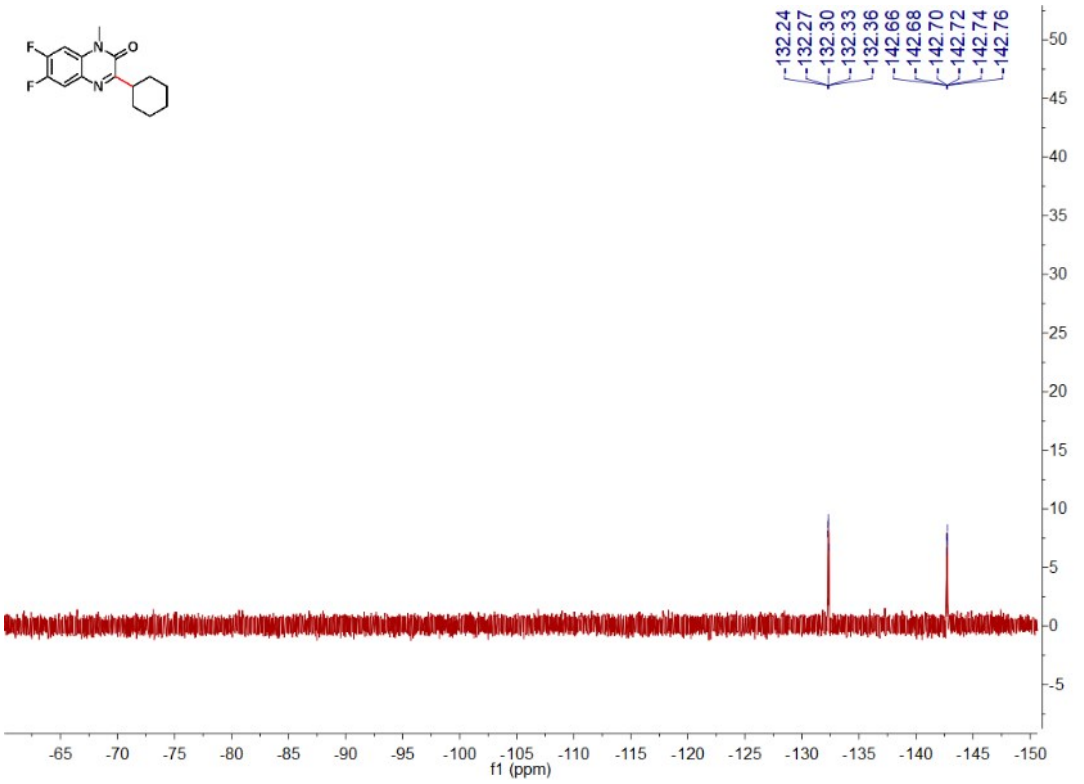
^{1}H NMR spectrum of compound **3ka**



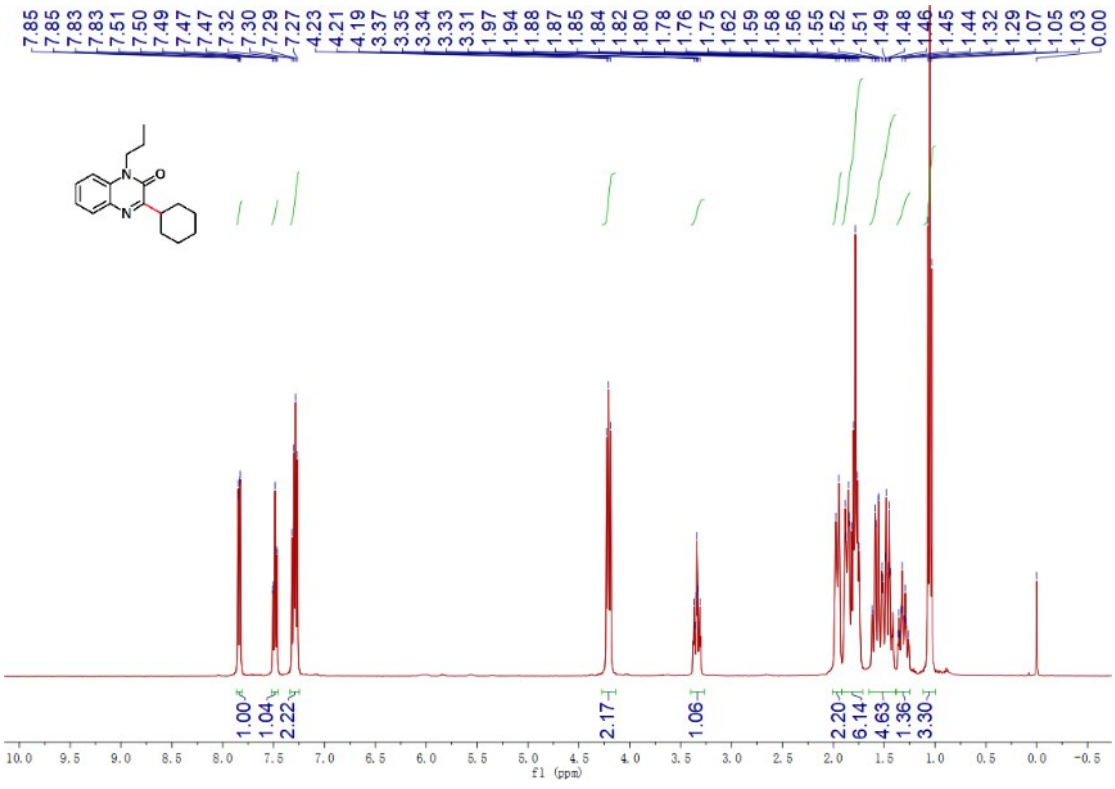
^{13}C NMR spectrum of compound **3ka**



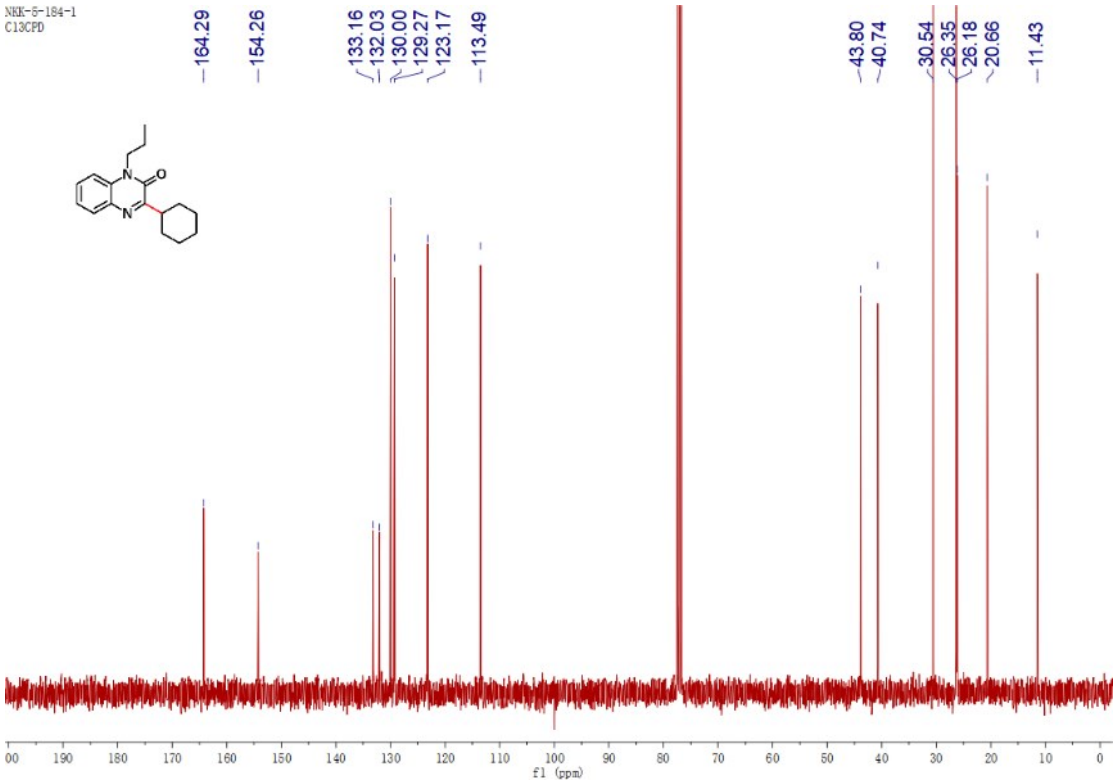
^{19}F NMR spectrum of compound **3ka**



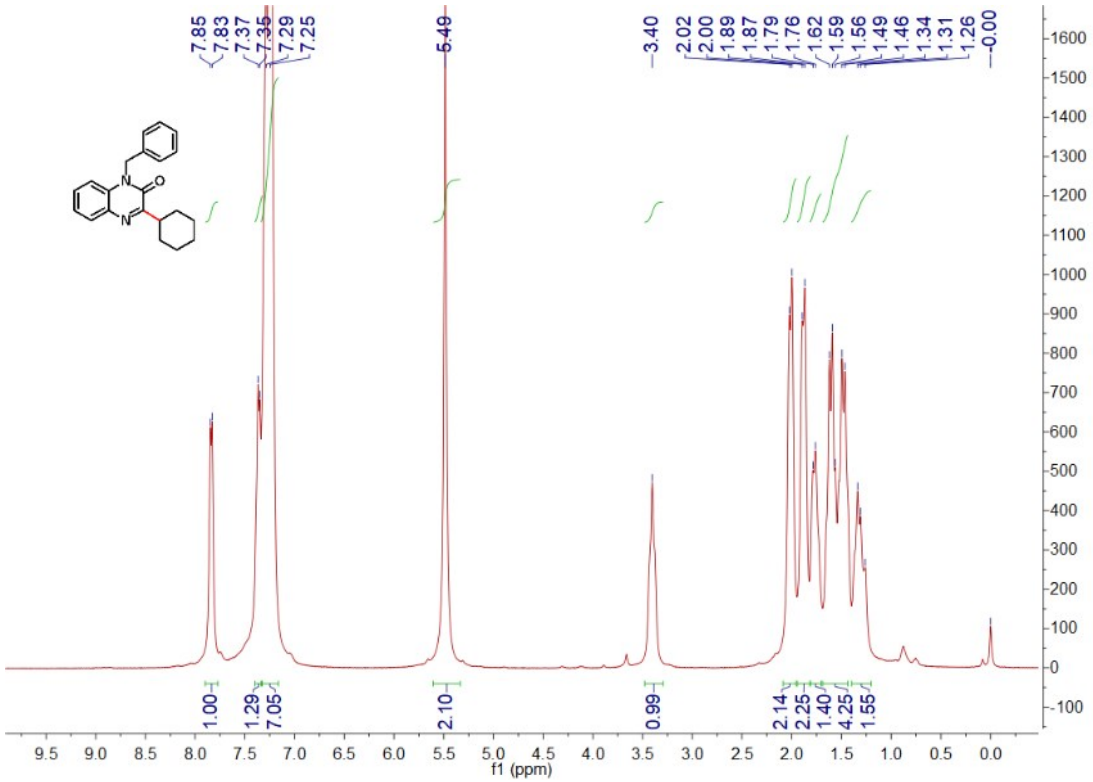
^{1}H NMR spectrum of compound **3la**



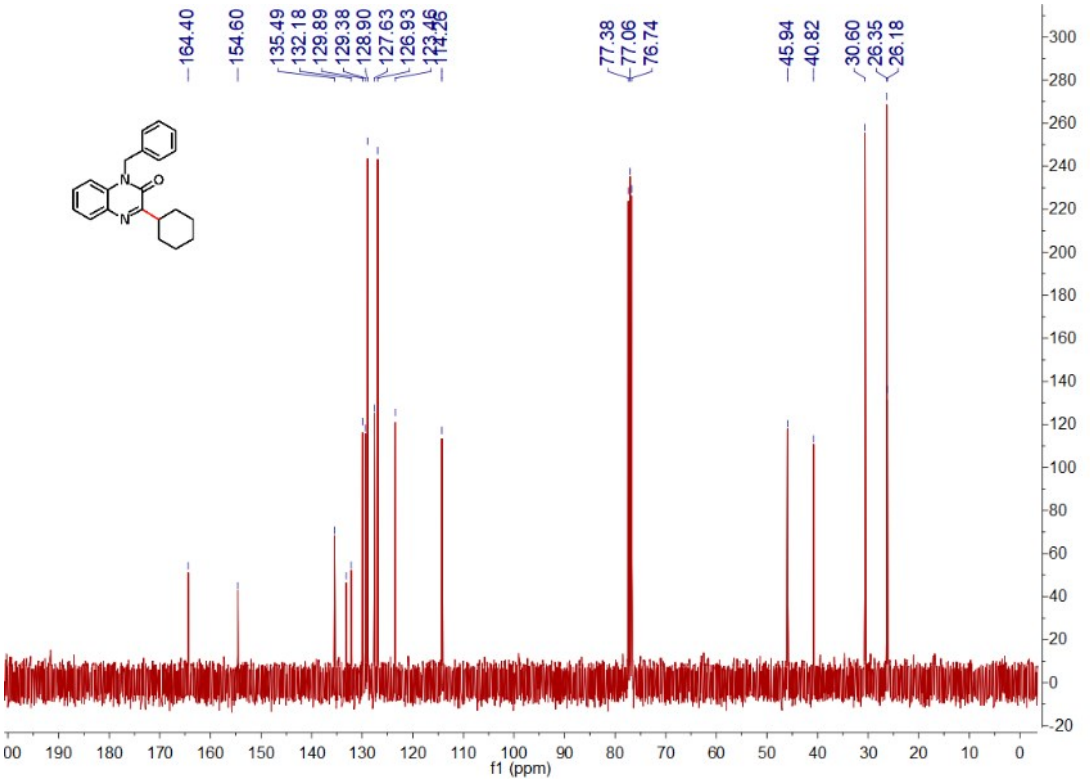
^{13}C NMR spectrum of compound **3la**



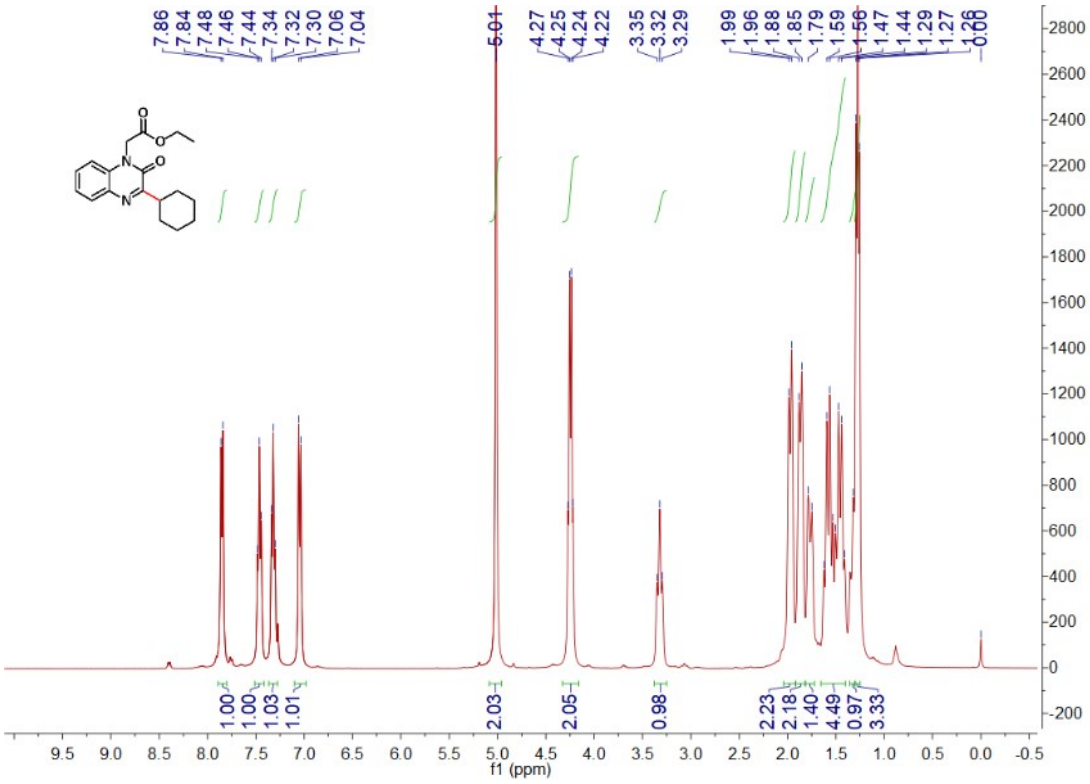
^{1}H NMR spectrum of compound **3ma**



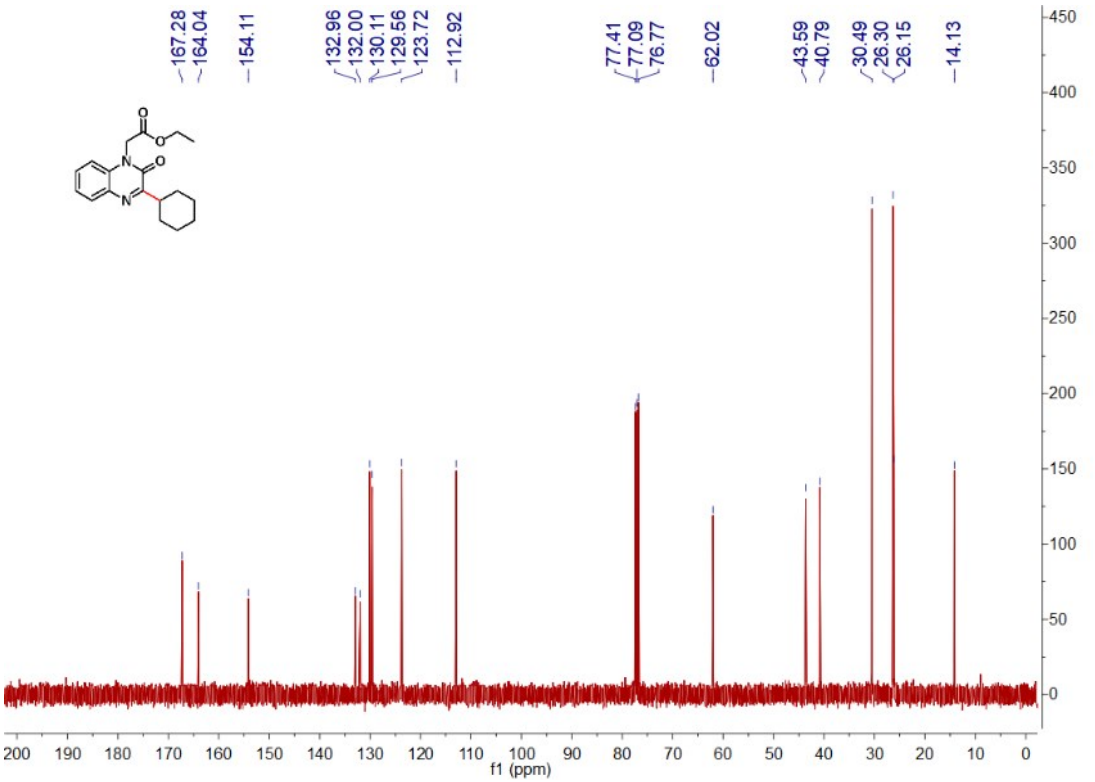
^{13}C NMR spectrum of compound **3ma**



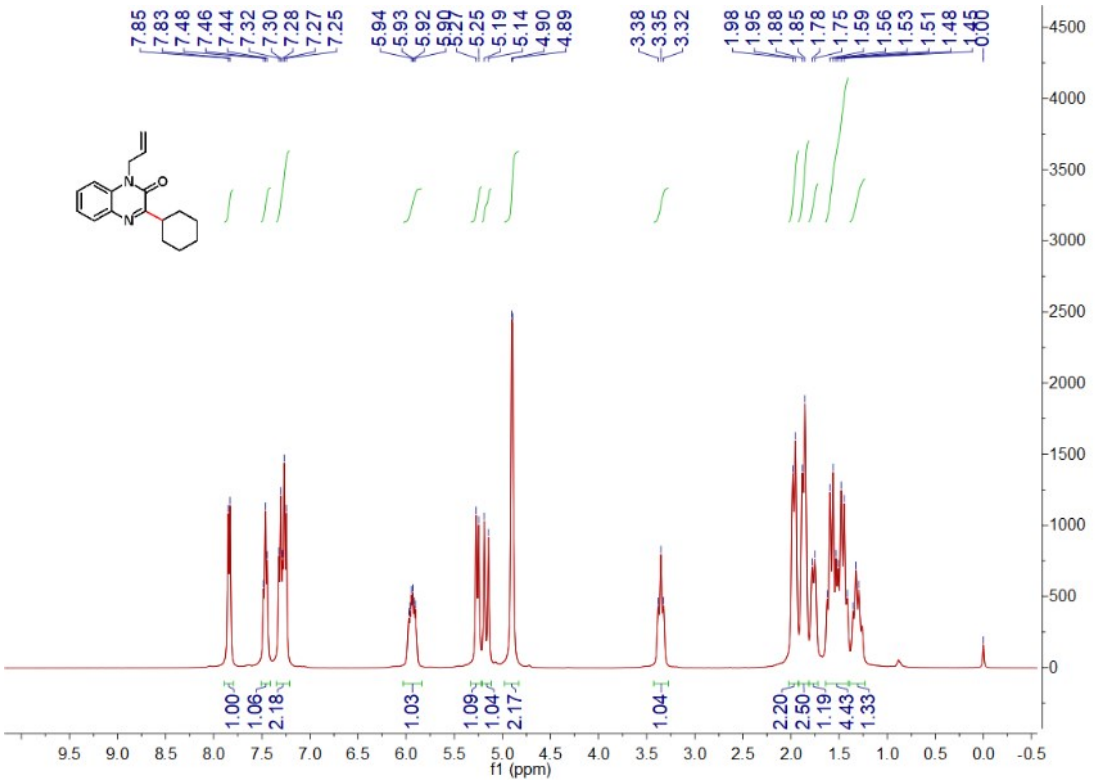
^{1}H NMR spectrum of compound **3na**



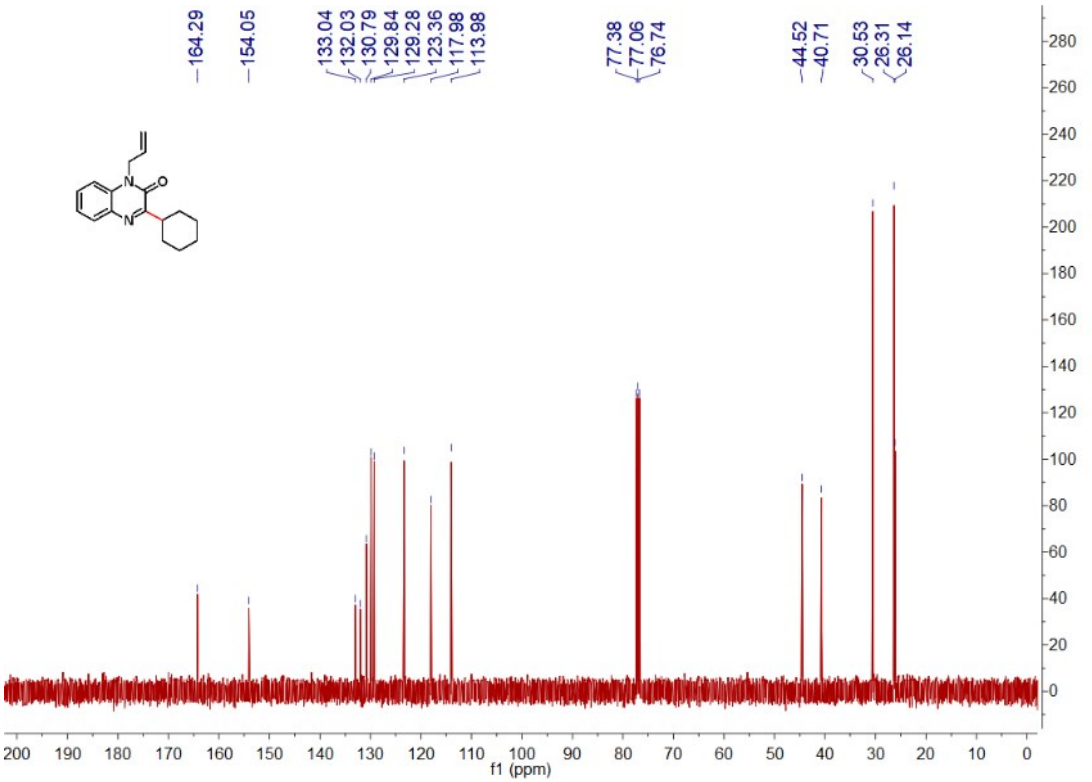
^{13}C NMR spectrum of compound **3na**



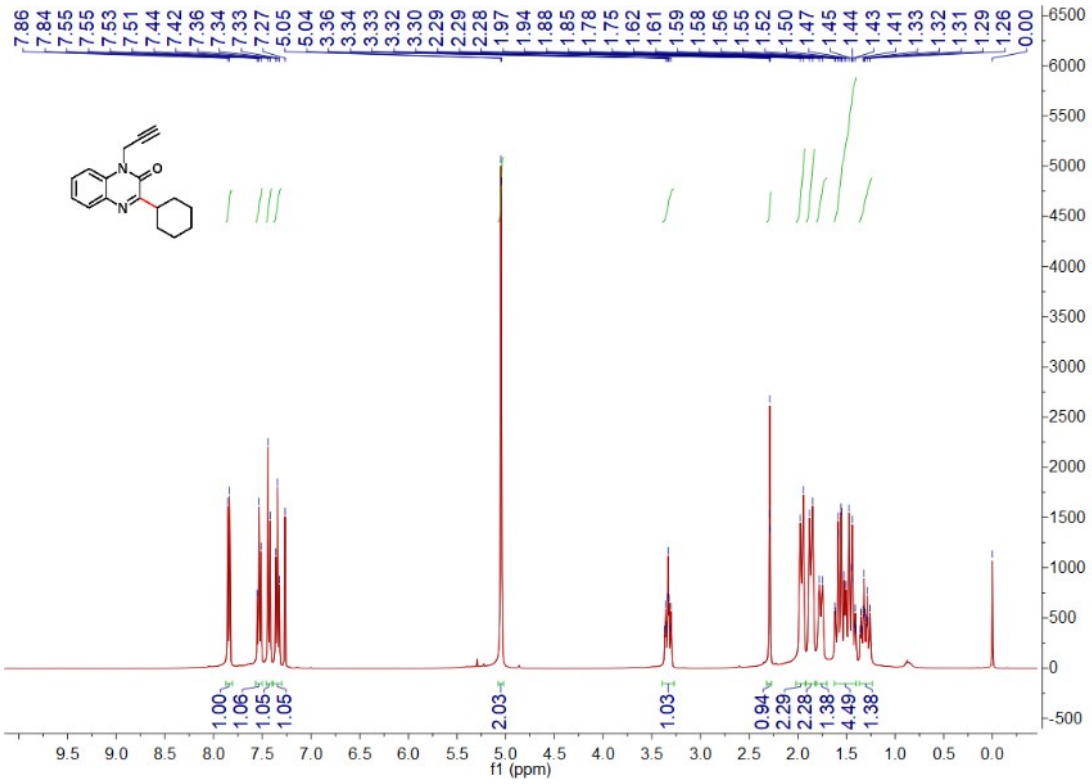
^{1}H NMR spectrum of compound **3oa**



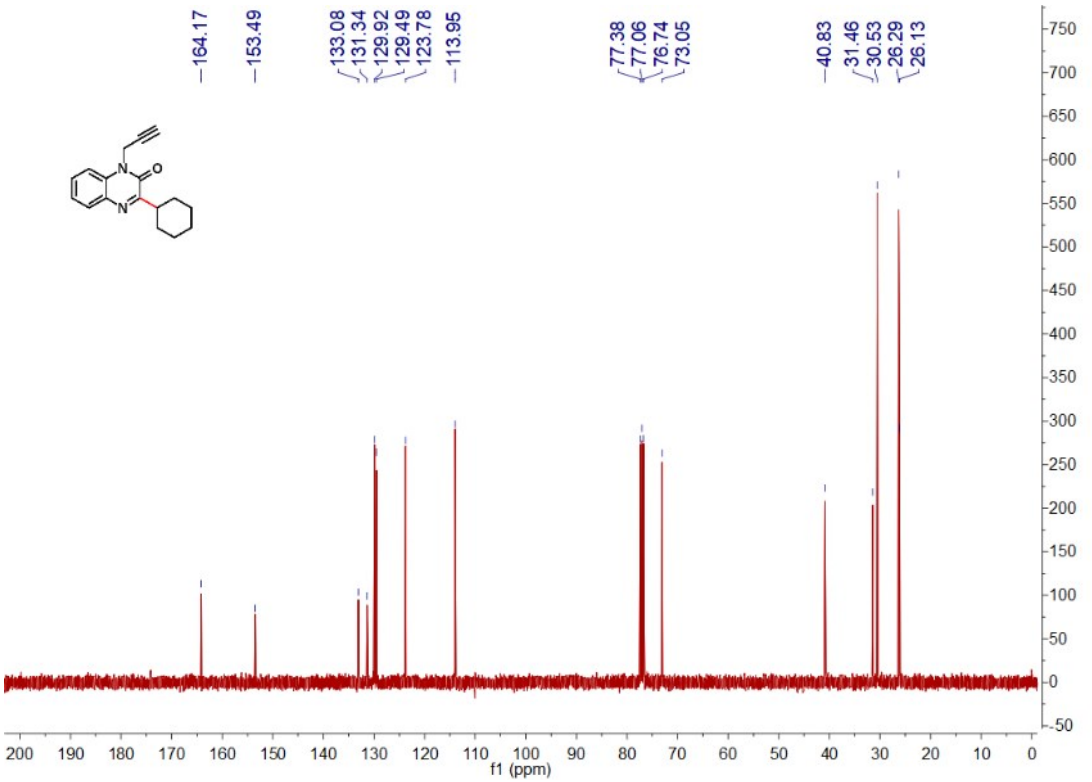
^{13}C NMR spectrum of compound **3oa**



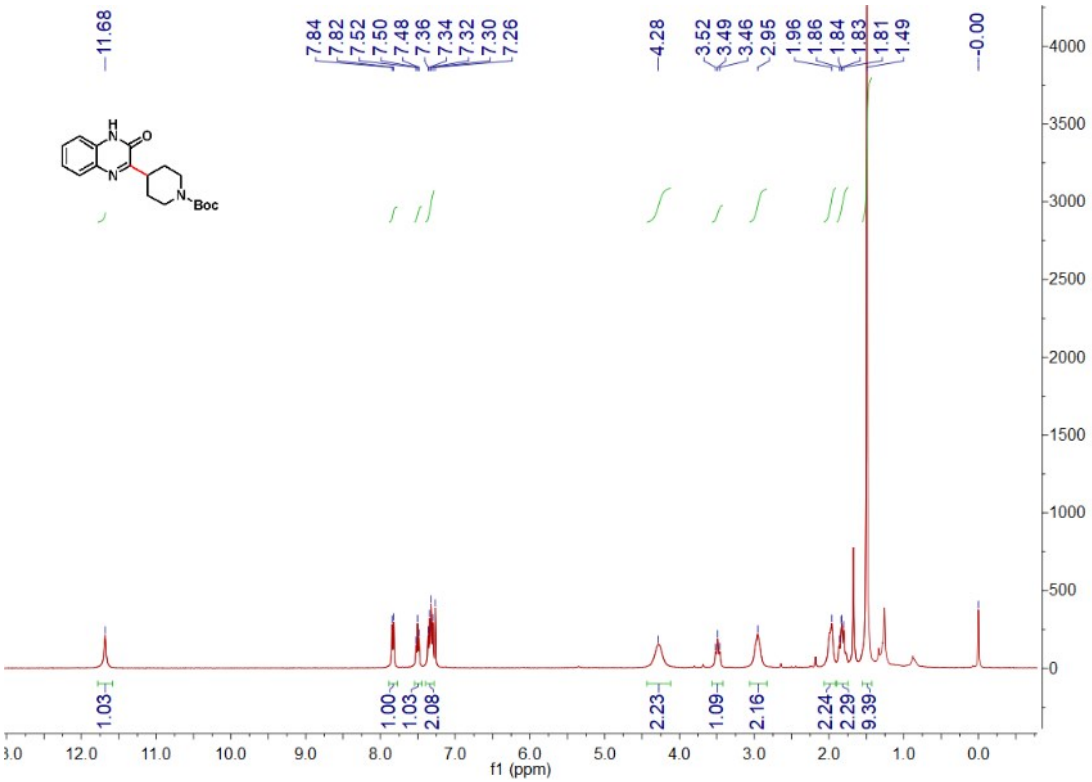
^{1}H NMR spectrum of compound **3pa**



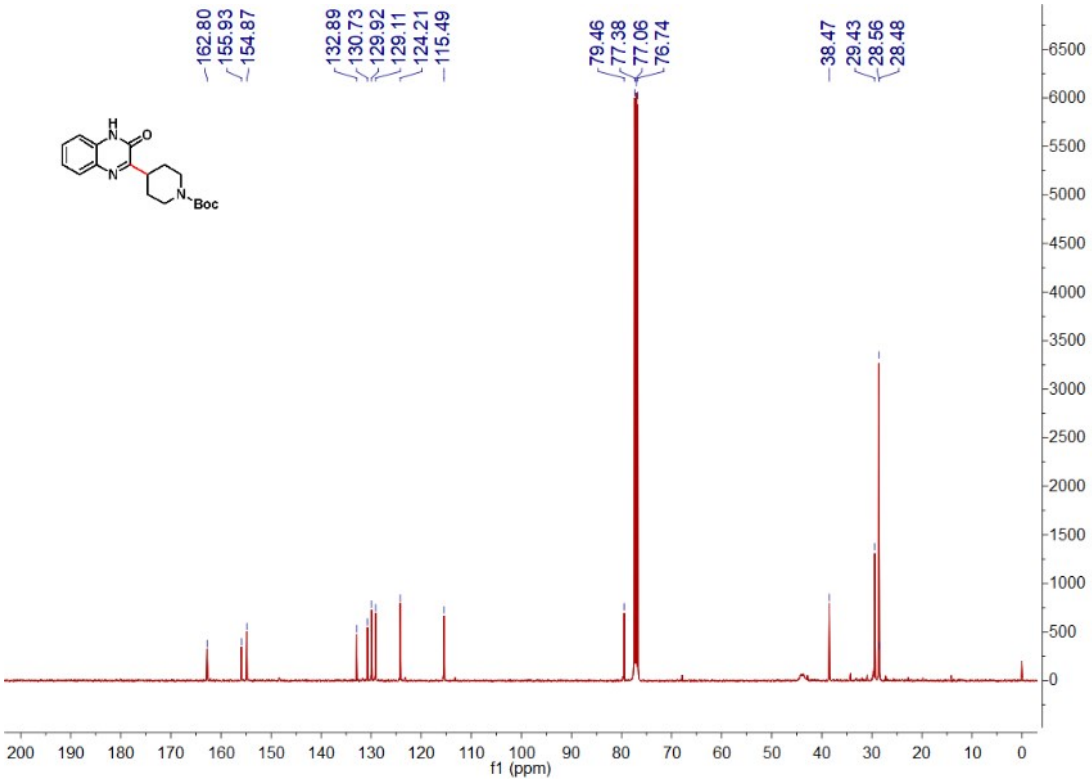
^{13}C NMR spectrum of compound **3pa**



^{1}H NMR spectrum of compound **3qa**



^{13}C NMR spectrum of compound **3qa**



12. Reference

1. J. Cornella, J. T. Edwards, T. Qin, S. Kawamura, J. Wang, C. M. Pan, R. Gianatassio, M. Schmidt, M. D. Eastgate, P. S. Baran, *J. Am. Chem. Soc.* **2016,** *138*, 2174-7.

2. M. Gao, Y. Li, L. Xie, R. Chauvin, X. Cui, *Chem. Commun.* **2016,** *52*, 2846-9.