Electrooxidative C-H Alkylation of Quinoxalin-2(1*H*)-ones with Organoboron Compounds

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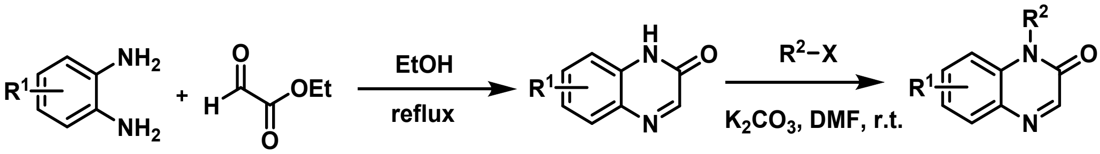
Supporting Information

1. General Information

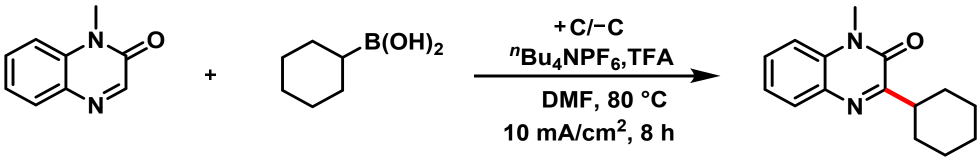
Unless otherwise noted, Reagents were purchased from commercial sources and were used as received. ^{1}H and ^{13}C Nuclear Magnetic Resonance (NMR) spectra were recorded on Bruker Avance 400 Ultrashield NMR spectrometers. Chemical shifts (δ) were given in parts per million (ppm) and were measured downfield 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 quinoxalin-2(1*H*)-one



Quinoxalin-2(1*H*)-one was prepared from 1,2-phenylenediamines following the procedure of Cui and co-workers [1] 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.

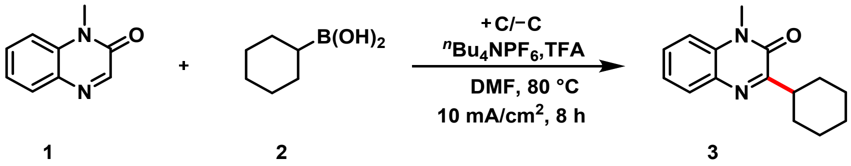
3. General procedure for the electrolysis



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 cyclohexylboronic acid (192.0 mg, 1.5 mmol, 3.0 equiv.), the trifluoroacetic acid (150 μL, 2.0 mmol, 4.0 equiv.) and $^{n}Bu_4NPF_6$ (387.4 mg, 1.0 mmol, 2.0 equiv.) was added into the undivided cell. And then DMF (5 mL) was added. The reaction mixture was stirred and electrolyzed at a constant current of 10.0 mA under 80 °C for 8 h. The reaction was quenched with saturated sodium carbonate (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).

4. Optimization Studies

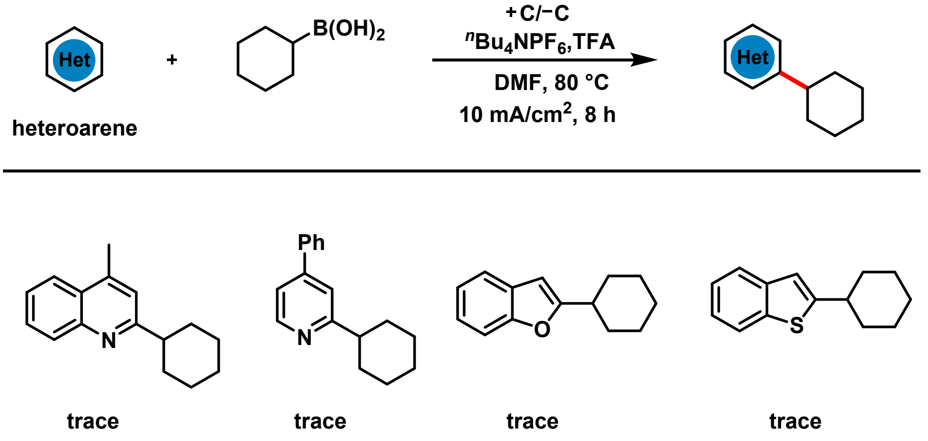
Optimization of reaction conditions[a]



Entry	Equivalent of 2	Solvent	Yield[b]
1	2.0	MeCN	62
2	2.0	H_2O	39
3	2.0	MeCN-H_2O/10:1	43
4	2.0	EtOH	36
5	2.0	THF	45
6	2.0	1,4-dioxane	19
7	2.0	DMF	80
8	3.0	DMF	87
9	4.0	DMF	88
10	5.0	DMF	87

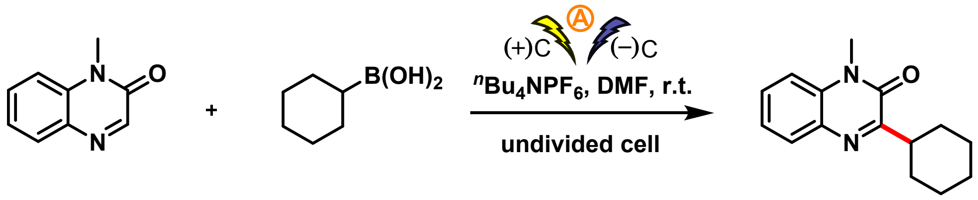
[a]Reaction conditions: quinoxalin-2(1*H*)-one (0.5 mmol), **2** (1.5 mmol), ^{n}Bu$_4$NPF$_6$ (2.0 equiv), TFA (4.0 equiv), DMF (5 mL), undivided cell with two graphite electrodes (each 1.0 × 1.0 cm^2), 80 °C, 10 mA/cm^2, 8 h. [b]Yield determined by ^{1}H-NMR analysis using dibromomethane as the internal standard.

5. Details of Unsuccessful Substrates



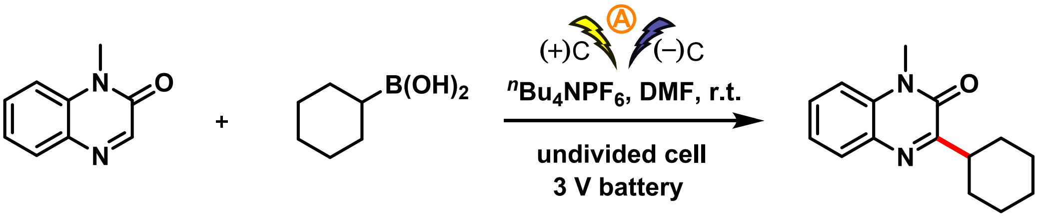
Reaction conditions: heteroarene (0.5 mmol), **2** (1.5 mmol), ^{n}Bu$_4$NPF$_6$ (2.0 equiv), TFA (4.0 equiv), DMF (5 mL), undivided cell with two graphite electrodes (each 1.0 × 1.0 cm^2), 80 °C, 10 mA/cm^2, 8 h. Isolated yield.

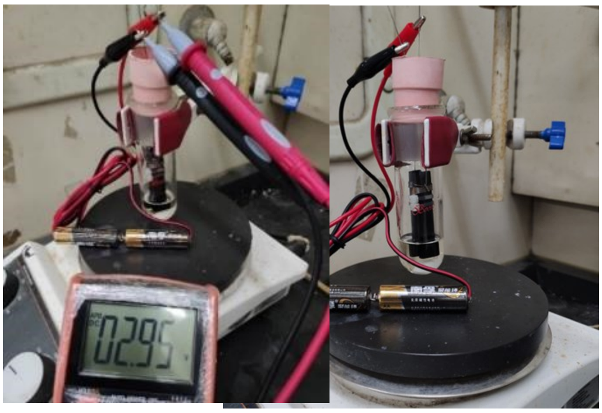
6. Gram-scale Reaction



A 200 mL bottle with a stir bar was charged with quinoxalin-2(1*H*)-one (0.80 g, 5.0 mmol, 1.0 equiv.), cyclohexylboronic acid (1.92 g, 15 mmol, 3.0 equiv.) trifluoroacetic acid (1.5 mL, 20 mmol, 4.0 equiv.), ^{n}Bu$_4$NPF$_6$ (3.87 g, 10 mmol, 2.0 equiv.) and DMF (100 mL). 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 60 mA under rome temperature for 36 h. After the reaction, the electrodes were removed and rinsed with EtOAc. Saturated sodium carbonate was then added and the resulting mixture was extracted with EtOAc. The combined organic layer was dried over anhydrous Na$_2$SO$_4$ and concentrated in vacuo. The crude material was purified by column chromatography (petroleum ether/ethyl acetate = 10:1) to furnish the desired product in 79% yield as a white solid.

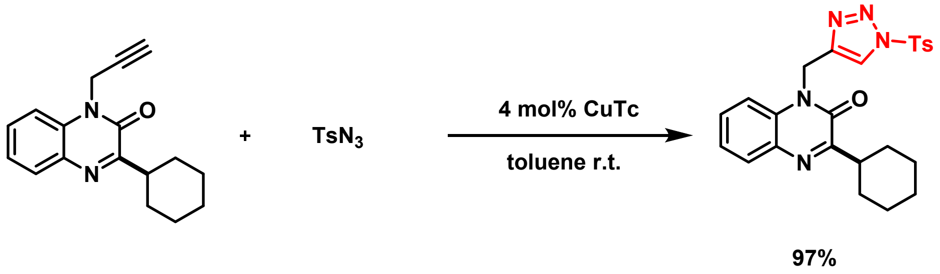
7. Use of 3-V battery as power source





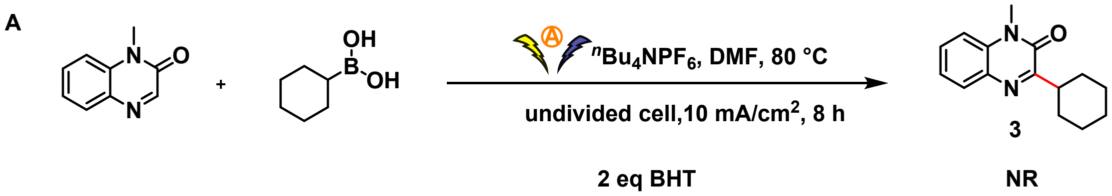
An oven-dried 10 mL undivided bottle was charged with quinoxalin-2(1*H*)-one (80.0 mg, 0.5 mmol, 1.0 equiv.), cyclohexylboronic acid (192.0 mg, 1.5 mmol, 3.0 equiv.), trifluoroacetic acid (150 μL, 2.0 mmol, 4.0 equiv.), ^{n}Bu$_4$NPF$_6$ (387.4 mg, 1.0 mmol, 2.0 equiv.) and DMF (5 mL). Two graphite sheet 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. The reaction mixture was electrolyzed under a constant current of 10 mA under rome temperature for 24 h. After the reaction, the resulting mixture was was quenched with saturated sodium carbonate 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 63% yield.

8. Click reaction



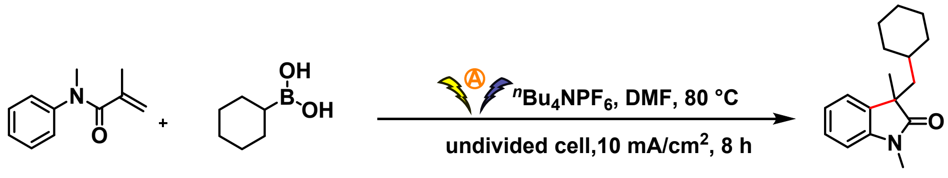
A solution of **14** (53.3 mg, 0.2 mmol) in toluene was added CuTc (1.6 mg, 0.008 mmol), then the mixture was stirred for 3 min at room temperature, followed by addition of TsN_3 (47.3 mg, 0.24 mmol) in one pot via syringe. The reaction mixture was stirred for 7 h at room temperature. The residue was purified by column chromatography (petroleum ether/EtOAc = 10/1) to afford **25** as white solid in 97% yield.

9. Mechanistic Studies



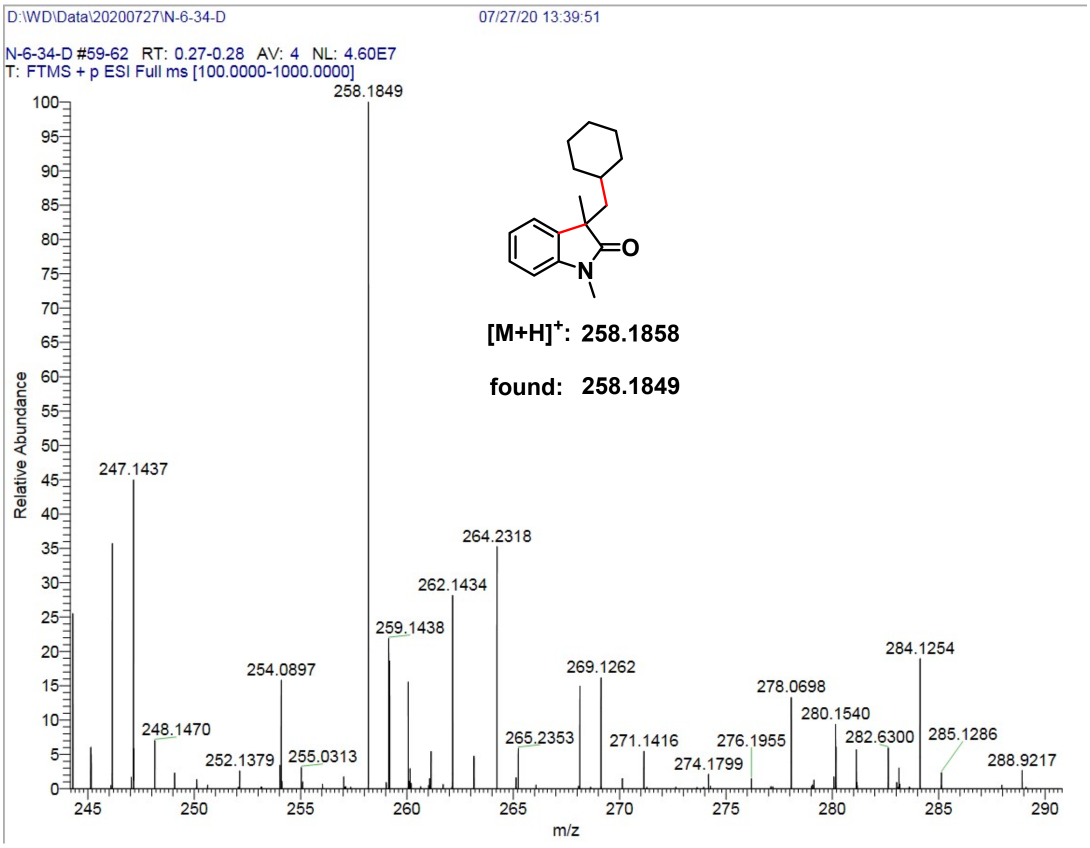
To a 10 mL oven-dried undivided bottle was added quinoxalin-2(1*H*)-one (80.0 mg, 0.5 mmol, 1.0 equiv.), cyclohexylboronic acid (192.0 mg, 1.5 mmol, 3.0 equiv.), BHT (220.4 mg, 1.0 mmol, 2.0 equiv.), trifluoroacetic acid (150 µL, 2.0 mmol, 4.0 equiv.), $^nBu_4NPF_6$ (387.4 mg, 1.0 mmol, 2.0 equiv.) and DMF (5 mL). The reaction mixture was stirred and electrolyzed at a constant current of 10.0 mA under 80 °C for 8 h. The reaction was completely suppressed.

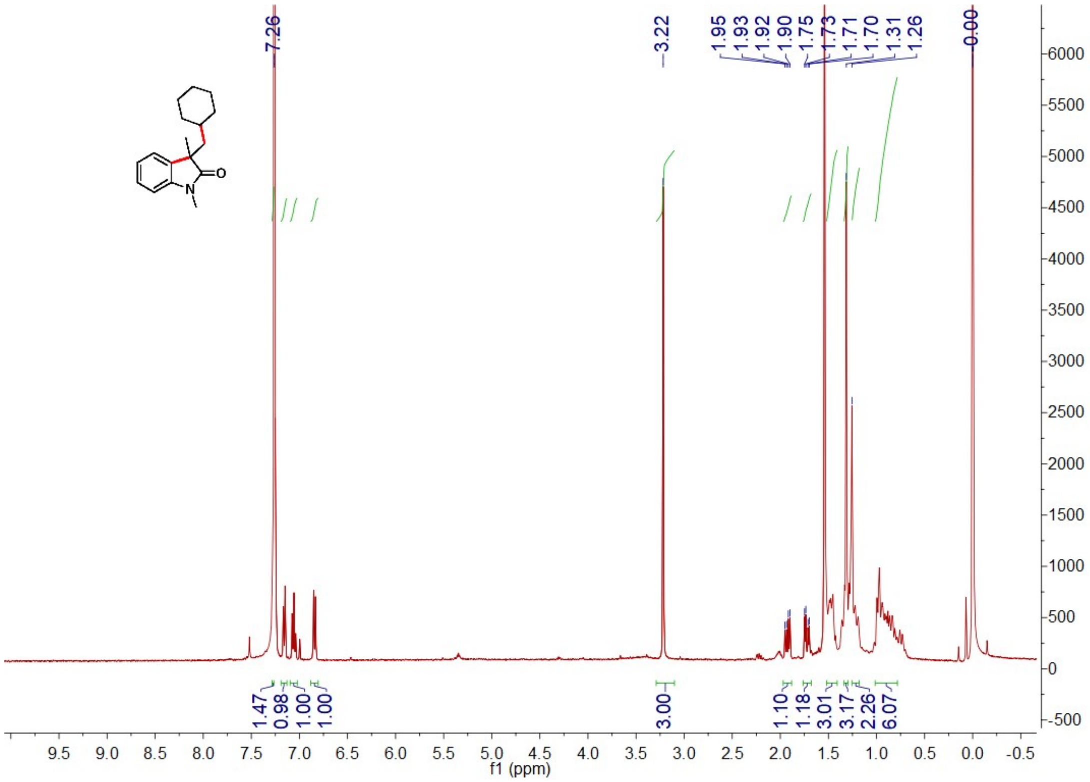
B



29, 8%

To a 10 mL oven-dried undivided bottle was added N-methyl-N-phenyl methacrylamide (87.6 mg, 0.5 mmol, 1.0 equiv.), cyclohexylboronic acid (192.0 mg, 1.5 mmol, 3.0 equiv.), trifluoroacetic acid (150 µL, 2.0 mmol, 4.0 equiv.), ^{n}Bu$_4$NPF$_6$ (387.4 mg, 1.0 mmol, 2.0 equiv.) and DMF (5 mL). The reaction mixture was stirred and electrolyzed at a constant current of 10.0 mA under 80 °C for 8 h. The product **27** can be observed by HR-MS (positive mode ESI). After the reaction, the resulting mixture was was quenched with saturated sodium carbonate and extracted with EtOAc. The combined organic layer was dried over anhydrous Na$_2$SO$_4$ and concentrated in vacuo. The crude material was purified by flash column chromatography (silica gel, 20% EtOAc in petroleum ether) to give **3** as a colorless oil (9.8 mg, 8% yield). ***Rf*** = 0.6 (Petroleum ether /EtOAc = 5:1); **^{1}H NMR (400 MHz, CDCl$_3$):** δ 7.27 (s, 1H), 7.16 (d, J = 7.2 Hz, 1H), 7.06 (t, J = 7.4 Hz, 1H), 6.84 (d, J = 7.7 Hz, 1H), 3.22 (s, 3H), 1.93 (dd, J = 14.0, 6.9 Hz, 1H), 1.72 (dd, J = 14.0, 5.2 Hz, 1H), 1.52 – 1.41 (m, 3H), 1.34 – 1.30 (m, 3H), 1.25 – 1.18 (m, 2H), 1.01 – 0.78 (m, 6H). The spectral data obtained were identical with those reported in literature.[2]

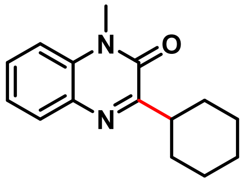




^{1}H NMR spectrum of compound **29**

10. Characterization Data for Electrolysis Products

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



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 **3** as a white solid (For boronic acid: 101.3 mg, 84% yield; For trifluoroborate: 68.6 mg, 60% yield; For boronic ester: 56.7 mg, 51% yield).

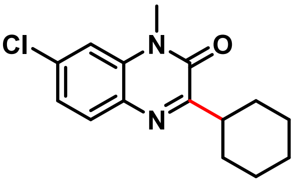
Rf = 0.7 (Petroleum ether /EtOAc = 5:1);

^{1}H NMR (400 MHz, CDCl$_3$): δ 7.83 (d, *J* = 7.8 Hz, 1H), 7.50 (t, *J* = 7.6 Hz, 1H), 7.36 – 7.26 (m, 2H), 3.69 (s, 3H), 3.34 (t, J = 11. 2 Hz, 1H), 1.96 (d, J = 11.6 Hz, 2H), 1.87 (d, J = 12.4 Hz, 2H), 1.77 (d, J = 12.4 Hz, 1H), 1.64 – 1.39 (m, 5H), 1.36 – 1.25 (m, 1H).

^{13}C NMR (101 MHz, CDCl$_3$): δ 164.3, 154.6, 132.9, 132.9, 129.8, 129.4, 123.4, 113.5, 40.8, 30.5, 29.1, 26.3, 26.2.

The spectral data obtained were identical with those reported in literature.[3]

7-chloro-3-cyclohexyl-1-methylquinoxalin-2(1*H*)-one 4



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 as a white solid (115.0 mg, 83% yield).

Rf = 0.8 (Petroleum ether /EtOAc = 5:1);

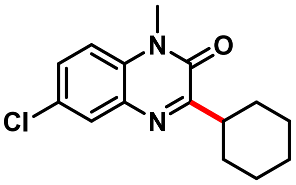
^{1}H NMR (400 MHz, CDCl$_3$): δ 7.74 (d, *J* = 9.1 Hz, 1H), 7.27 (d, *J* = 5.9 Hz, 2H), 3.65 (s, 3H), 3.31 (t, *J* = 11.2 Hz, 1H), 1.94 (d, *J* = 12.2 Hz, 2H), 1.86 (d, *J* = 12.4 Hz, 2H), 1.76 (d, *J* = 12.6 Hz, 1H), 1.60 –

1.40 (m, 5H), 1.36 – 1.26 (m, 1H).

^{13}C NMR (101 MHz, CDCl$_3$): *δ* 164.4, 154.3, 135.2, 133.8, 131.4, 130.8, 123.7, 113.5,40.8, 30.5, 29.2, 26.3, 26.1.

The spectral data obtained were identical with those reported in literature.[3]

6-chloro-3-cyclohexyl-1-methylquinoxalin-2(1*H*)-one 5



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 as a white solid (113.1 mg, 82% yield).

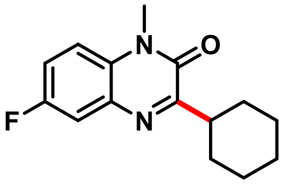
Rf =0.8 (Petroleum ether /EtOAc = 5:1);

^{1}H NMR (400 MHz, CDCl$_3$): *δ* 7.83 (d, *J* = 2.3 Hz, 1H), 7.44 (dd, *J* = 8.9, 2.4 Hz, 1H), 7.20 (d, *J* = 8.9 Hz, 1H), 3.67 (s, 3H), 3.32 (tt, *J* = 11.3, 3.1 Hz, 1H), 1.94 (d, *J* = 12.1 Hz, 3H), 1.86 (d, *J* = 12.5 Hz, 3H), 1.76 (d, *J* = 12.8 Hz, 1H), 1.60 – 1.39 (m, 5H), 1.36 – 1.26 (m, 1H).

^{13}C NMR (101 MHz, CDCl$_3$): *δ* 165.7, 154.2, 133.4, 131.6, 129.3, 129.2, 128.7, 114.6, 40.9, 30.5, 29.2, 26.2, 26.1.

The spectral data obtained were identical with those reported in literature.[3]

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



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 as a white solid (70.8 mg, 54% yield).

Rf = 0.5 (Petroleum ether /EtOAc = 5:1);

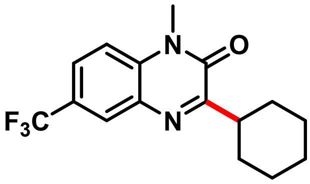
^{1}H NMR (400 MHz, CDCl$_3$): *δ* 7.52 (d, *J* = 8.8 Hz, 1H), 7.24 (d, *J* = 5.4 Hz, 2H), 3.69 (s, 3H), 3.33 (t, *J* = 10.9 Hz, 1H), 1.94 (d, *J* = 11.8 Hz, 2H), 1.86 (d, *J* = 12.3 Hz, 2H), 1.76 (d, *J* = 12.6 Hz, 1H), 1.65 – 1.38 (m, 5H), 1.35 – 1.25 (m, 1H).

^{13}C NMR (101 MHz, CDCl$_3$): *δ* 165.8, 159.8, 157.4, 154.2, 133.5 (d, *J* = 11.4 Hz), 129.5, 117.1, 116.8, 115.3, 115.1, 114.5 (d, *J* = 8.8 Hz), 40.9, 30.5, 29.3, 26.1.

^{19}F NMR (377 MHz, CDCl$_3$): *δ* -119.5.

The spectral data obtained were identical with those reported in literature.[3]

3-cyclohexyl-1-methyl-6-(trifluoromethyl)quinoxalin-2(1*H*)-one 7



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 as a white solid (113.3mg, 73% yield).

Rf = 0.5 (Petroleum ether /EtOAc = 5:1);

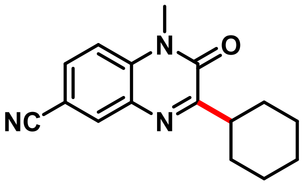
^{1}H NMR (400 MHz, CDCl$_3$): *δ* 8.13 (s, 1H), 7.73 (d, *J* = 8.1 Hz, 1H), 7.37 (d, *J* = 8.5 Hz, 1H), 3.72 (s, 3H), 3.36 (d, *J* = 11.0 Hz, 1H), 1.97 (s, 2H), 1.88 (d, *J* = 11.7 Hz, 2H), 1.78 (d, *J* = 11.9 Hz, 1H), 1.62 – 1.39 (m, 5H), 1.32 – 1.26 (m, 2H).

^{13}C NMR (101 MHz, CDCl$_3$): *δ* 166.1, 154.4, 135.3, 132.3, 127.7 – 126.9 (m), 125.9 – 125.4 (m), 114.1, 40.9, 30.5, 29.4, 26.3, 26.1

^{19}F NMR (377 MHz, CDCl$_3$): δ -61.9.

The spectral data obtained were identical with those reported in literature.[3]

3-cyclohexyl-1-methyl-2-oxo-1,2-dihydroquinoxaline-6-carbonitrile 8



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 as a yellow solid (86.7 mg, 65% yield).

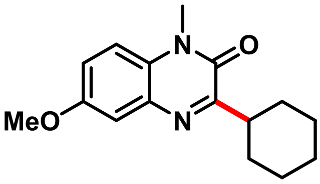
Rf = 0.2 (Petroleum ether /EtOAc = 5:1);

^{1}H NMR (400 MHz, CDCl$_3$): δ 7.50 (d, J = 8.6 Hz, 1H), 7.22 (d, J = 5.8 Hz, 2H), 3.68 (s, 3H), 3.32 (s, 1H), 1.93 (d, J = 12.0 Hz, 3H), 1.85 (d, J = 12.2 Hz, 2H), 1.75 (d, J = 12.6 Hz, 1H), 1.61 – 1.40 (m, 5H), 1.33 – 1.25 (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, 26.1, 26.0.

The spectral data obtained were identical with those reported in literature.[3]

3-cyclohexyl-6-methoxy-1-methylquinoxalin-2(1*H*)-one 9



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 as a white solid (111.3 mg, 82% yield).

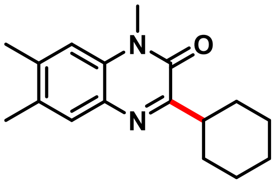
Rf = 0.5 (Petroleum ether /EtOAc = 5:1);

^{1}H NMR (400 MHz, CDCl$_3$): δ 7.74 (d, J = 8.8 Hz, 1H), 6.96 – 6.84 (m, 1H), 6.70 (d, J = 2.3 Hz, 1H), 3.92 (s, 3H), 3.66 (s, 3H), 3.33 – 3.22 (m, 1H), 1.93 (d, J = 11.4 Hz, 2H), 1.86 (d, J = 12.5 Hz, 2H), 1.76 (d, J = 12.5 Hz, 1H), 1.62 – 1.40 (m, 5H), 1.31 (s, 1H), 1.30 – 1.27 (m, 1H).

^{13}C NMR (101 MHz, CDCl$_3$): δ 160.8, 160.6, 155.0, 134.3, 131.0, 127.8, 110.2, 97.9, 55.8, 40.6, 30.6, 29.1, 26.4, 26.2.

The spectral data obtained were identical with those reported in literature.[3]

3-cyclohexyl-1,6,7-trimethylquinoxalin-2(1*H*)-one 10



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 as a white solid (114.2 mg, 84% yield).

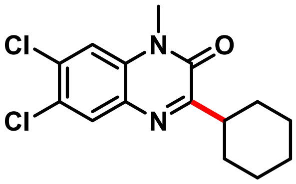
Rf = 0.6 (Petroleum ether /EtOAc = 5:1);

^{1}H NMR (400 MHz, CDCl$_3$): δ 7.60 (s, 1H), 7.03 (s, 1H), 3.66 (s, 3H), 3.37 – 3.26 (m, 1H), 2.40 (s, 3H), 2.33 (s, 3H), 1.94 (d, J = 12.6 Hz, 2H), 1.86 (d, J = 12.5 Hz, 2H), 1.76 (d, J = 12.6 Hz, 1H), 1.62 – 1.39 (m, 5H), 1.33 – 1.27 (m, 1H).

^{13}C NMR (101 MHz, CDCl$_3$): δ 163.0, 154.6, 138.9, 132.2, 131.3, 130.8, 129.9, 114.1, 77.4, 77.0, 76.7, 40.6, 30.6, 28.9, 26.4, 26.2, 20.5, 19.1.

The spectral data obtained were identical with those reported in literature.[3]

6,7-dichloro-3-cyclohexyl-1-methylquinoxalin-2(1*H*)-one 11



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 as a white solid (155.6 mg, 58% yield).

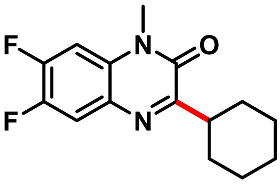
Rf = 0.7 (Petroleum ether /EtOAc = 5:1);

^{1}H NMR (400 MHz, CDCl$_3$): *δ* 7.88 (s, 1H), 7.34 (s, 1H), 3.64 (s, 3H), 3.29 (t, *J* = 10.2 Hz, 1H), 1.93 (d, *J* = 10.8 Hz, 2H), 1.86 (d, *J* = 10.7 Hz, 2H), 1.77 (d, *J* = 12.5 Hz, 1H), 1.58 – 1.37 (m, 5H), 1.35 – 1.26 (m, 1H).

^{13}C NMR (101 MHz, CDCl$_3$): *δ* 165.8, 153.9, 133.2, 132.3, 131.9, 130.6, 127.0, 114.9, 40.9, 30.4, 29.3, 26.2, 26.1.

The spectral data obtained were identical with those reported in literature.[3]

3-cyclohexyl-6,7-difluoro-1-methylquinoxalin-2(1*H*)-one 12



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 as a white solid (71.7 mg, 52% yield).

Rf = 0.6 (Petroleum ether /EtOAc = 5:1);

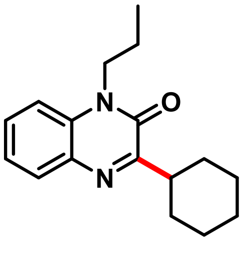
^{1}H NMR (400 MHz, CDCl$_3$): *δ* 7.70 – 7.58 (m, 1H), 7.15 – 7.02 (m, 1H), 3.67 (s, 1H), 3.30 (d, *J* = 9.4 Hz, 1H), 2.00 – 1.82 (m, 4H), 1.77 (s, 1H), 1.60 – 1.39 (m, 4H), 1.35 – 1.26 (m, 2H).

^{13}C NMR (101 MHz, CDCl$_3$): *δ* 164.8, 154.1, 152.1 (d, *J* = 16.4 Hz), 149.6 (d, *J* = 16.1 Hz), 147.7 (d, *J* = 13.7 Hz), 145.3 (d, *J* = 12.4 Hz), 130.0 (d, *J* = 9.1 Hz), 129.1, 117.4 (d, *J* = 18.0 Hz), 102.2, 101.9, 40.8, 30.4, 29.5, 26.2, 26.1.

^{19}F NMR (377 MHz, CDCl$_3$): *δ* -132.0 – -132.5 (m), -142.5 – -143.0 (m).

The spectral data obtained were identical with those reported in literature.[3]

3-cyclohexyl-1-propylquinoxalin-2(1*H*)-one 13



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 as a white solid (51.3 mg, 38% yield).

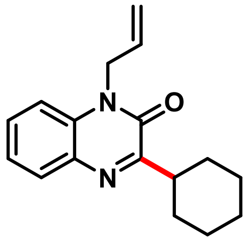
Rf = 0.8 (Petroleum ether /EtOAc = 5:1);

^{1}H NMR (400 MHz, CDCl$_3$): *δ* 7.84 (d, *J* = 7.8 Hz, 1H), 7.53 – 7.45 (m, 1H), 7.33 – 7.24 (m, 2H), 4.28 – 4.15 (m, 2H), 3.41 – 3.28 (m, 1H), 1.96 (d, *J* = 12.1 Hz, 2H), 1.86 (d, *J* = 12.9 Hz, 2H), 1.83 – 1.73 (m, 3H), 1.67 – 1.37 (m, 5H), 1.37 – 1.26 (m, 1H), 1.09 – 1.01 (m, 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.

The spectral data obtained were identical with those reported in literature.[3]

1-allyl-3-cyclohexylquinoxalin-2(1*H*)-one 14



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 as a white solid (70.0 mg, 52% yield).

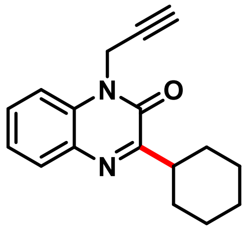
Rf = 0.3(Petroleum ether /EtOAc = 20:1);

^{1}H NMR (400 MHz, CDCl$_3$): *δ* 7.87 – 7.81 (m, 1H), 7.50 – 7.43 (m, 1H), 7.33 – 7.23 (m, 3H), 6.00 – 5.87 (m, 1H), 5.26 (d, *J* = 10.4 Hz, 1H), 5.17 (d, *J* = 17.3 Hz, 1H), 4.90 (d, *J* = 5.1 Hz, 2H), 3.35 (ddd, *J* = 11.5, 7.4, 3.2 Hz, 1H), 1.97 (d, *J* = 11.4 Hz, 2H), 1.92 – 1.83 (m, 3H), 1.76 (d, *J* = 12.6 Hz, 2H), 1.59 – 1.41 (m, 5H), 1.34 – 1.28 (m, 1H).

^{13}C NMR (101 MHz, CDCl$_3$): *δ* 164.3, 154.1, 133.1, 132.1, 130.8, 129.9, 129.3, 123.4, 118.0, 114.0, 44.5, 40.8, 30.6, 26.3, 26.2.

The spectral data obtained were identical with those reported in literature.[3]

3-cyclohexyl-1-(prop-2-yn-1-yl)quinoxalin-2(1*H*)-one 15



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 as a white solid (55.6 mg, 42% yield).

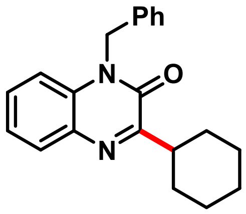
Rf = 0.3 (Petroleum ether /EtOAc = 20:1);

^{1}H NMR (400 MHz, CDCl$_3$): *δ* 7.85 (d, *J* = 8.0 Hz, 1H), 7.54 (t, *J* = 7.7 Hz, 1H), 7.43 (d, *J* = 8.2 Hz, 1H), 7.35 (t, *J* = 7.6 Hz, 1H), 5.05 (s, 2H), 3.33 (t, *J* = 11.4 Hz, 1H), 2.28 (s, 1H), 1.96 (d, *J* = 12.1 Hz, 2H), 1.87 (d, *J* = 12.6 Hz, 2H), 1.77 (d, *J* = 12.7 Hz, 1H), 1.59 – 1.43 (m, 4H), 1.33 – 1.27 (m, 2H).

^{13}C NMR (101 MHz, CDCl$_3$): *δ* 164.2, 153.5, 133.1, 131.4, 129.9, 129.5, 123.8, 113.9, 73.0, 40.8, 31.5, 30.5, 26.3, 26.1.

The spectral data obtained were identical with those reported in literature.[3]

1-benzyl-3-cyclohexylquinoxalin-2(1*H*)-one 16



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 as a white solid (80.3mg, 51% yield).

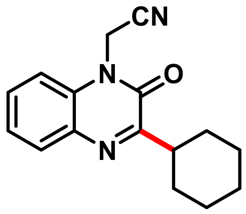
Rf =0.3 (Petroleum ether /EtOAc = 20:1);

^{1}H NMR (400 MHz, CDCl$_3$): *δ* 7.87 – 7.82 (m, 1H), 7.41 – 7.28 (m, 4H), 7.25 – 7.21 (m, 3H), 5.49 (s, 2H), 3.45 – 3.35 (m, 1H), 2.01 (d, *J* = 11.5 Hz, 2H), 1.89 (d, *J* = 12.8 Hz, 2H), 1.78 (d, *J* = 12.8 Hz, 1H), 1.61 – 1.48 (m, 4H), 1.37 – 1.30 (m, 1H).

^{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.

The spectral data obtained were identical with those reported in literature.[3]

2-(3-cyclohexyl-2-oxoquinoxalin-1(2*H*)-yl)acetonitrile 17



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 as a white solid (66.7 mg, 50% yield).

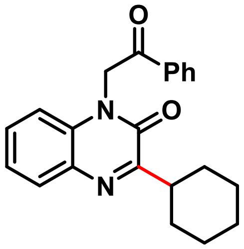
Rf = 0.2 (Petroleum ether /EtOAc = 5:1);

^{1}H NMR (400 MHz, CDCl$_3$): *δ* 7.92 – 7.87 (m, 1H), 7.62 – 7.54 (m, 1H), 7.44 – 7.38 (m, 1H), 7.31 – 7.24 (m, 2H), 5.19 (s, 2H), 3.30 (tt, *J* = 11.5, 3.2 Hz, 1H), 1.96 (d, *J* = 11.3 Hz, 2H), 1.91 – 1.84 (m, 2H), 1.79 – 1.73 (m, 1H), 1.60 – 1.43 (m, 4H), 1.33 – 1.28 (m, 1H).

^{13}C NMR (101 MHz, CDCl$_3$): *δ* 163.8, 153.2, 133.0, 130.6, 130.5, 130.1, 124.7, 113.8, 112.8, 41.0, 30.5, 29.3, 26.2, 26.1.

The spectral data obtained were identical with those reported in literature.[3]

3-cyclohexyl-1-(2-oxo-2-phenylethyl)quinoxalin-2(1*H*)-one 18



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 as a white solid (87.6 mg, 51% yield).

M. p. = 42 – 43 °C;

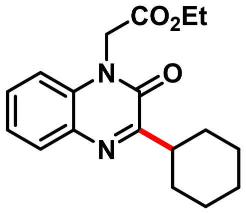
Rf = 0.5 (Petroleum ether /EtOAc = 5:1);

^{1}H NMR (400 MHz, CDCl$_3$): *δ* 8.08 (d, *J* = 7.7 Hz, 2H), 7.87 (d, *J* = 7.9 Hz, 1H), 7.67 (t, *J* = 7.4 Hz, 1H), 7.55 (t, *J* = 7.6 Hz, 2H), 7.39 (t, *J* = 7.7 Hz, 1H), 7.30 (t, *J* = 7.6 Hz, 1H), 6.93 (d, *J* = 8.3 Hz, 1H), 5.72 (s, 2H), 3.33 (t, *J* = 11.6 Hz, 1H), 1.99 (d, *J* = 12.3 Hz, 2H), 1.87 (d, *J* = 12.6 Hz, 2H), 1.76 (d, *J* = 12.2 Hz, 1H), 1.66 – 1.56 (m, 2H), 1.50 – 1.38 (m, 2H), 1.38 – 1.27 (m, 1H).

^{13}C NMR (101 MHz, CDCl$_3$): *δ* 191.4, 163.9, 154.3, 134.7, 134.3, 133.1, 132.3, 130.1, 129.4, 129.0, 128.2, 123.6, 113.3, 48.5, 40.9, 30.5, 26.3, 26.2.

HRMS (ESI): calc'd for $C_{22}H_{22}N_2O_2$ ([M+H]$^+$) 347.1759, found 347.1754.

ethyl 2-(3-cyclohexyl-2-oxoquinoxalin-1(2*H*)-yl)acetate 19



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 as a white solid (62.8 mg, 40% yield).

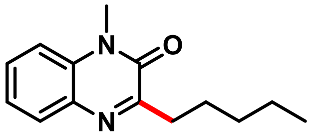
Rf = 0.6 (Petroleum ether /EtOAc = 5:1);

^{1}H NMR (400 MHz, CDCl$_3$): *δ* 7.85 (d, *J* = 7.6 Hz, 1H), 7.45 (d, *J* = 7.4 Hz, 1H), 7.32 (t, *J* = 7.6 Hz, 1H), 7.04 (d, *J* = 8.3 Hz, 1H), 5.01 (s, 2H), 4.24 (q, *J* = 7.1 Hz, 2H), 3.32 (tt, *J* = 11.5, 3.2 Hz, 1H), 1.97 (d, *J* = 11.8 Hz, 2H), 1.87 (d, *J* = 12.7 Hz, 2H), 1.73 (d, *J* = 15.5 Hz, 1H), 1.64 – 1.52 (m, 2H), 1.52 – 1.42 (m, 2H), 1.37 – 1.29 (m, 1H), 1.26 (t, *J* = 7.0 Hz, 3H).

^{13}C NMR (101 MHz, CDCl$_3$): *δ* 167.3, 164.1, 154.1, 133.0, 132.0, 130.1, 129.5, 123.7, 112.9, 62.0, 43.6, 40.8, 30.5, 26.3, 26.2, 14.1.

The spectral data obtained were identical with those reported in literature.[3]

1-methyl-3-pentylquinoxalin-2(1*H*)-one 20



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 as a white solid (45.4 mg, 42% yield).

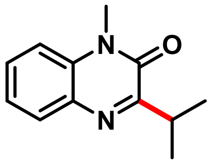
Rf = 0.5 (Petroleum ether /EtOAc = 5:1);

^{1}H NMR (400 MHz, CDCl$_3$): *δ* 7.83 (d, *J* = 8.0 Hz, 1H), 7.52 (s, 1H), 7.36 – 7.27 (m, 2H), 3.70 (s, 3H), 2.94 (t, *J* = 8.0 Hz, 2H), 1.85 – 1.73 (m, 2H), 1.46 – 1.36 (m, 4H), 0.92 (t, *J* = 7.2 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, 34.4, 31.8, 29.0, 26.6, 22.6, 14.1.

The spectral data obtained were identical with those reported in literature.[3]

3-isopropyl-1-methylquinoxalin-2(1*H*)-one 21



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 as a white solid (For boronic acid 63.7 mg, 63% yield; For trifluoroborate 78.3 mg, 78% yield).

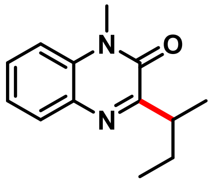
Rf = 0.5 (Petroleum ether /EtOAc = 5:1);

^{1}H NMR (400 MHz, CDCl$_3$): *δ* 7.85 (d, *J* = 7.9 Hz, 1H), 7.51 (t, *J* = 7.7 Hz, 1H), 7.36 – 7.27 (m, 2H), 3.70 (s, 3H), 3.63 (dt, *J* = 13.6, 6.8 Hz, 1H), 1.33 (s, 3H), 1.31 (s, 3H).

^{13}C NMR (101 MHz, CDCl$_3$): *δ* 165.0, 154.5, 133.0, 132.8, 129.8, 129.4, 123.4, 113.5, 31.2, 29.0, 20.2.

The spectral data obtained were identical with those reported in literature.[3]

3-(*sec*-butyl)-1-methylquinoxalin-2(1*H*)-one 22



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 as a white solid (81.8 mg, 76% yield).

M. p. =132 – 133 °C;

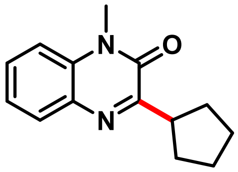
Rf = 0.5 (Petroleum ether /EtOAc = 5:1);

^{1}H NMR (400 MHz, CDCl$_3$): *δ* 7.82 (d, *J* = 7.7 Hz, 1H), 7.50 (t, *J* = 7.5 Hz, 1H), 7.32 (d, *J* = 7.5 Hz, 1H), 7.30 – 7.26 (m, 1H), 3.76 – 3.65 (m, 4H), 2.13 – 2.00 (m, 2H), 1.99 – 1.88 (m, 2H), 1.87 – 1.77 (m, 2H),

^{13}C NMR (101 MHz, CDCl$_3$): *δ* 164.5, 154.7, 132.9, 132.8, 129.8, 129.4, 123.4, 113.5, 37.8, 29.1, 27.5, 17.9, 12.0.

HRMS (EI): calc'd for $C_{13}H_{16}N_2O$ ($[M+H]^+$) 217.1341, found 217.1331.

3-cyclopentyl-1-methylquinoxalin-2(1*H*)-one 23



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 as a white solid (For boronic acid 43.8 mg, 38% yield; For trifluoroborate 52.5 mg, 46% yield).

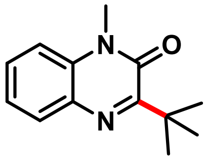
Rf = 0.6 (Petroleum ether /EtOAc = 5:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82 (d, J = 7.7 Hz, 1H), 7.50 (t, J = 7.5 Hz, 1H), 7.32 (d, J = 7.5 Hz, 1H), 7.30 – 7.26 (m, 1H), 3.76 – 3.65 (m, 4H), 2.13 – 2.00 (m, 2H), 1.99 – 1.88 (m, 2H), 1.87 – 1.77 (m, 2H), 1.74 – 1.67 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 163.7, 155.0, 133.0, 132.7, 129.8, 129.3, 123.4, 113.4, 42.7, 30.8, 29.0, 25.9.

The spectral data obtained were identical with those reported in literature.[3]

3-(*tert*-butyl)-1-methylquinoxalin-2(1*H*)-one 24



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 as a white solid (For boronic acid: 45.3 mg, 42% yield; For trifluoroborate: 56.3 mg, 52% yield; For boronic ester: 70.1 mg, 65% yield).

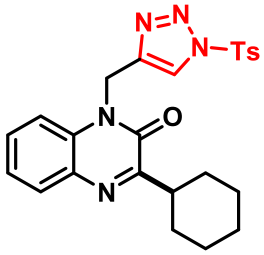
Rf = 0.6 (Petroleum ether /EtOAc = 5:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 (d, J = 7.9 Hz, 1H), 7.50 (s, 1H), 7.34 – 7.25 (m, 2H), 3.67 (s, 3H), 1.49 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 165.3, 153.8, 133.4, 132.2, 130.1, 129.5, 123.2, 113.2, 39.5, 28.7, 27.9.

The spectral data obtained were identical with those reported in literature.[3]

3-cyclohexyl-1-((1-tosyl-1H-1,2,3-triazol-4-yl)methyl)quinoxalin-2(1*H*)-one 25



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 as a white solid (92.6 mg, 97% yield).

M. p. =141 – 142 °C;

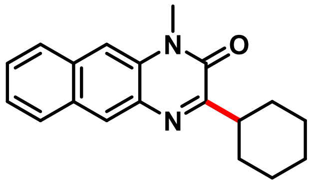
Rf = 0.5 (Petroleum ether /EtOAc = 10:1);

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.19 (d, J = 2.8 Hz, 1H), 8.00 – 7.92 (m, 2H), 7.81 (d, J = 6.2 Hz, 1H), 7.71 – 7.64 (m, 1H), 7.49 (d, J = 6.9 Hz, 1H), 7.48 – 7.44 (m, 1H), 7.36 (d, J = 6.0 Hz, 2H), 7.33 – 7.28 (m, 1H), 5.50 (s, 1H), 3.32 (dd, J = 11.2, 8.4 Hz, 1H), 2.42 (s, 3H), 1.95 (d, J = 11.9 Hz, 2H), 1.88 (d, J = 10.5 Hz, 2H), 1.78 (d, J = 11.6 Hz, 1H), 1.59 – 1.43 (m, 4H), 1.36 – 1.30 (m, 1H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 163.9, 154.2, 147.5, 142.3, 133.1, 132.7, 131.7, 130.5, 129.9, 129.8, 128.9, 123.9, 123.5, 114.2, 40.8, 37.6, 30.6, 26.3, 26.1, 21.8.

HRMS (ESI): calc'd for $C_{24}H_{25}N_5O_3S$ ($[M+H]^+$) 464.1756, found 464.1746.

3-cyclohexyl-1-methylbenzo[g]quinoxalin-2(1*H*)-one 26



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 as a yellow solid (78.6 mg, 49% yield).

M. p. = 127 – 128 °C;

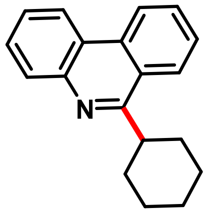
Rf = 0.2 (Petroleum ether /EtOAc = 10:1);

^{1}H NMR (400 MHz, CDCl$_3$): *δ* 8.34 (s, 1H), 7.95 (d, *J* = 8.2 Hz, 1H), 7.89 (d, *J* = 8.3 Hz, 1H), 7.57 (s, 1H), 7.54 (d, *J* = 8.1 Hz, 1H), 7.50 – 7.42 (m, 1H), 3.76 (d, *J* = 0.6 Hz, 3H), 3.38 (ddd, *J* = 11.5, 7.4, 3.0 Hz, 1H), 2.00 (d, *J* = 12.0 Hz, 2H), 1.89 (d, *J* = 12.7 Hz, 2H), 1.79 (d, *J* = 12.4 Hz, 1H), 1.64 – 1.49 (m, 4H), 1.39 – 1.30 (m, 1H).

^{13}C NMR (101 MHz, CDCl$_3$): *δ* 164.8, 154.4, 133.3, 132.3, 131.7, 128.6, 128.4, 127.5, 127.1, 125.1, 109.7, 40.9, 30.7, 29.1, 26.4, 26.2.

HRMS (ESI): calc'd for $C_{19}H_{20}N_2O$ ($[M+H]^+$) 293.1654, found 293.1644.

6-cyclohexylphenanthridine 27



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 as a colorless oil (84.9 mg, 65% yield).

Rf = 0.8 (Petroleum ether /EtOAc = 20:1);

^{1}H NMR (400 MHz, CDCl$_3$): *δ* 8.62 (d, *J* = 8.2 Hz, 1H), 8.51 (d, *J* = 8.1 Hz, 1H), 8.29 (d, *J* = 8.2 Hz, 1H), 8.13 (d, *J* = 8.1 Hz, 1H), 7.82 – 7.75 (m, 1H), 7.72 – 7.63 (m, 2H), 7.63 – 7.54 (m, 1H), 3.71 – 3.53 (m, 1H), 2.07 (d, *J* = 11.1 Hz, 2H), 2.02 – 1.90 (m, 4H), 1.84 (d, *J* = 12.0 Hz, 1H), 1.63 – 1.51 (m, 2H), 1.51 – 1.38 (m, 1H).

^{13}C NMR (101 MHz, CDCl$_3$): *δ* 165.3, 143.9, 133.0, 130.0, 128.4, 127.1, 126.1, 125.6, 124.8, 123.4, 122.6, 121.8, 77.4, 77.1, 76.8, 42.0, 32.3, 26.9, 26.4.

The spectral data obtained were identical with those reported in literature.[4]

11. X-ray Crystallography

Table 1 Crystal data and structure refinement for p20200606a.

Identification code	p20200606a
Empirical formula	$C_{15}H_{16}F_2N_2O$
Formula weight	278.30
Temperature/K	294.15
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	9.85240(10)
b/Å	12.04300(10)
c/Å	12.43970(10)

α/°	90
β/°	112.9820(10)
γ/°	90
Volume/Å^3	1358.85(2)
Z	4
ρ_{calc}g/cm^3	1.360
μ/mm^{-1}	0.885
F(000)	584.0
Crystal size/mm^3	0.18 × 0.16 × 0.14
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	9.752 to 158.746
Index ranges	$-12 \le h \le 12, -13 \le k \le 15, -15 \le l \le 15$
Reflections collected	14345
Independent reflections	2879 [R_{int} = 0.0201, R_{sigma} = 0.0171]
Data/restraints/parameters	2879/0/183
Goodness-of-fit on F^2	1.092
Final R indexes [I>=2σ (I)]	R_1 = 0.0379, wR_2 = 0.1144
Final R indexes [all data]	R_1 = 0.0403, wR_2 = 0.1169
Largest diff. peak/hole / e Å^{-3}	0.15/-0.17

Table 2 Fractional Atomic Coordinates (×104) and Equivalent Isotropic Displacement Parameters (Å2×103) for p20200606a. Ueq is defined as 1/3 of of the trace of the orthogonalised UI*J* tensor.

Atom	***x***	***y***	***z***	**U(eq)**
F1	2143.9(11)	1948.1(7)	5542.7(9)	83.6(3)
F2	3767.4(11)	1248.2(7)	4379.4(9)	82.7(3)
O1	4325.3(10)	6775.7(7)	2726.3(8)	58.8(2)
N1	4112.8(10)	4962.9(8)	3150.1(8)	46.3(2)
N2	2381.6(10)	5659.0(8)	4349.3(8)	48.4(2)
C1	5063.8(16)	4621.2(11)	2551.2(12)	60.1(3)
C2	3605.8(12)	4178.5(9)	3727.1(9)	45.1(3)
C3	3937.7(13)	3049.4(10)	3728.3(11)	53.8(3)
C4	3432.3(15)	2335.5(10)	4344.5(12)	58.5(3)
C5	2595.9(15)	2695.5(11)	4947.1(12)	59.6(3)
C6	2255.2(14)	3791.2(11)	4948.0(11)	55.6(3)
C7	2756.2(12)	4554.0(9)	4332.1(10)	46.7(3)
C8	2853.9(12)	6374.8(9)	3803.6(9)	44.0(2)
C9	3815.0(12)	6074.4(9)	3177.2(9)	45.5(2)
C10	2469.4(12)	7583.0(9)	3801.6(9)	45.4(3)
C11	1388.7(14)	7974.6(11)	2595.3(10)	55.7(3)

C12	1126.6(15)	9218.9(11)	2609.8(12)	59.3(3)
C13	548.4(16)	9526.5(12)	3532.4(14)	67.2(4)
C14	1567.2(18)	9110.2(12)	4728.0(12)	67.5(4)
C15	1867.2(16)	7872.6(11)	4723.3(11)	57.1(3)

Table 3 Anisotropic Displacement Parameters (Å^2×10^3) for p20200606a. 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}
F1	99.6(7)	60.5(5)	110.5(7)	22.0(5)	62.5(6)	-2.6(4)
F2	103.0(7)	42.3(4)	116.8(7)	3.2(4)	58.1(6)	1.9(4)
O1	71.4(6)	50.6(5)	72.0(5)	5.7(4)	47.1(5)	1.9(4)
N1	49.5(5)	45.7(5)	50.8(5)	-2.9(4)	27.2(4)	1.2(4)
N2	51.7(5)	48.1(5)	52.0(5)	0.6(4)	27.4(4)	2.8(4)
C1	71.5(8)	55.9(7)	70.6(7)	-3.9(6)	47.0(6)	4.0(6)
C2	43.4(5)	45.6(6)	46.3(5)	-2.7(4)	17.7(4)	-1.6(4)
C3	55.2(6)	46.8(6)	62.4(7)	-5.8(5)	26.3(5)	-0.2(5)
C4	61.4(7)	41.8(6)	71.5(8)	-0.4(5)	25.2(6)	-1.5(5)
C5	60.9(7)	52.0(7)	69.0(7)	9.9(6)	28.9(6)	-5.1(5)
C6	57.0(7)	55.4(7)	61.8(7)	4.2(5)	31.3(5)	-0.4(5)
C7	45.9(5)	46.5(6)	50.3(6)	1.0(4)	21.6(4)	0.1(4)
C8	45.1(5)	46.1(6)	44.0(5)	-1.2(4)	20.8(4)	1.2(4)
C9	47.6(5)	46.0(6)	47.2(5)	-0.8(4)	23.0(4)	0.5(4)
C10	48.8(5)	45.7(6)	46.2(5)	-1.3(4)	23.6(4)	2.5(4)
C11	58.8(7)	60.0(7)	48.5(6)	-0.3(5)	21.3(5)	8.1(5)
C12	57.8(7)	60.2(7)	61.7(7)	10.5(6)	25.1(6)	11.6(5)
C13	69.0(8)	57.9(7)	86.3(9)	7.0(6)	42.9(7)	17.1(6)
C14	91.7(10)	57.6(7)	68.3(8)	-4.4(6)	47.7(7)	13.2(7)
C15	73.6(8)	54.7(7)	55.6(6)	2.4(5)	38.7(6)	9.4(6)

Table 4 Bond Lengths for p20200606a.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
F1	C5	1.3472(15)	C4	C5	1.382(2)
F2	C4	1.3470(14)	C5	C6	1.3618(19)
O1	C9	1.2250(14)	C6	C7	1.4035(16)
N1	C1	1.4654(15)	C8	C9	1.4869(15)

N1	C2	1.3918(15)	C8	C10	1.5034(15)
N1	C9	1.3735(14)	C10	C11	1.5363(16)
N2	C7	1.3834(14)	C10	C15	1.5230(15)
N2	C8	1.2912(15)	C11	C12	1.5219(18)
C2	C3	1.3985(16)	C12	C13	1.5132(19)
C2	C7	1.4012(16)	C13	C14	1.518(2)
C3	C4	1.3685(18)	C14	C15	1.5199(18)

Table 5 Bond Angles for p20200606a.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C2	N1	C1	120.07(10)	N2	C7	C6	117.93(11)
C9	N1	C1	118.12(10)	C2	C7	C6	119.55(11)
C9	N1	C2	121.72(9)	N2	C8	C9	123.17(10)
C8	N2	C7	118.75(10)	N2	C8	C10	120.31(10)
N1	C2	C3	121.99(10)	C9	C8	C10	116.50(9)
N1	C2	C7	117.90(10)	O1	C9	N1	122.06(10)
C3	C2	C7	120.11(11)	O1	C9	C8	122.10(10)
C4	C3	C2	118.38(12)	N1	C9	C8	115.83(10)
F2	C4	C3	119.38(12)	C8	C10	C11	112.13(9)
F2	C4	C5	118.54(12)	C8	C10	C15	113.11(9)
C3	C4	C5	122.08(12)	C15	C10	C11	109.87(10)
F1	C5	C4	118.93(12)	C12	C11	C10	110.29(10)
F1	C5	C6	120.82(13)	C13	C12	C11	111.47(11)
C6	C5	C4	120.24(12)	C12	C13	C14	111.19(11)
C5	C6	C7	119.63(12)	C13	C14	C15	112.03(12)
N2	C7	C2	122.52(10)	C14	C15	C10	111.16(10)

Table 6 Torsion Angles for p20200606a.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
F1	C5	C6	C7	179.20(11)	C4	C5	C6	C7	0.1(2)
F2	C4	C5	F1	-0.20(19)	C5	C6	C7	N2	179.71(11)
F2	C4	C5	C6	178.93(12)	C5	C6	C7	C2	-0.23(18)
N1	C2	C3	C4	178.15(10)	C7	N2	C8	C9	-1.50(16)
N1	C2	C7	N2	1.60(16)	C7	N2	C8	C10	179.96(9)
N1	C2	C7	C6	-178.47(10)	C7	C2	C3	C4	-0.95(17)
N2	C8	C9	O1	-175.39(10)	C8	N2	C7	C2	-1.21(16)

N2	C8	C9	N1	3.70(16)	C8	N2	C7	C6	178.85(10)
N2	C8	C10	C11	-110.02(12)	C8	C10	C11	C12	-175.51(10)
N2	C8	C10	C15	14.91(15)	C8	C10	C15	C14	177.16(11)
C1	N1	C2	C3	-2.02(16)	C9	N1	C2	C3	-178.29(10)
C1	N1	C2	C7	177.10(10)	C9	N1	C2	C7	0.83(15)
C1	N1	C9	O1	-0.47(17)	C9	C8	C10	C11	71.35(12)
C1	N1	C9	C8	-179.56(10)	C9	C8	C10	C15	-163.72(10)
C2	N1	C9	O1	175.87(10)	C10	C8	C9	O1	3.19(16)
C2	N1	C9	C8	-3.23(15)	C10	C8	C9	N1	-177.71(9)
C2	C3	C4	F2	-178.50(11)	C10	C11	C12	C13	-57.42(14)
C2	C3	C4	C5	0.82(19)	C11	C10	C15	C14	-56.70(15)
C3	C2	C7	N2	-179.27(10)	C11	C12	C13	C14	55.13(16)
C3	C2	C7	C6	0.67(16)	C12	C13	C14	C15	-53.84(17)
C3	C4	C5	F1	-179.52(12)	C13	C14	C15	C10	55.12(16)
C3	C4	C5	C6	-0.4(2)	C15	C10	C11	C12	57.80(14)

Table 7 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for p20200606a.

Atom	*x*	*y*	*z*	U(eq)
H1A	5285.99	5253.92	2179.44	90
H1B	4565.66	4070.76	1973.26	90
H1C	5962.62	4314.91	3108.63	90
H3	4488.55	2789.91	3320.27	65
H6	1693.62	4032.77	5354.44	67
H10	3383.4	8005.55	3988.49	54
H11A	1788.18	7805.71	2014.2	67
H11B	460.12	7582.99	2384.69	67
H12A	2043.9	9610.04	2762.02	71
H12B	422	9449.92	1849.69	71
H13A	-424.27	9207.39	3329.7	81
H13B	458.45	10327.36	3555.87	81
H14A	1124.66	9261.18	5285.84	81
H14B	2493.59	9510.87	4977.66	81
H15A	960.93	7464.74	4567.33	69
H15B	2573.69	7651.13	5486.45	69

Experimental

Single crystals of $C_{15}H_{16}F_2N_2O$ **[p20200606a]** were **[]**. A suitable crystal was selected and **[]** on a **Rigaku Xtalab P200** diffractometer. The crystal was kept at 294.15 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 [p20200606a]

Crystal Data for $C_{15}H_{16}F_2N_2O$ (M =278.30 g/mol): monoclinic, space group $P2_1/c$ (no. 14), a = 9.85240(10) Å, b = 12.04300(10) Å, c = 12.43970(10) Å, β = 112.9820(10)°, V = 1358.85(2) Å^3, Z = 4, T = 294.15 K, μ(CuKα) = 0.885 mm^{-1}, *Dcalc* = 1.360 g/cm^3, 14345 reflections measured (9.752° ≤ 2Θ ≤ 158.746°), 2879 unique (R_{int} = 0.0201, R_{sigma} = 0.0171) which were used in all calculations. The final R_1 was 0.0379 (I > 2σ(I)) and wR_2 was 0.1169 (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)

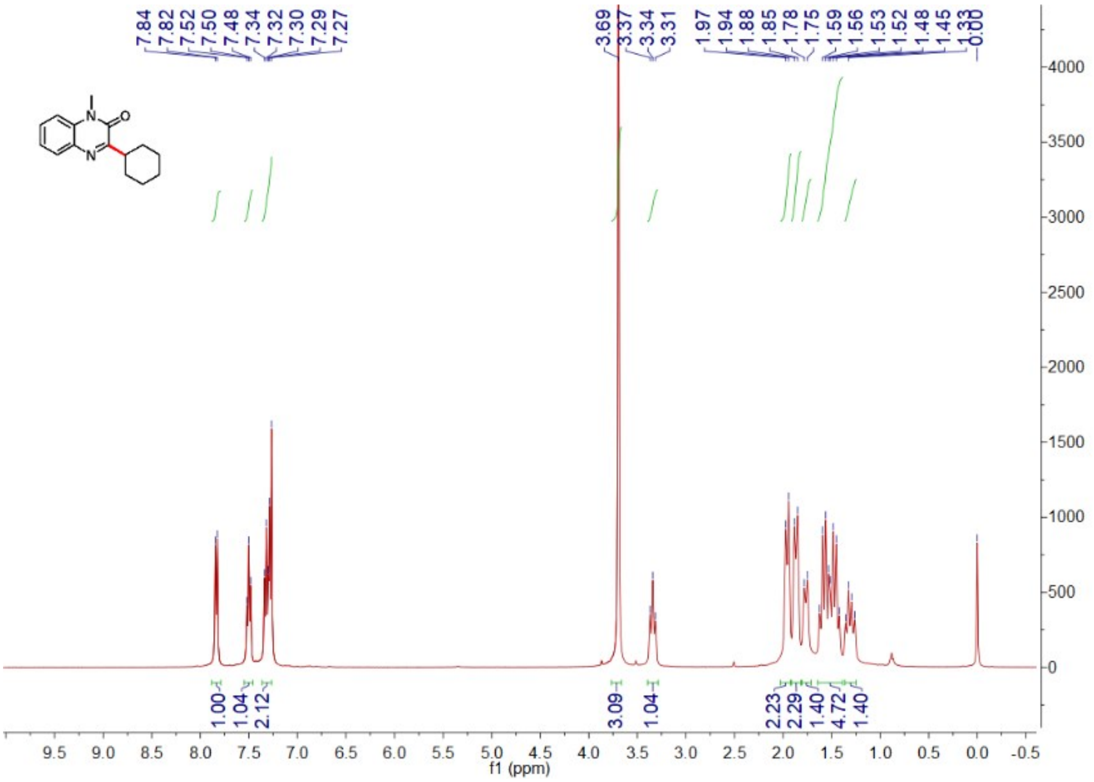
2.b Secondary CH2 refined with riding coordinates:
  C11(H11A,H11B), C12(H12A,H12B), C13(H13A,H13B), C14(H14A,H14B), C15(H15A,H15B)

2.c Aromatic/amide H refined with riding coordinates:
  C3(H3), C6(H6)

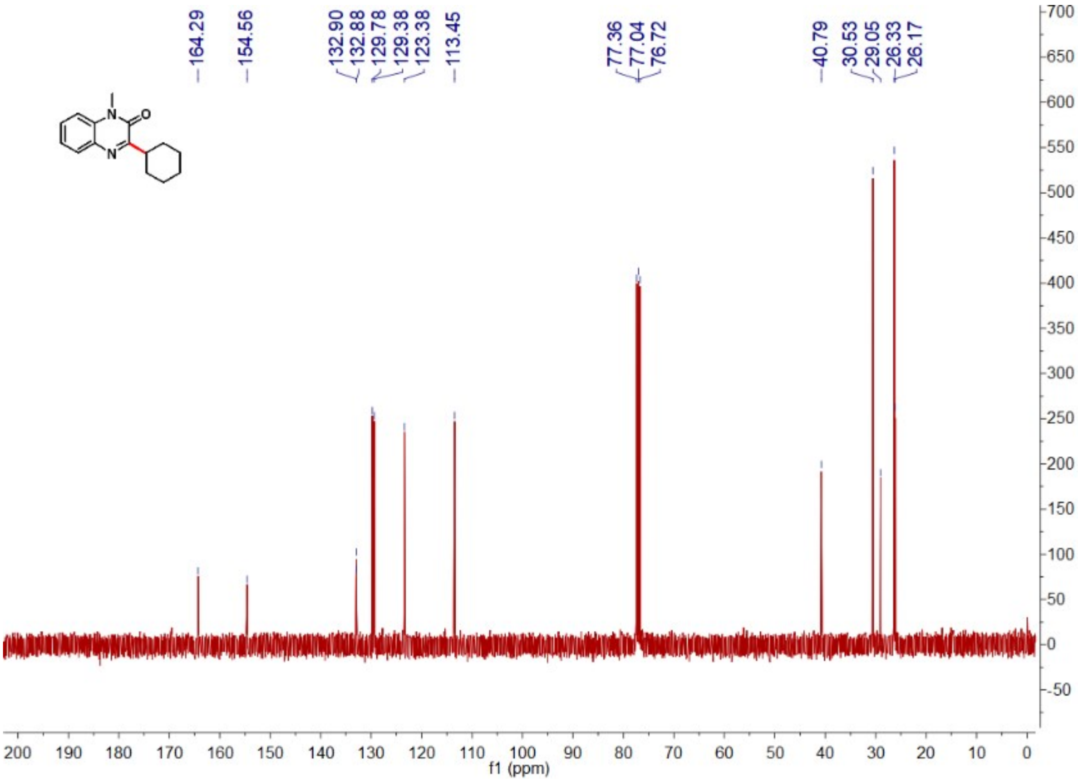
2.d Idealised Me refined as rotating group:
  C1(H1A,H1B,H1C)

12. Spectra of prepared compounds

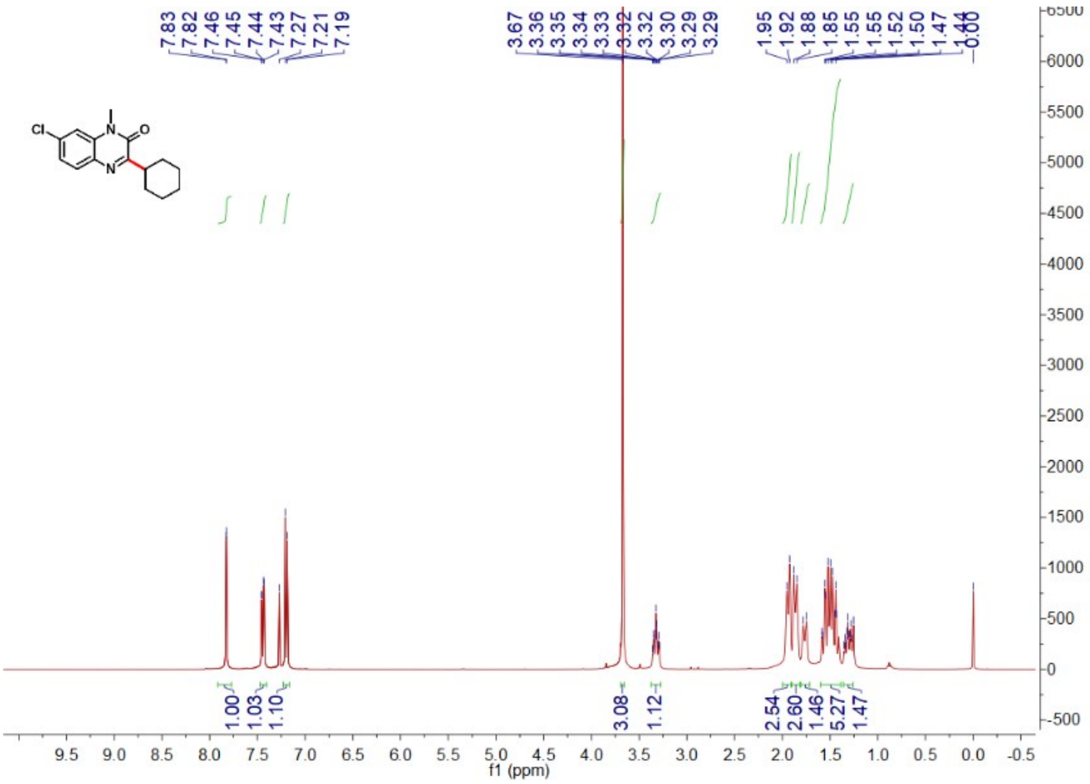
^{1}H NMR spectrum of compound **3**



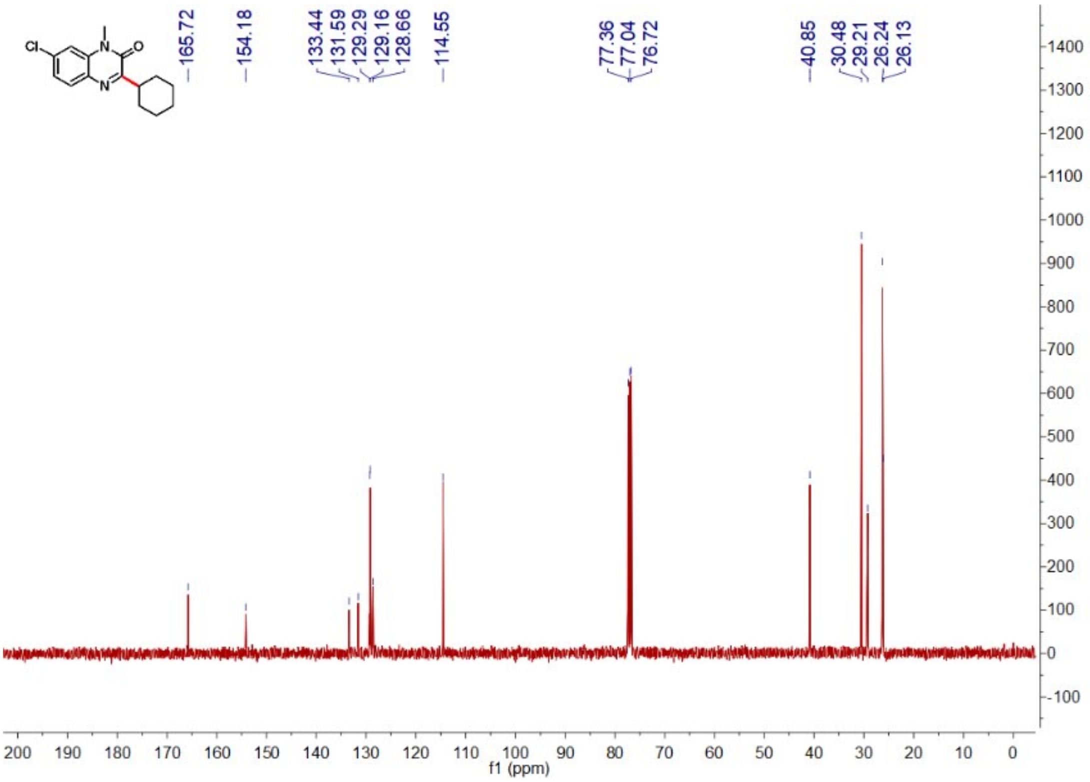
^{13}C NMR spectrum of compound **3**



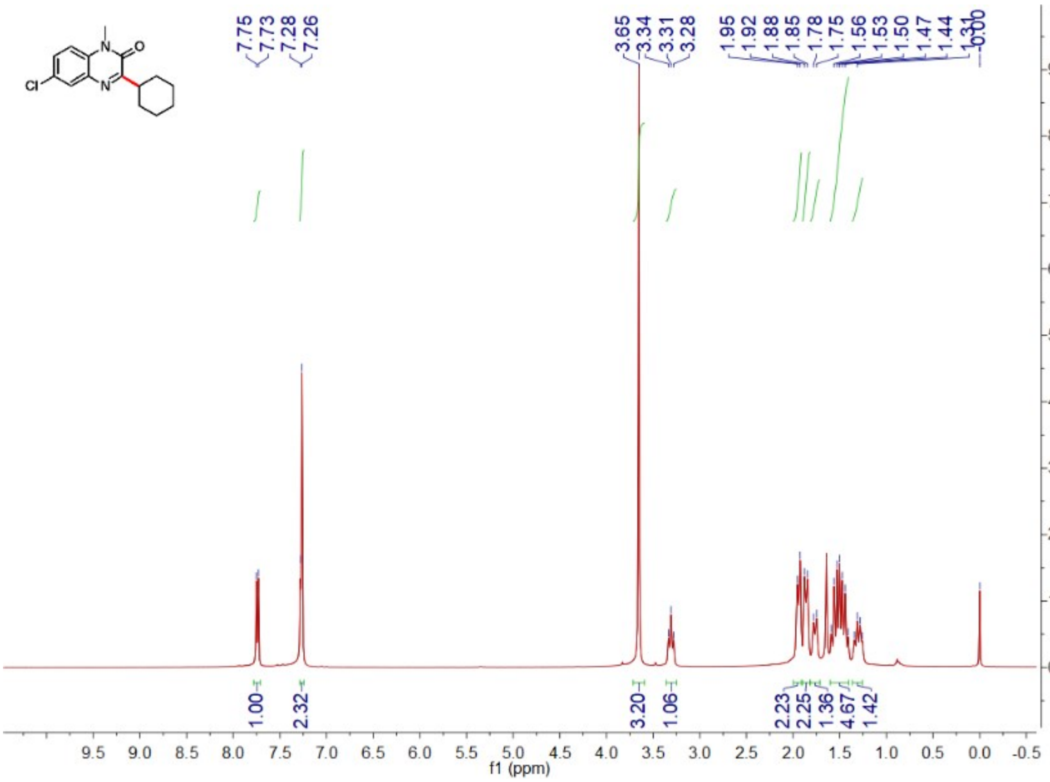
^{1}H NMR spectrum of compound **4**



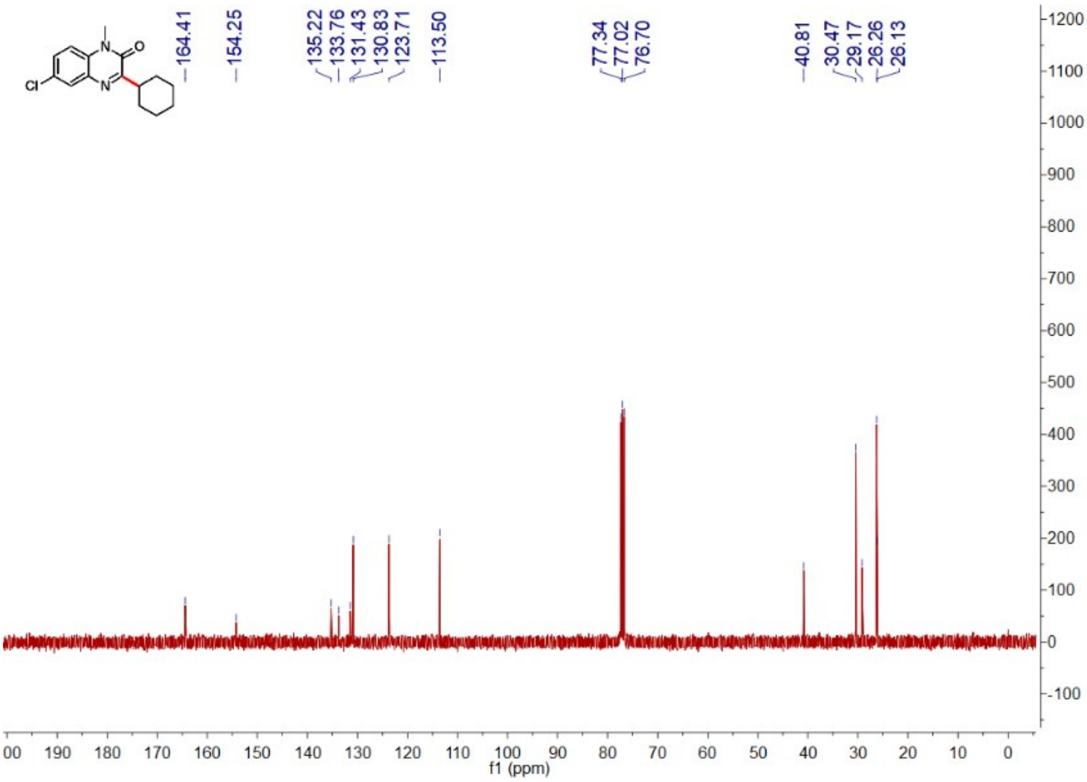
^{13}C NMR spectrum of compound **4**



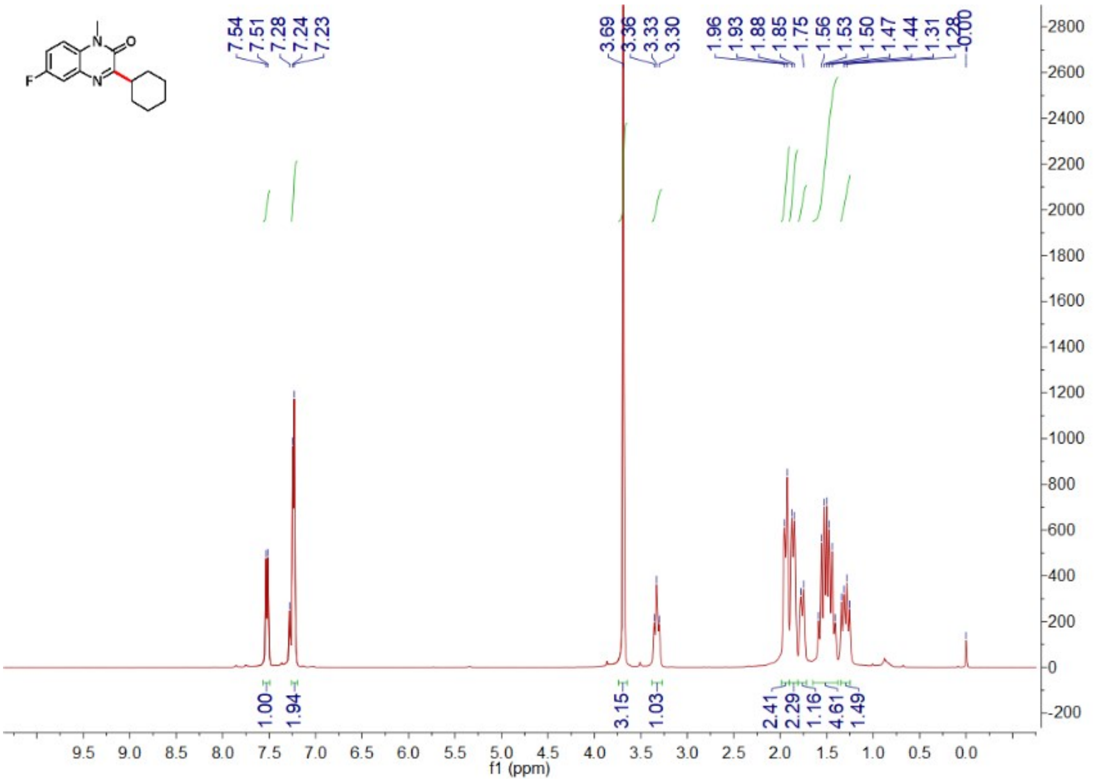
^{1}H NMR spectrum of compound **5**



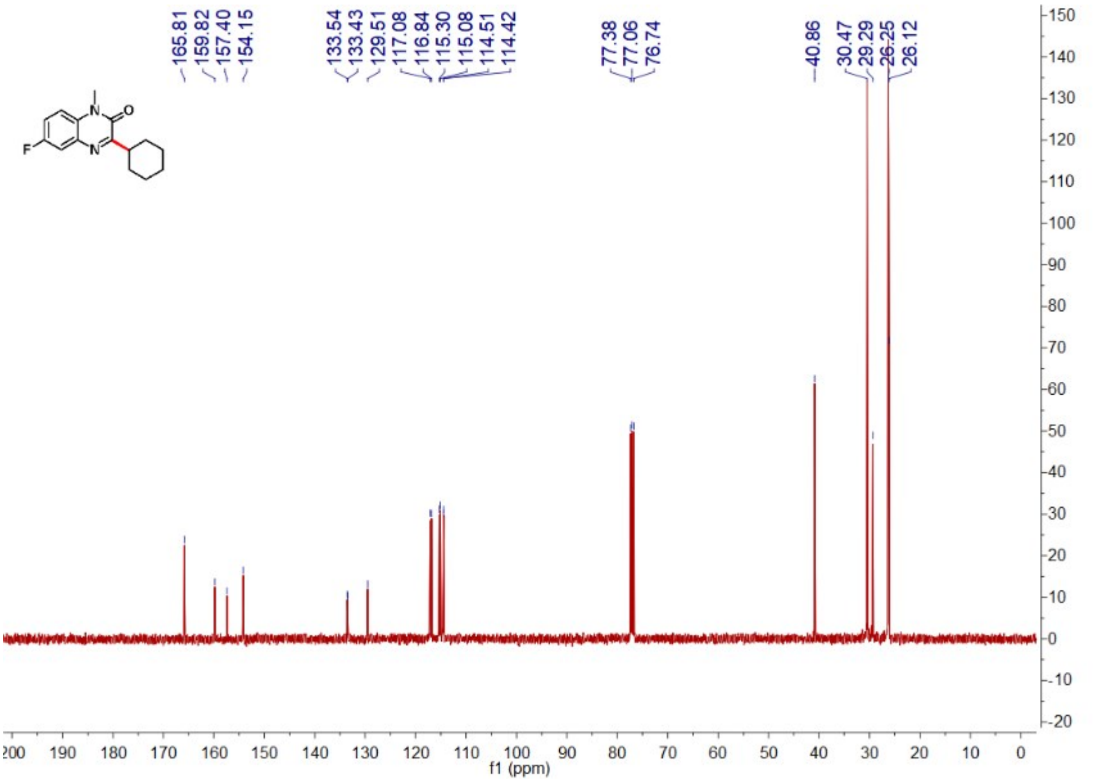
^{13}C NMR spectrum of compound **5**



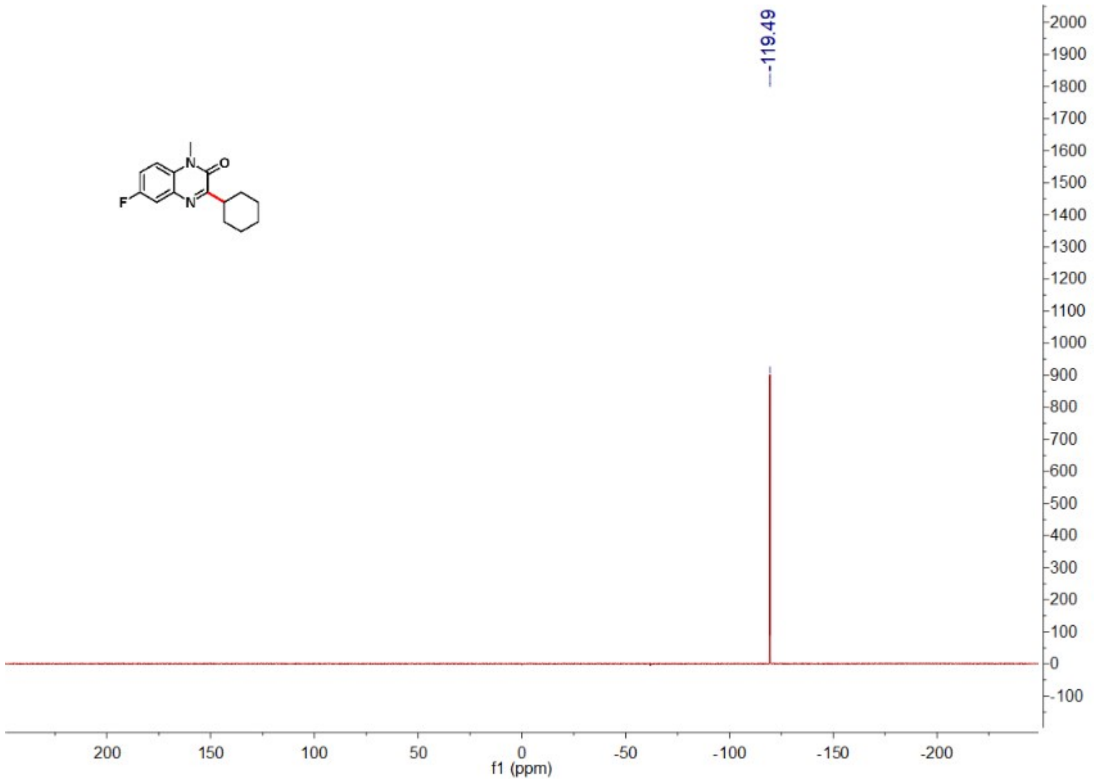
^{1}H NMR spectrum of compound **6**



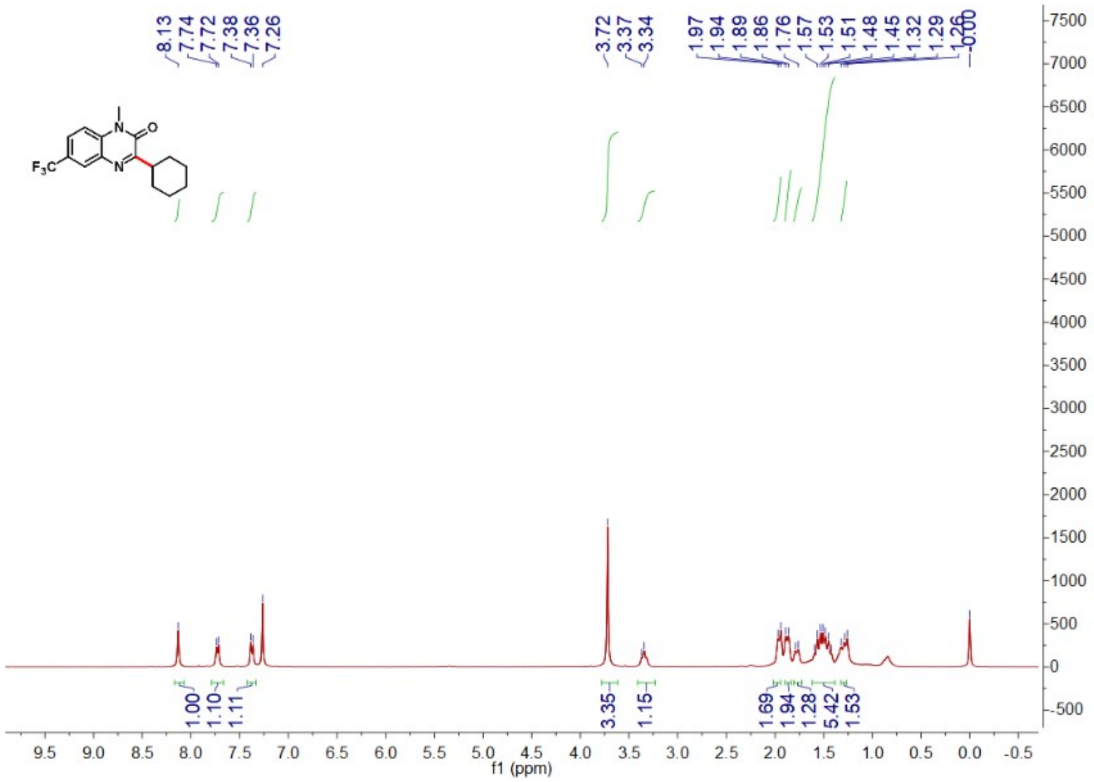
^{13}C NMR spectrum of compound **6**



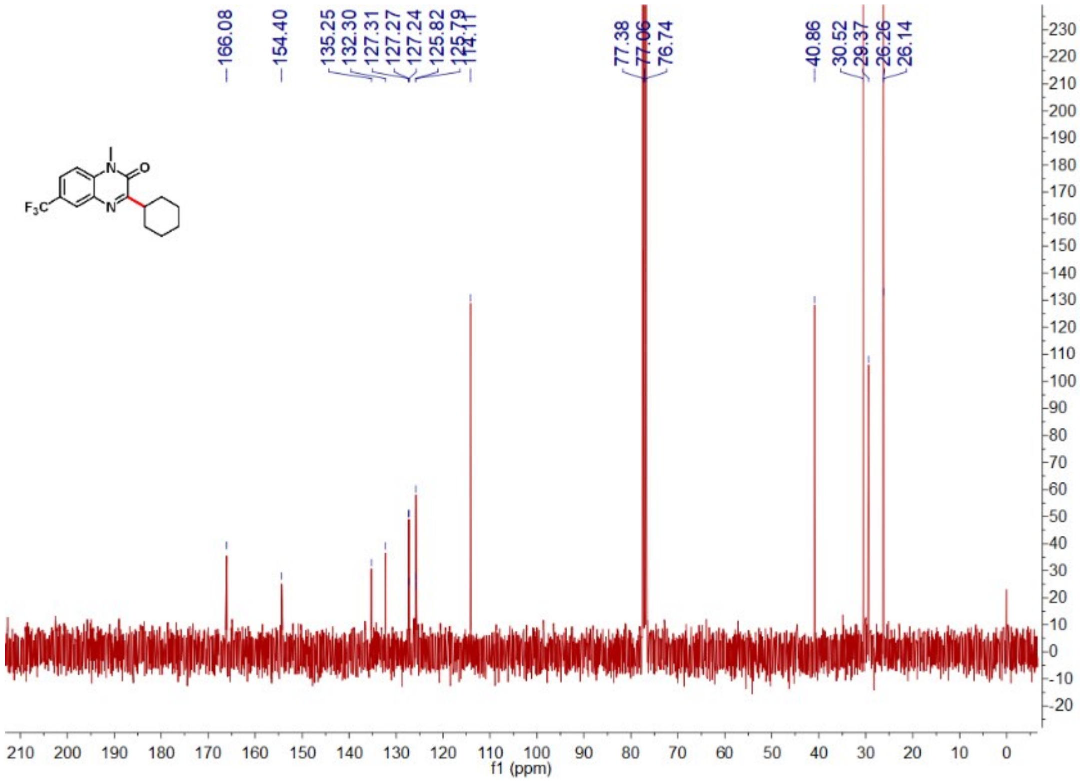
^{19}F NMR spectrum of compound **6**



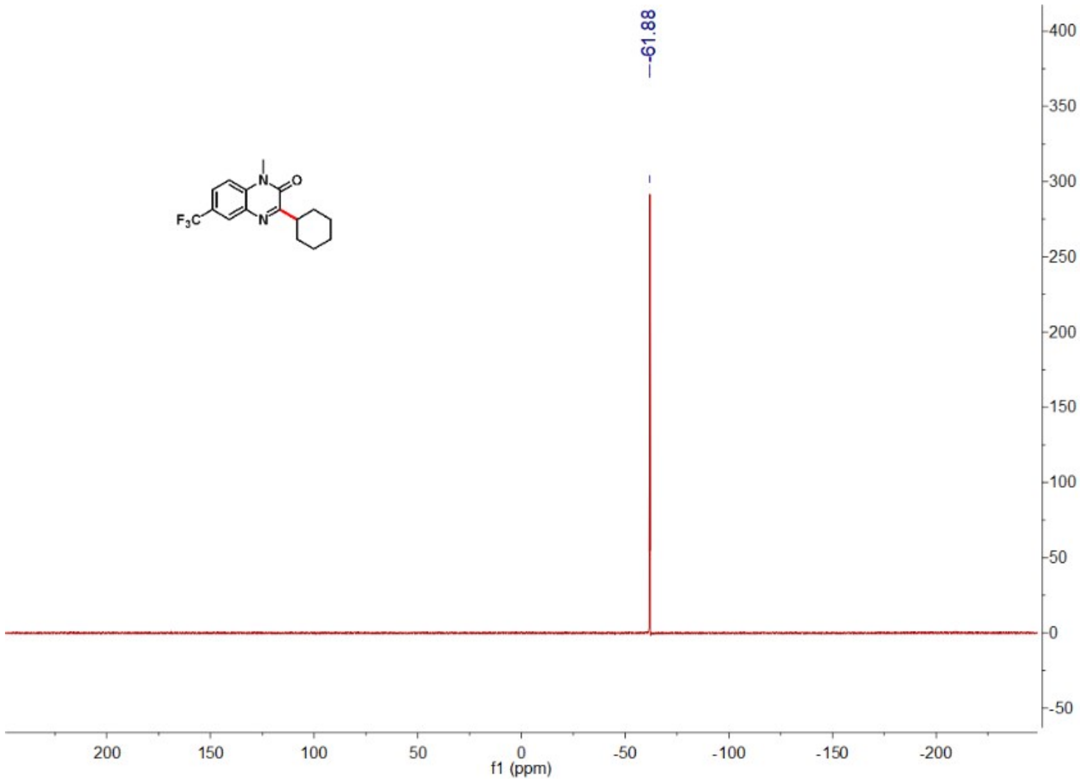
^{1}H NMR spectrum of compound **7**



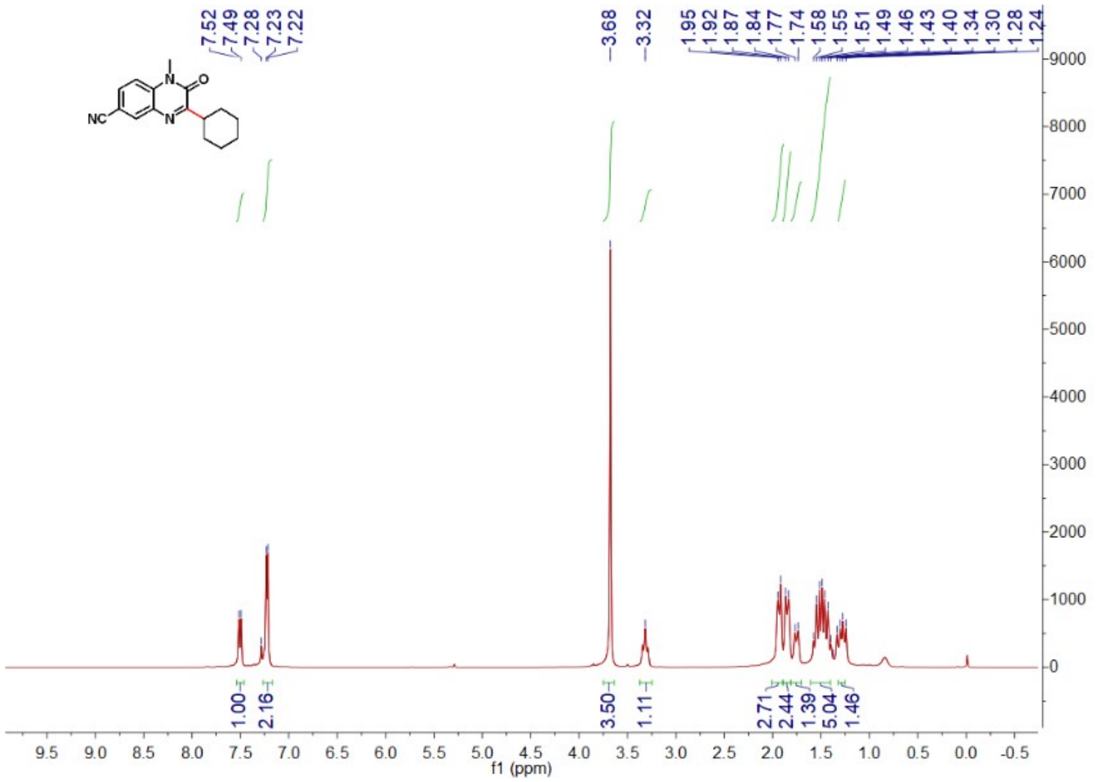
^{13}C NMR spectrum of compound **7**



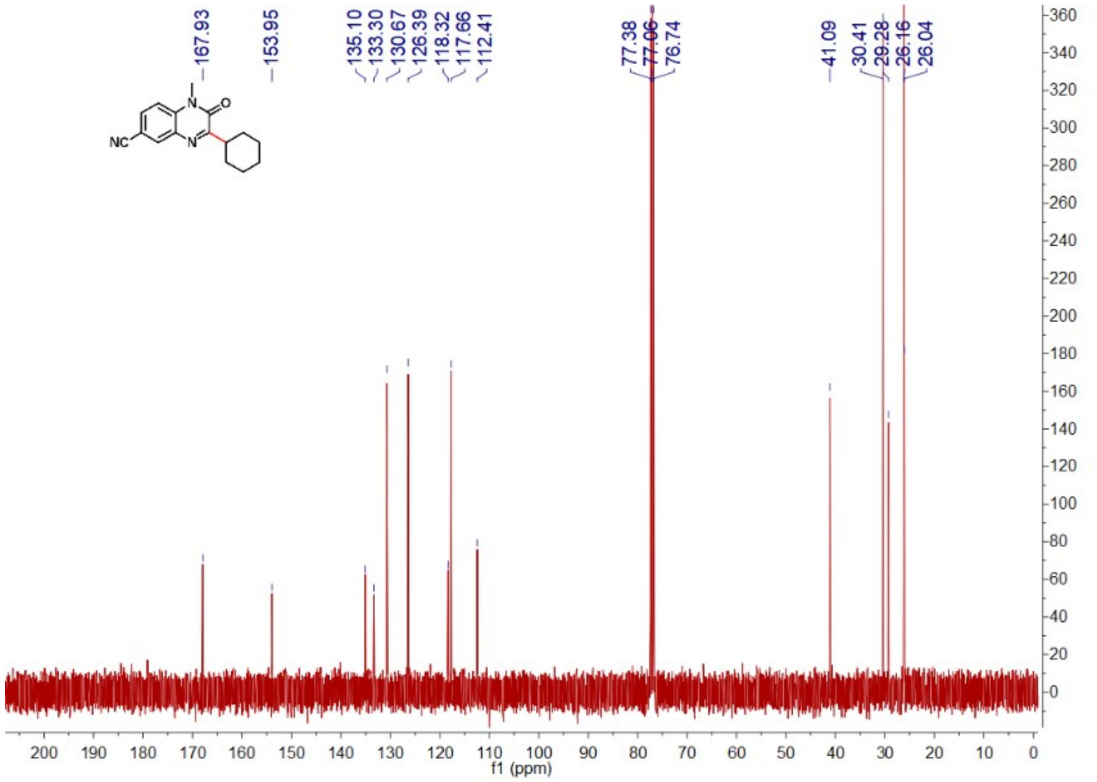
^{19}F NMR spectrum of compound **7**



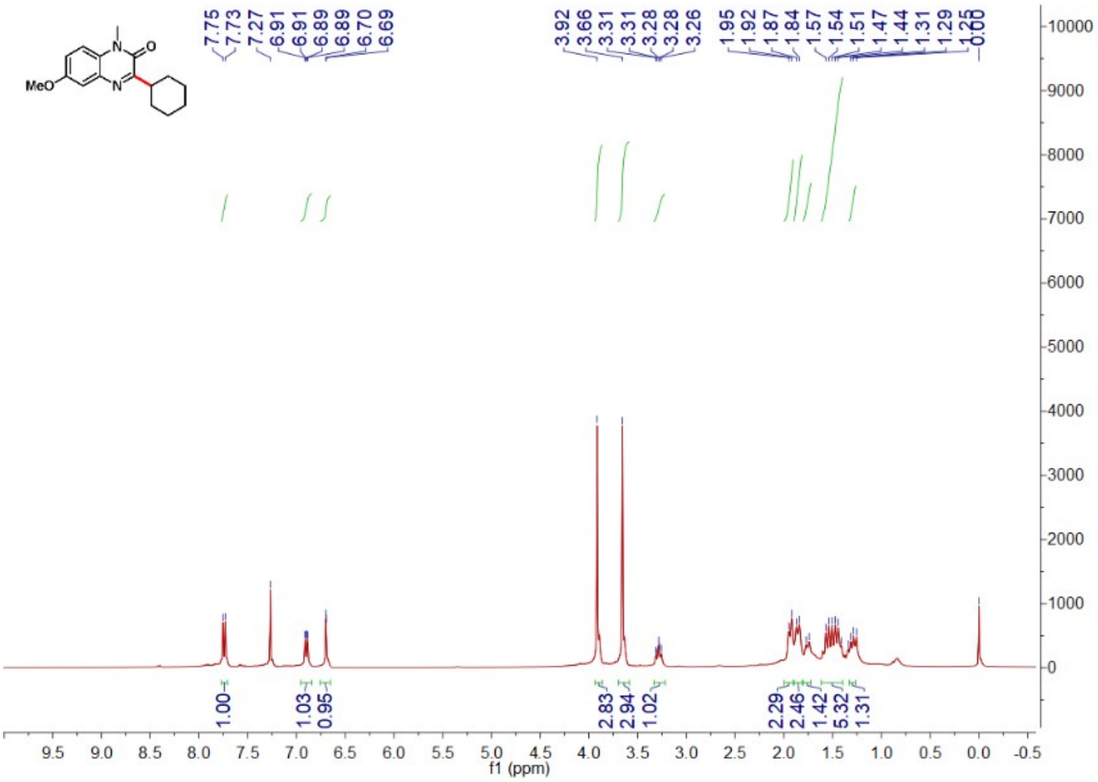
^{1}H NMR spectrum of compound **8**



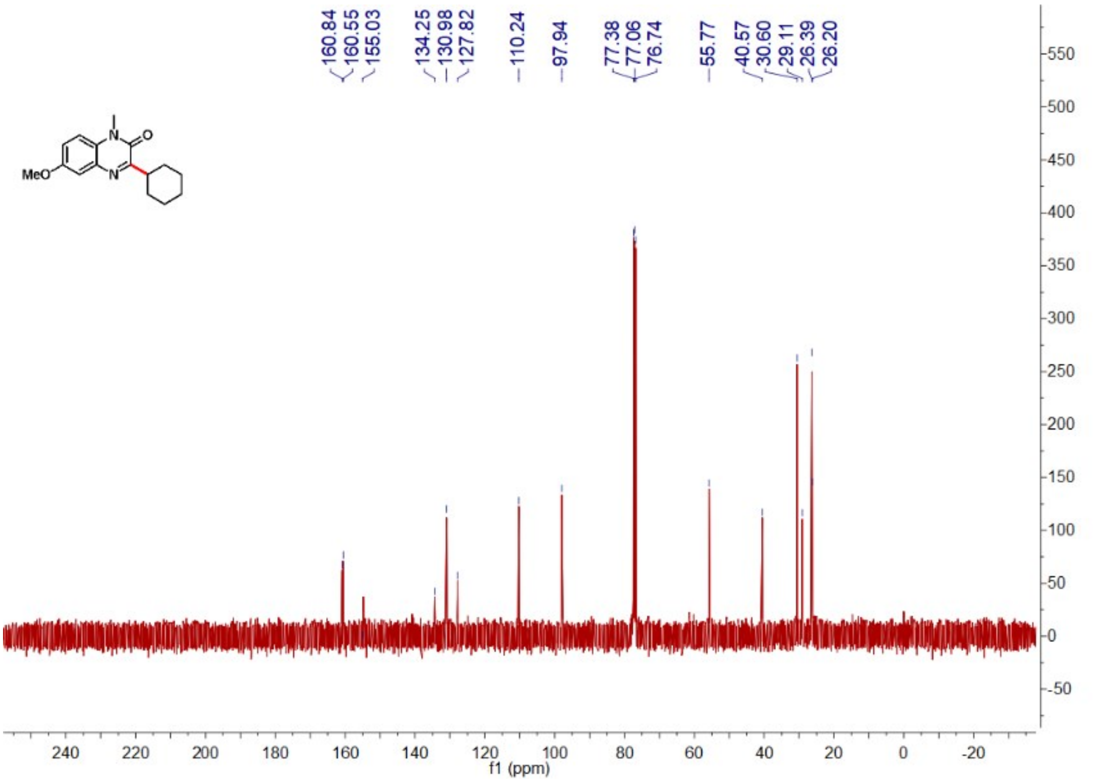
^{13}C NMR spectrum of compound **8**



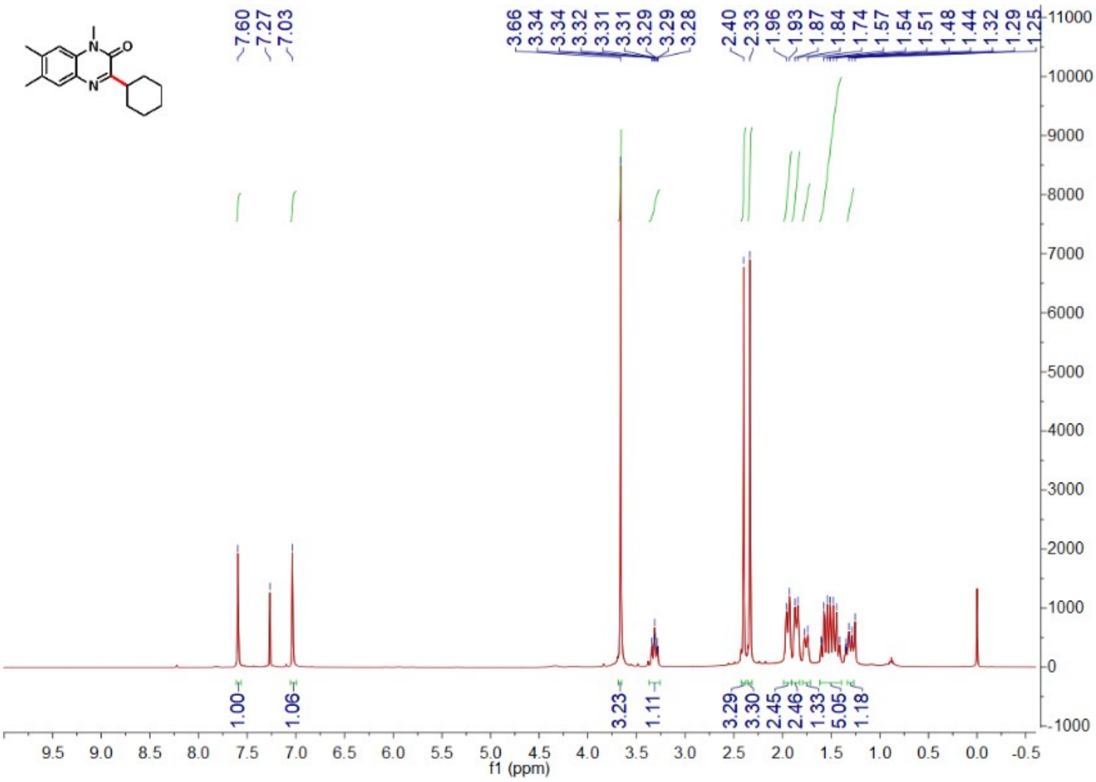
^{1}H NMR spectrum of compound **9**



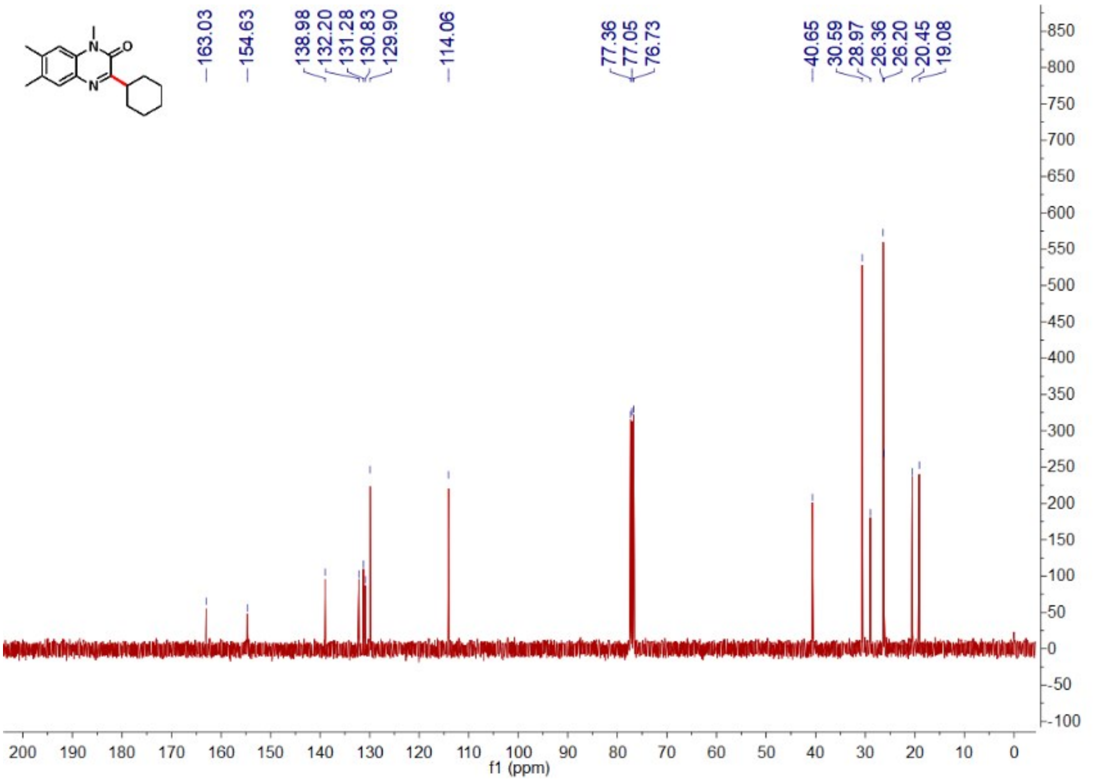
^{13}C NMR spectrum of compound **9**



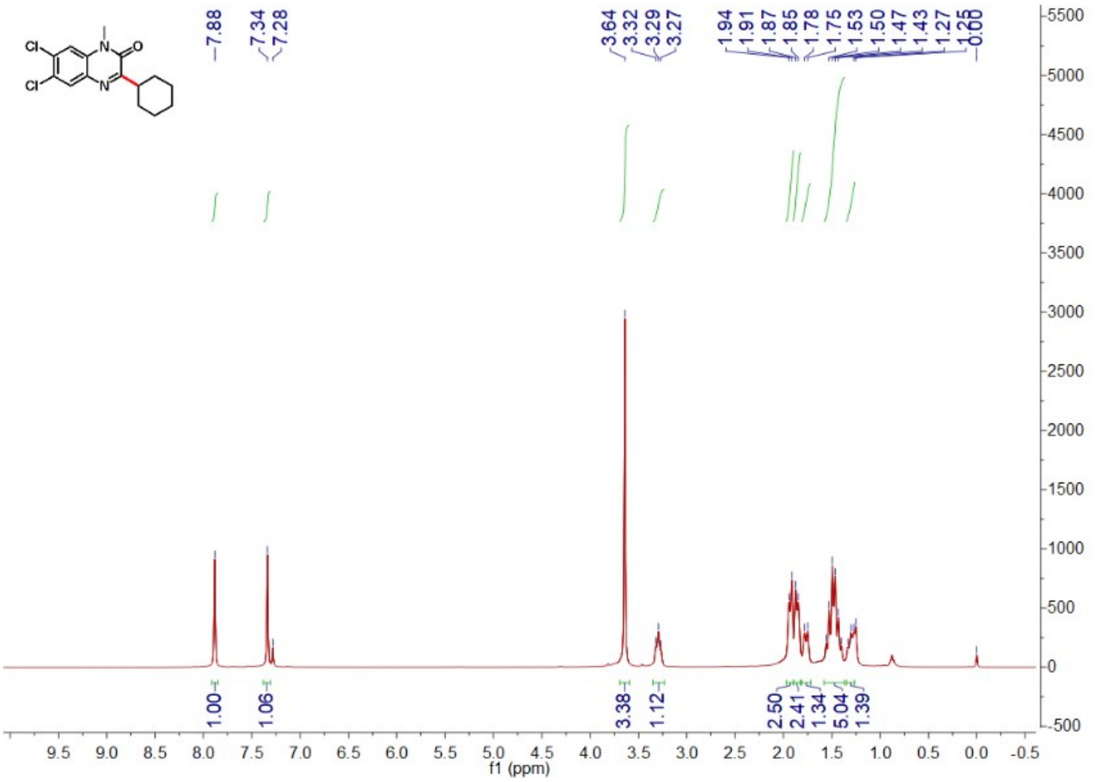
^{1}H NMR spectrum of compound **10**



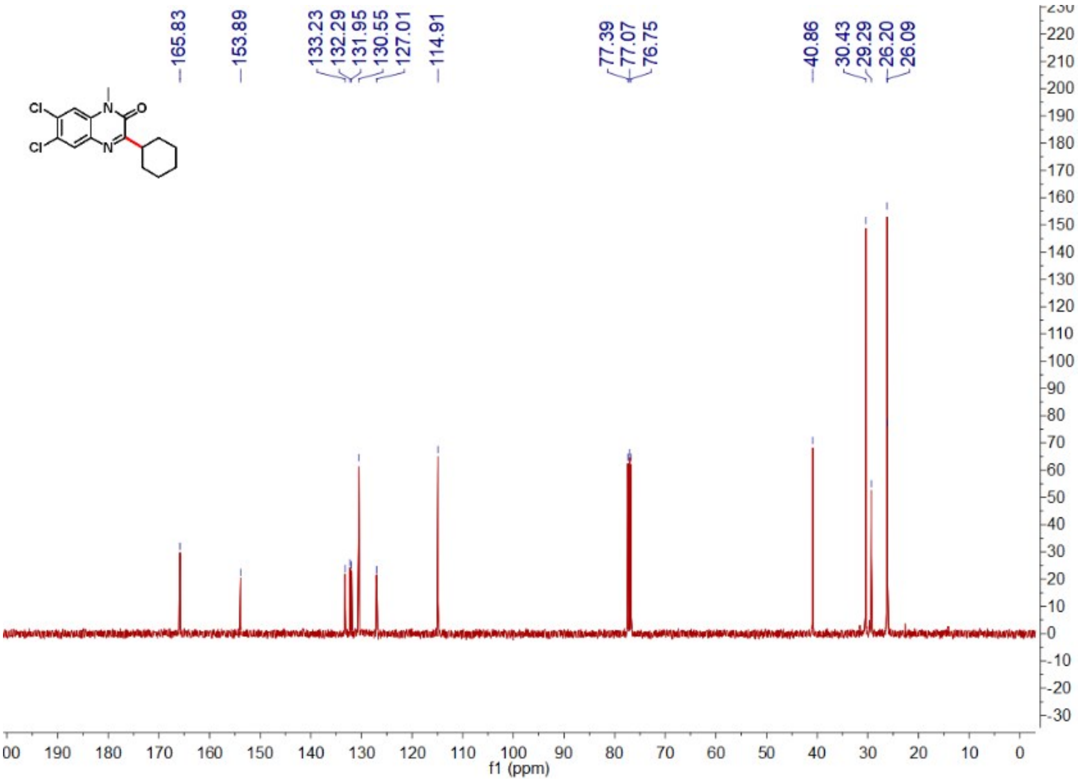
^{13}C NMR spectrum of compound **10**



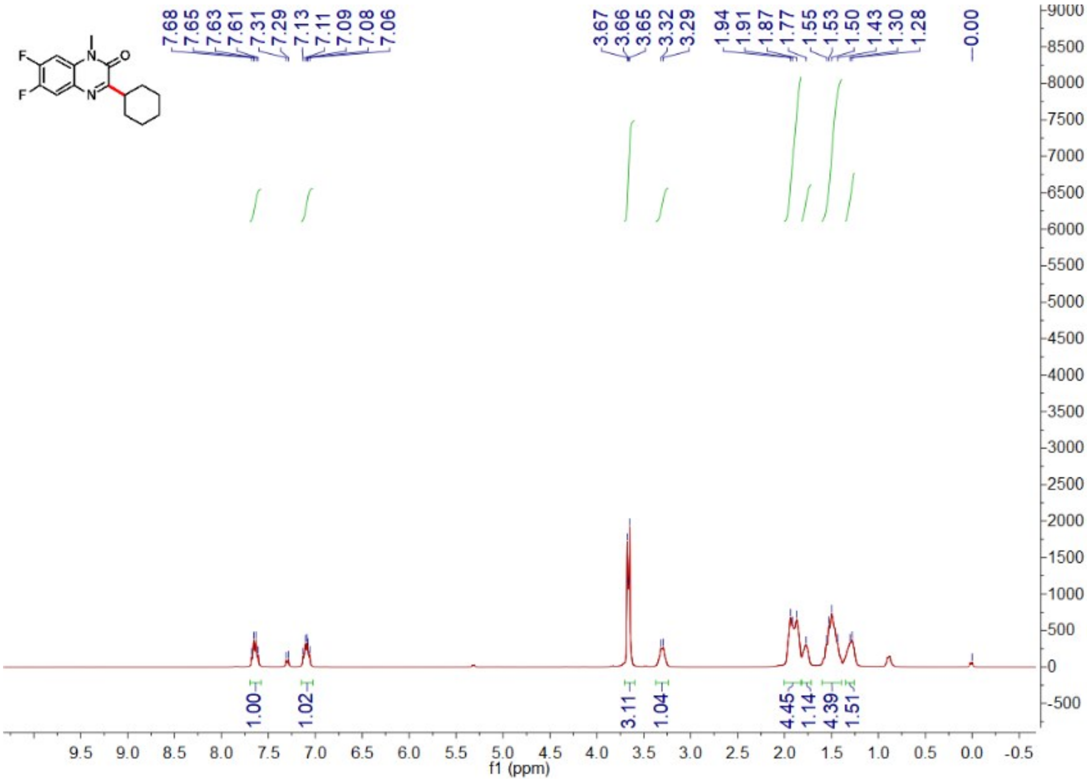
^{1}H NMR spectrum of compound **11**



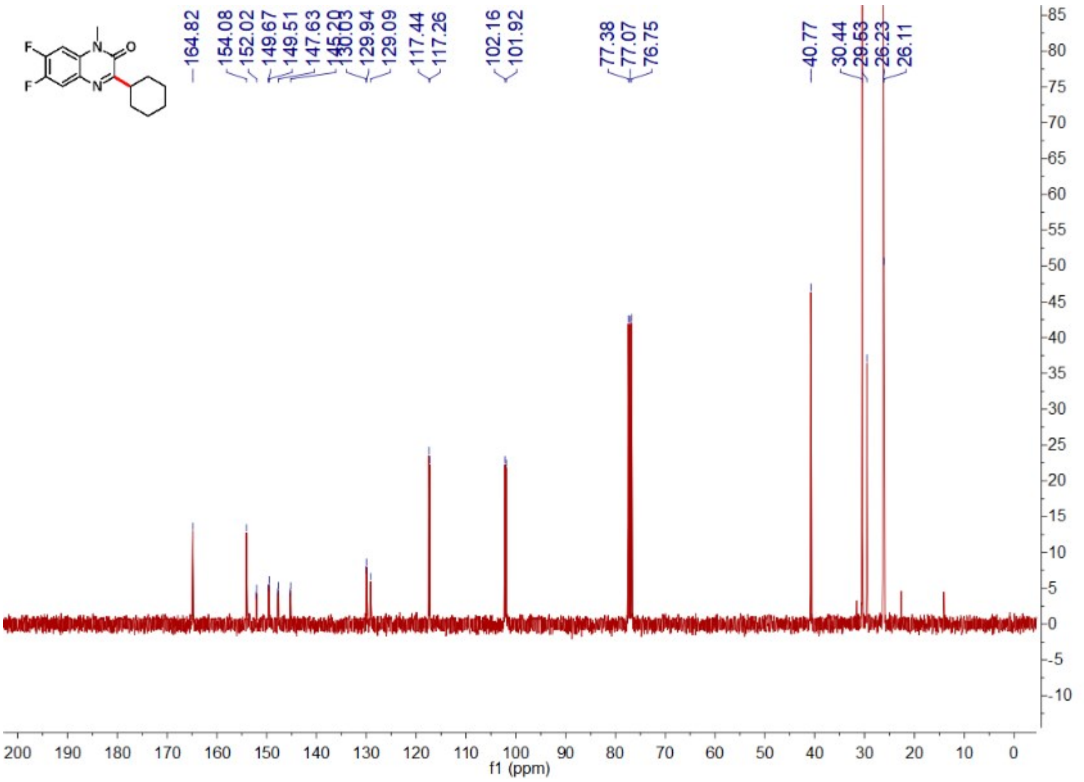
^{13}C NMR spectrum of compound **11**



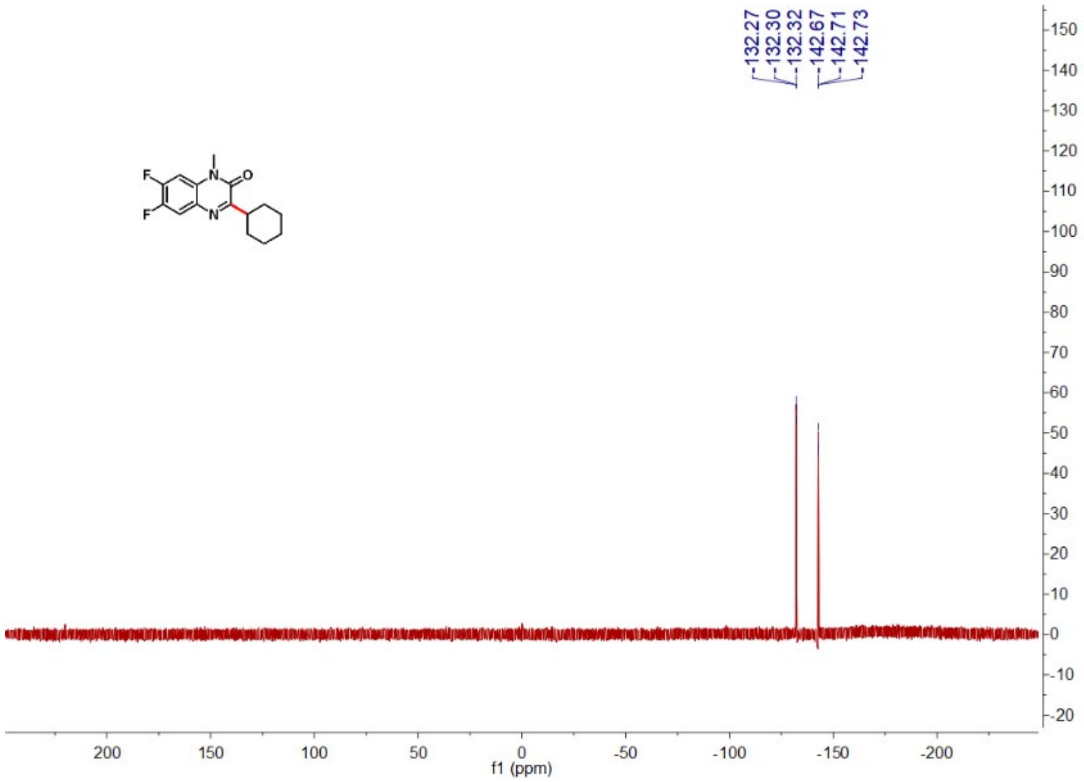
^{1}H NMR spectrum of compound **12**



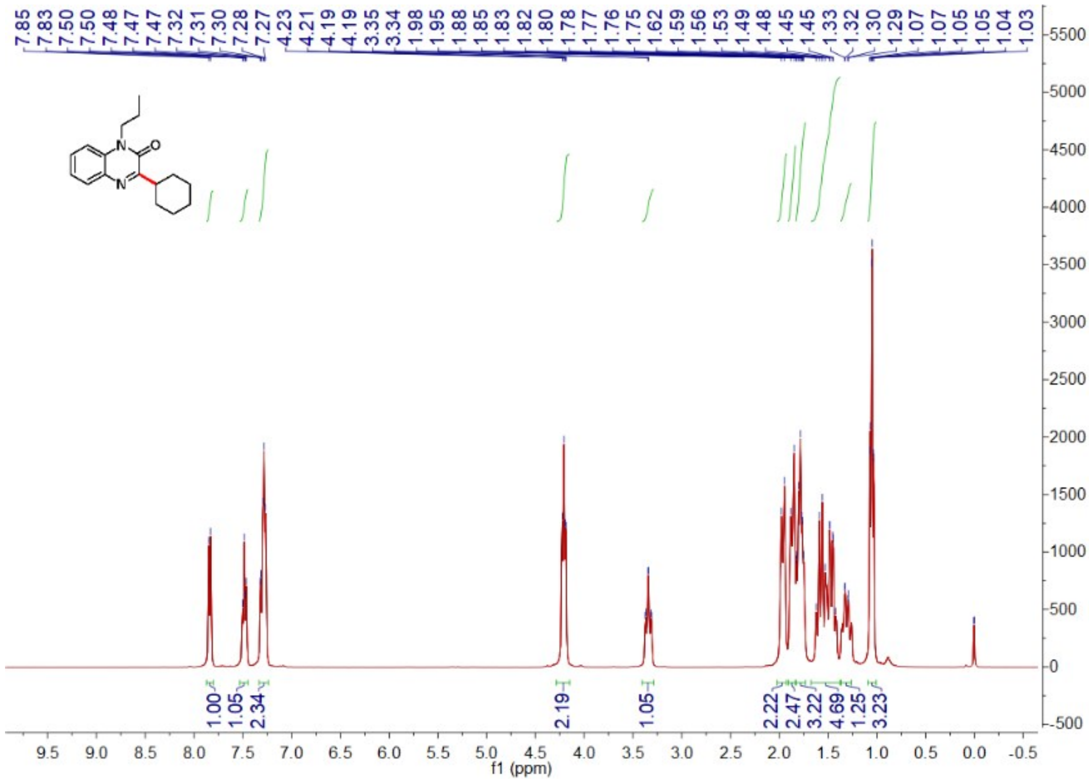
^{13}C NMR spectrum of compound **12**



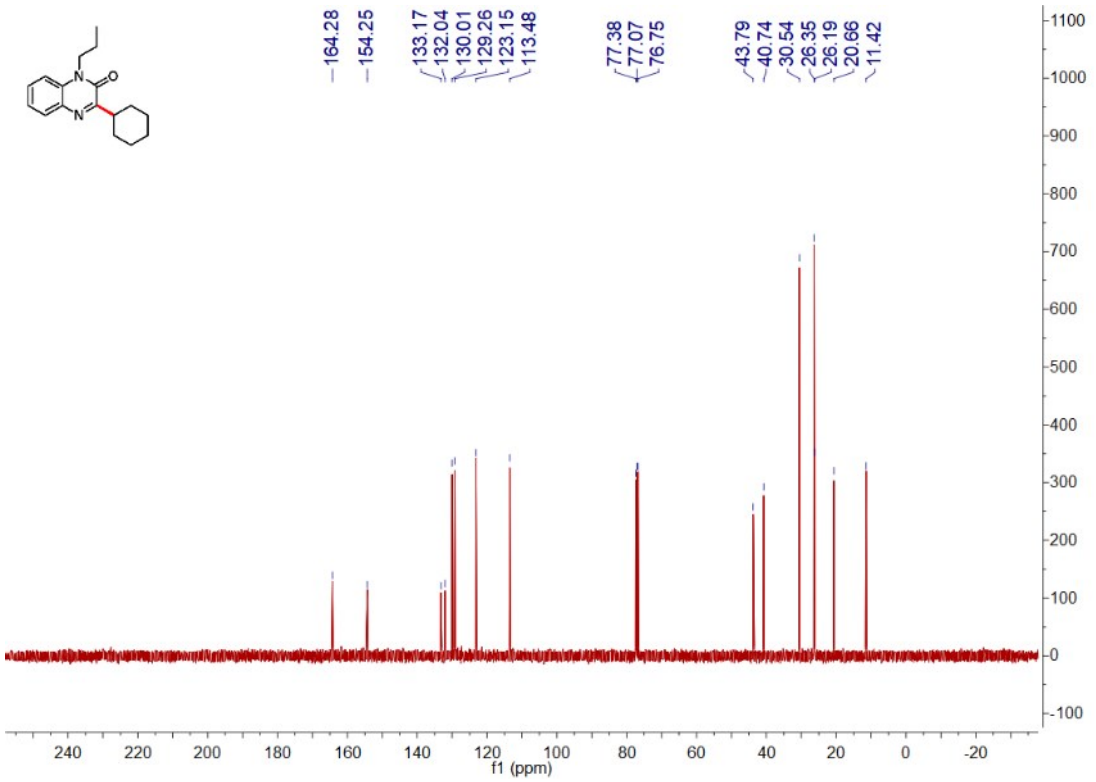
^{19}F NMR spectrum of compound **12**



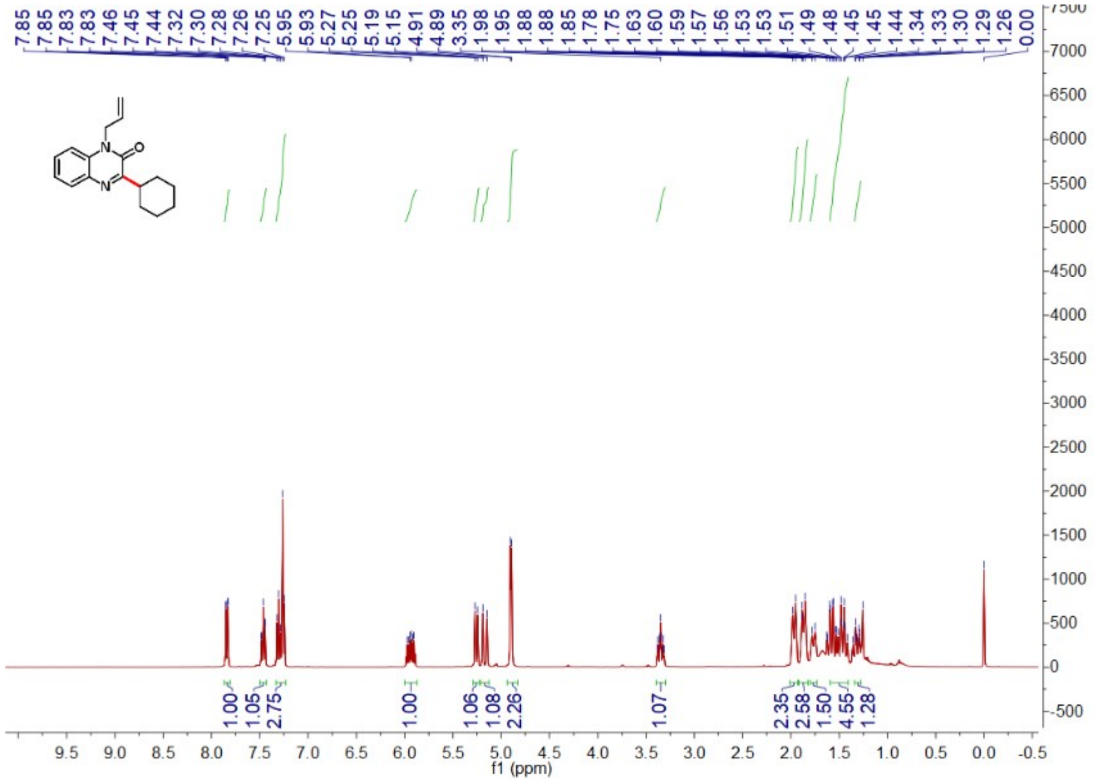
^{1}H NMR spectrum of compound **13**



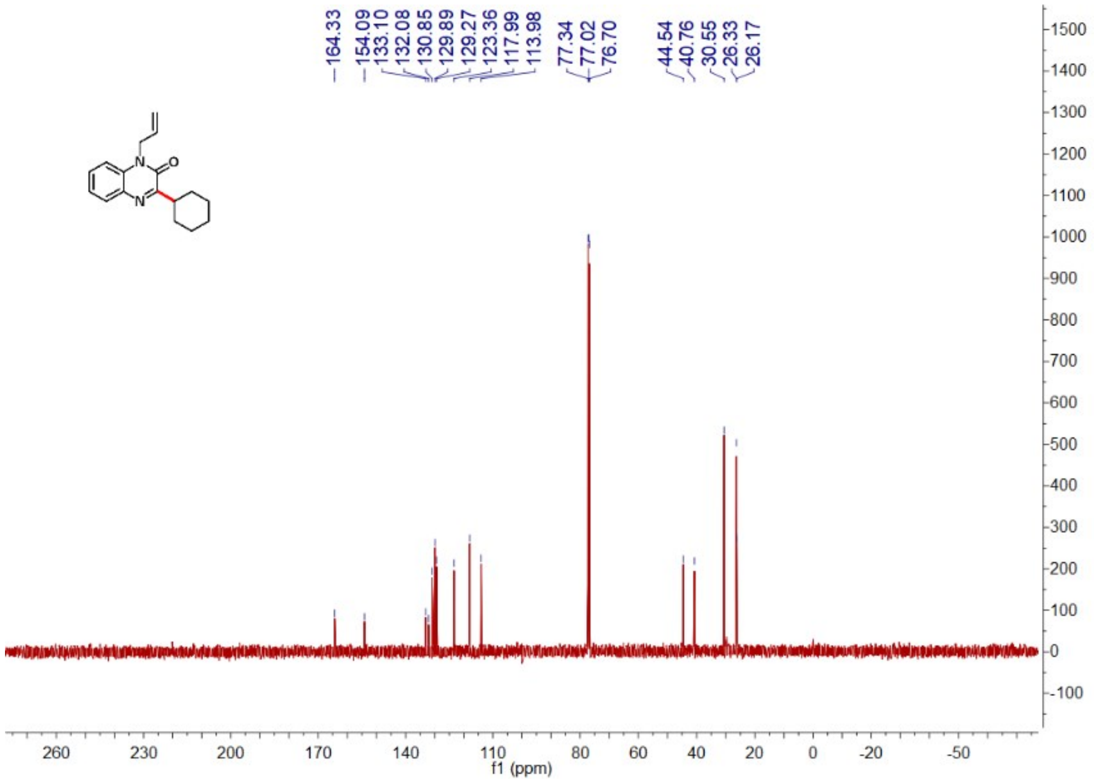
^{13}C NMR spectrum of compound **13**



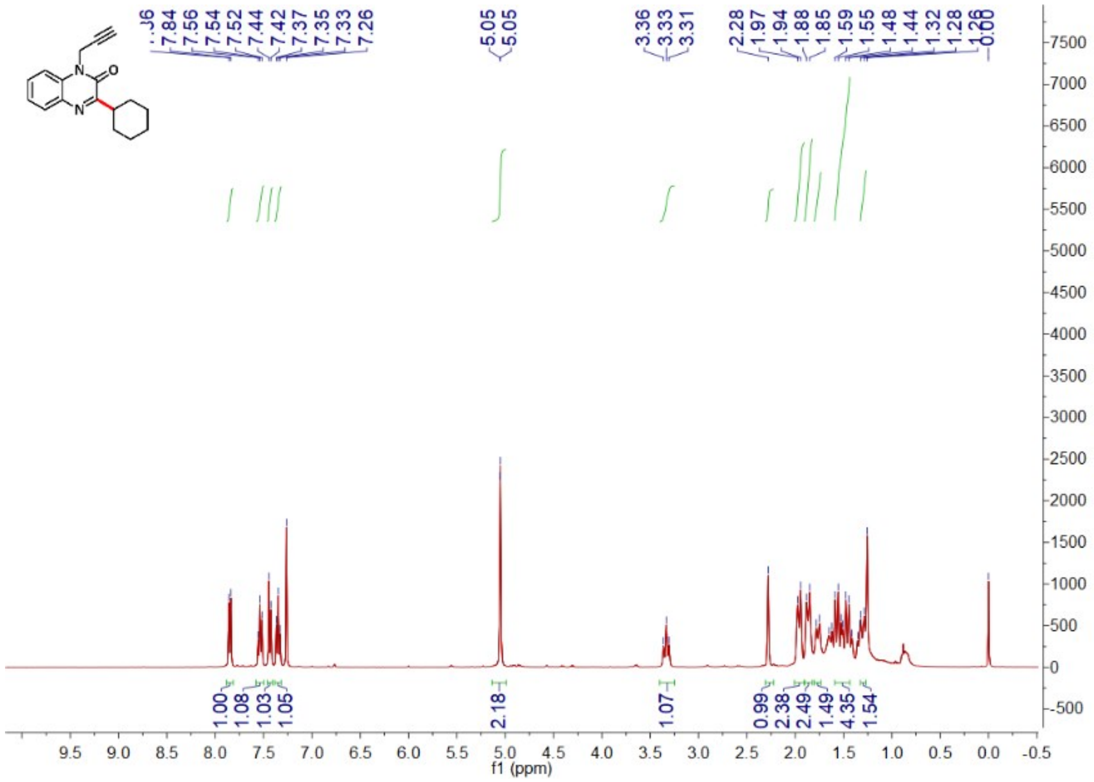
^{1}H NMR spectrum of compound **14**



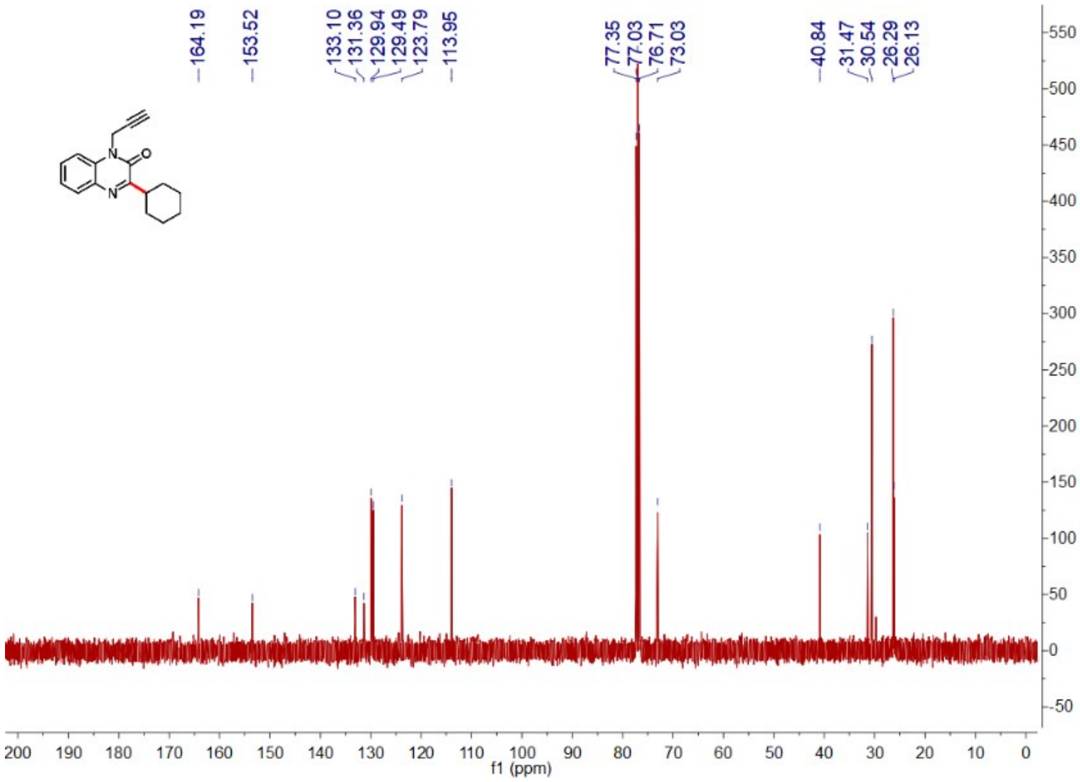
^{13}C NMR spectrum of compound **14**



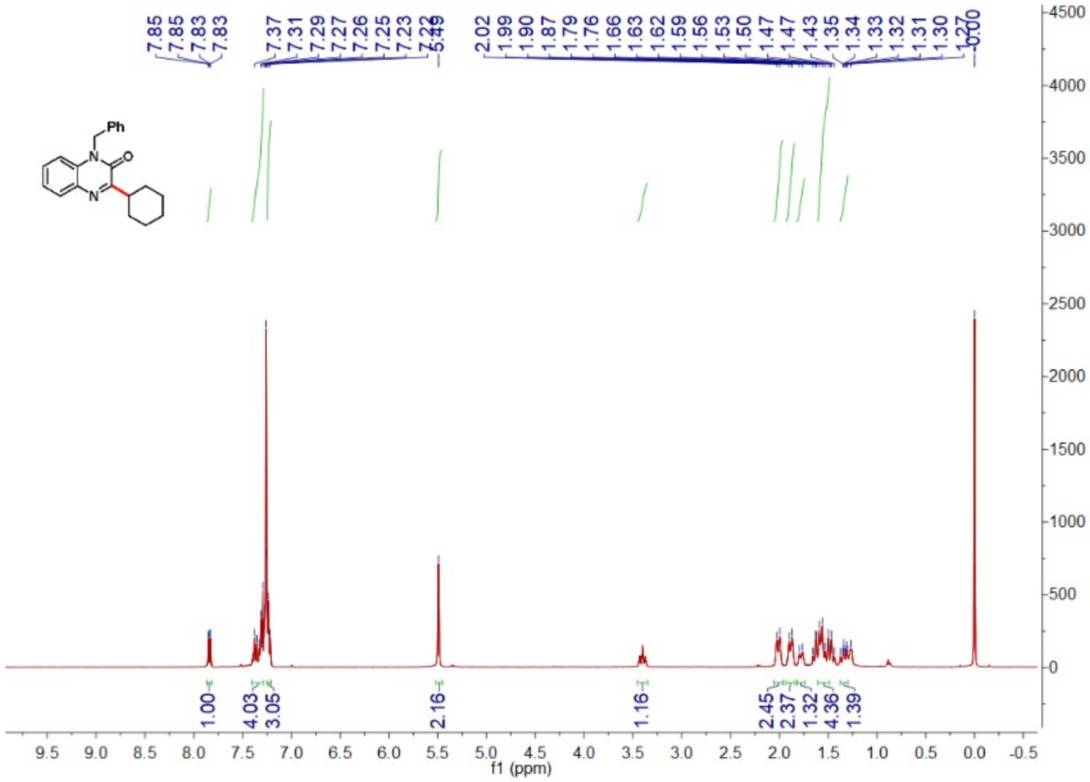
^{1}H NMR spectrum of compound **15**



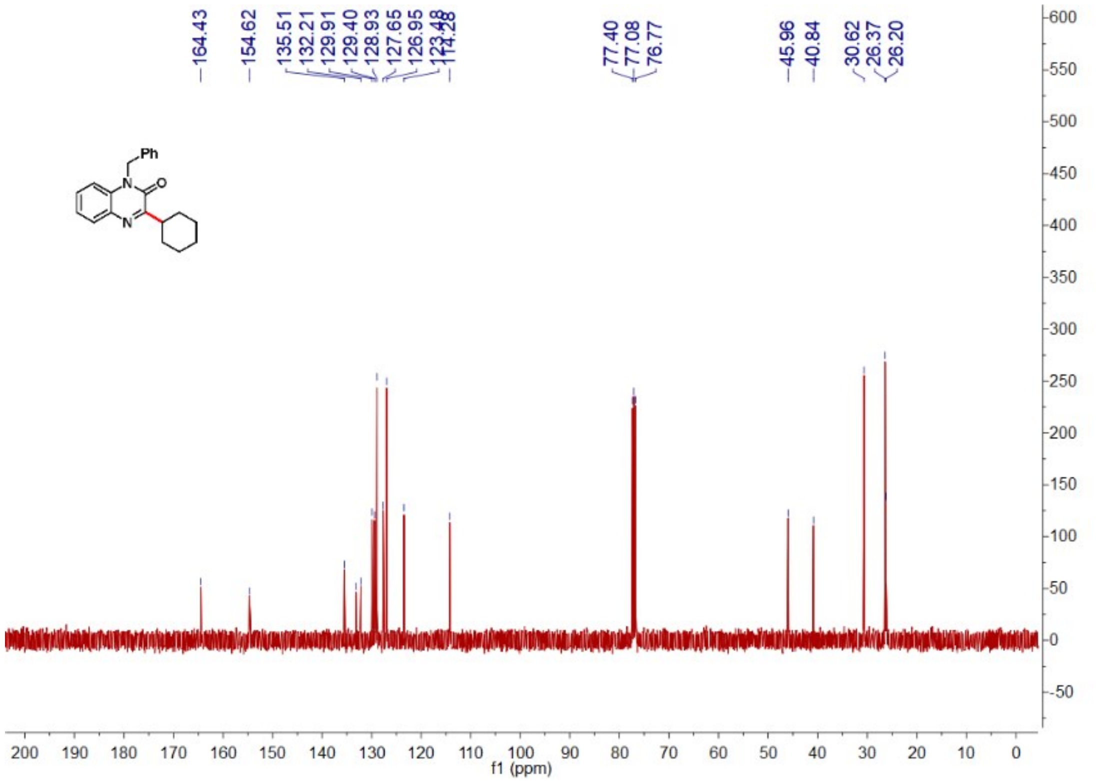
^{13}C NMR spectrum of compound **15**



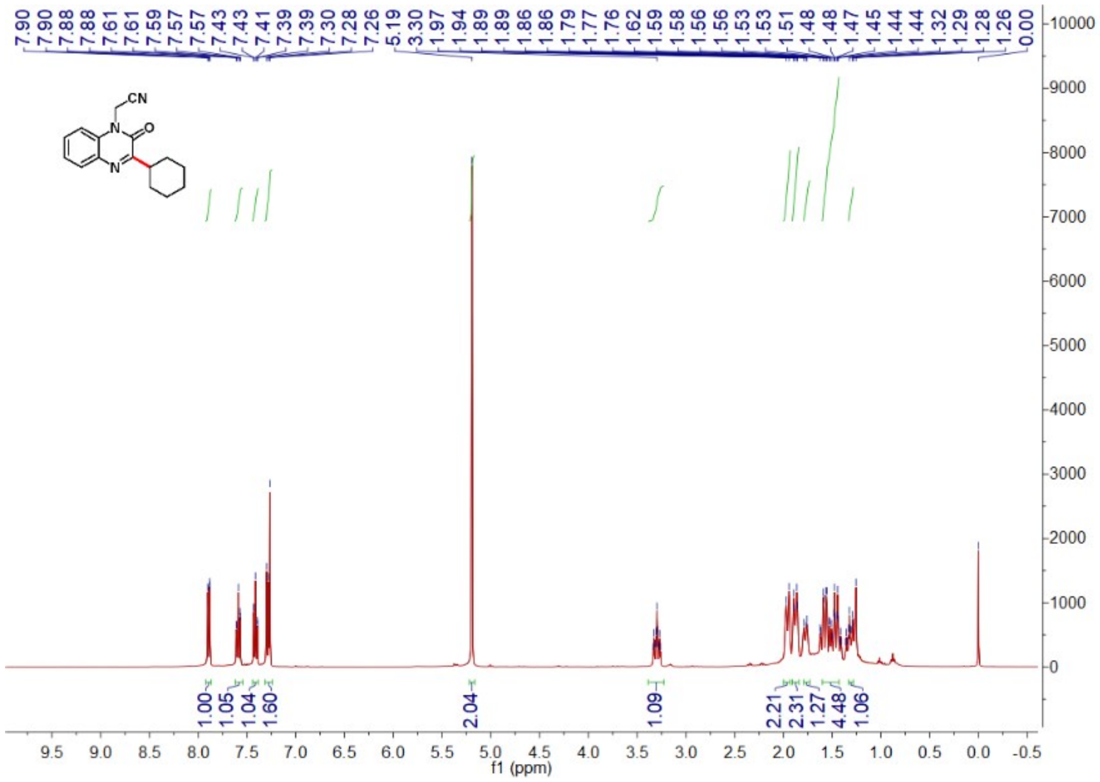
^{1}H NMR spectrum of compound **16**



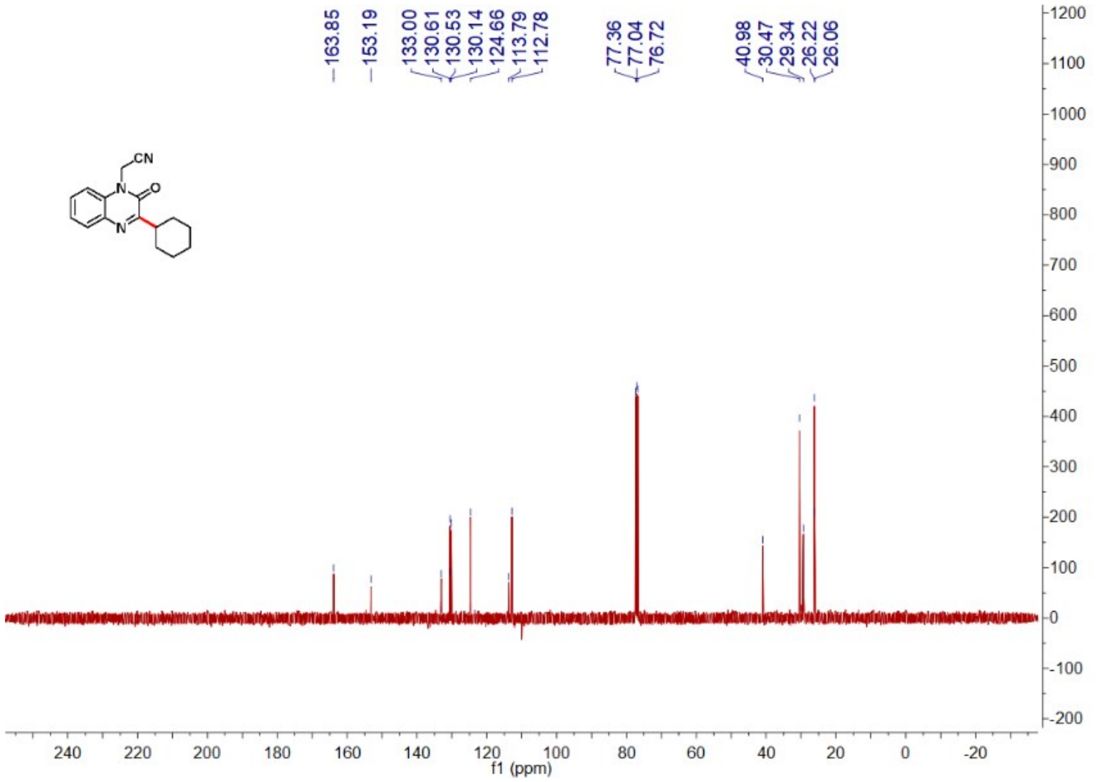
^{13}C NMR spectrum of compound **16**



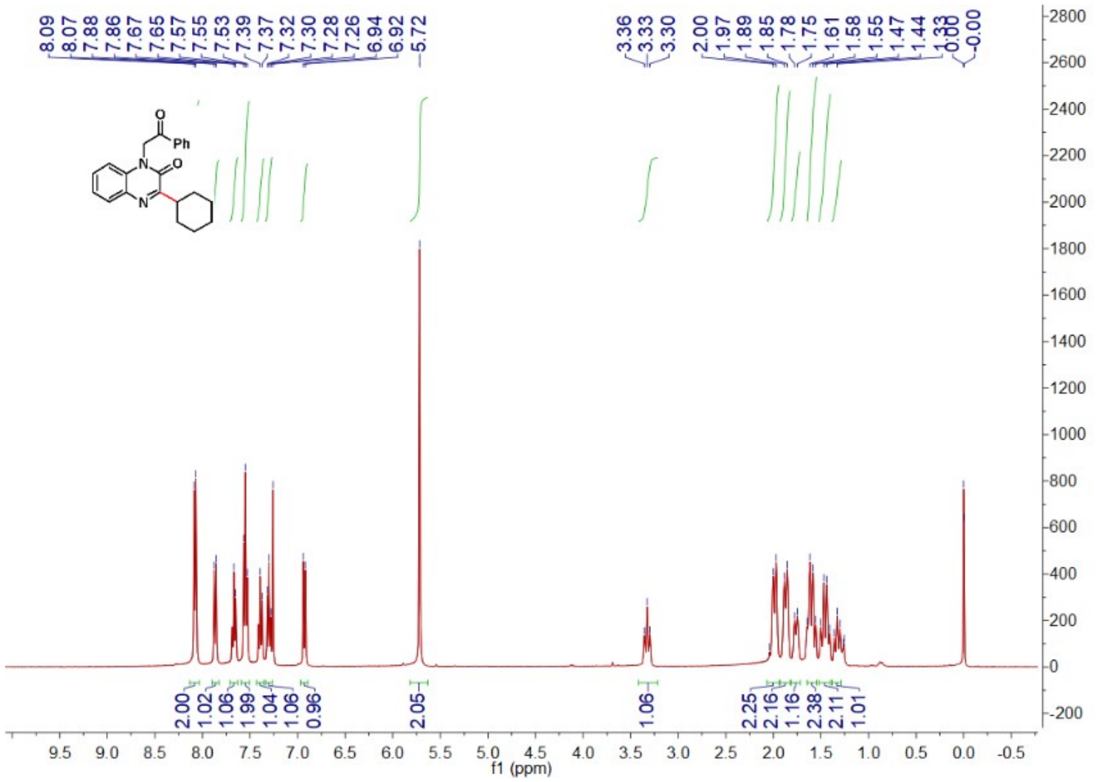
^{1}H NMR spectrum of compound **17**



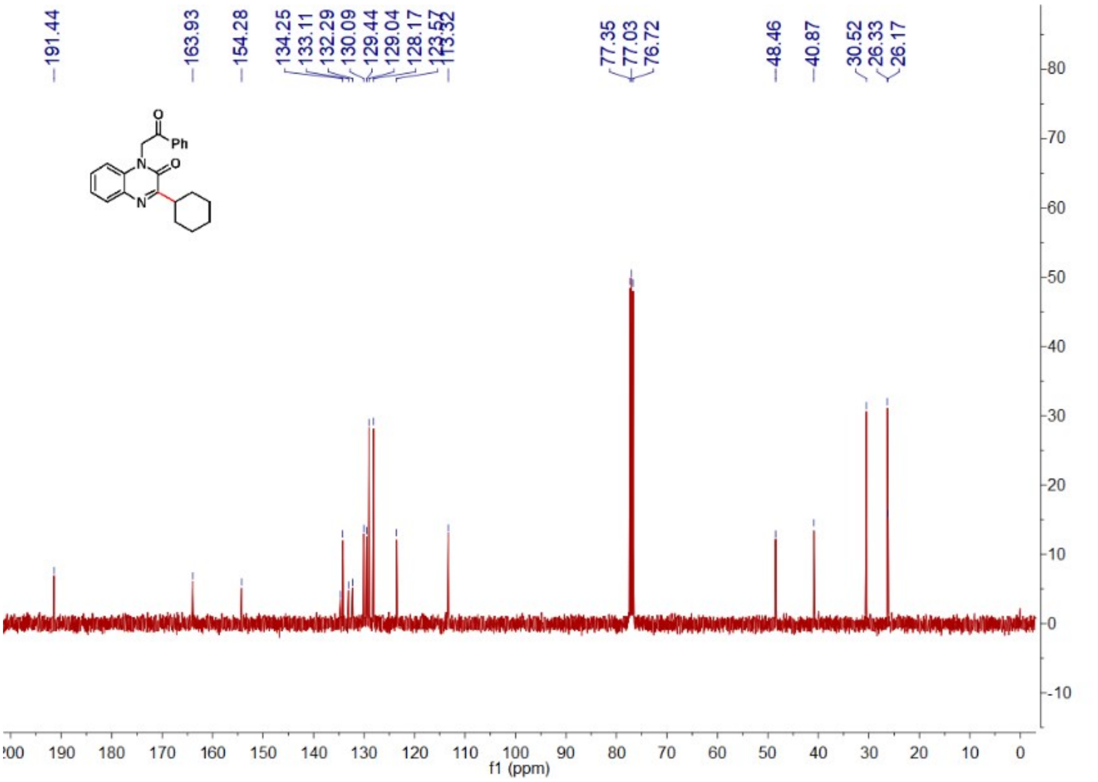
^{13}C NMR spectrum of compound **17**



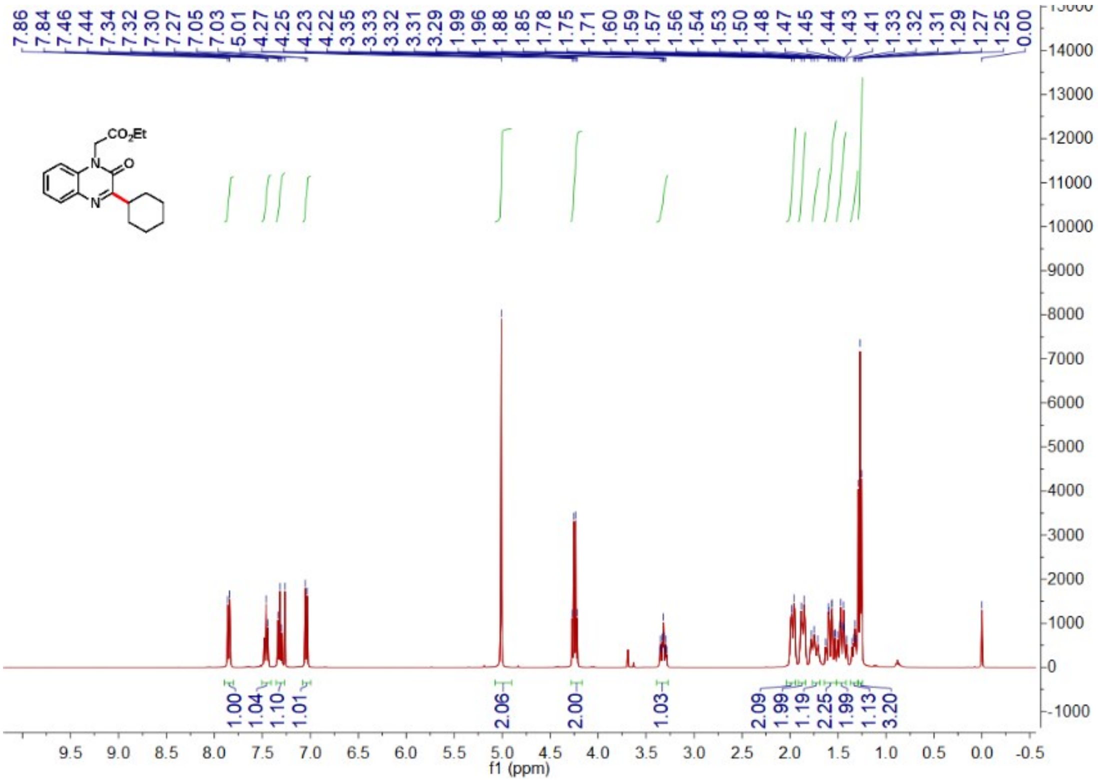
^{1}H NMR spectrum of compound **18**



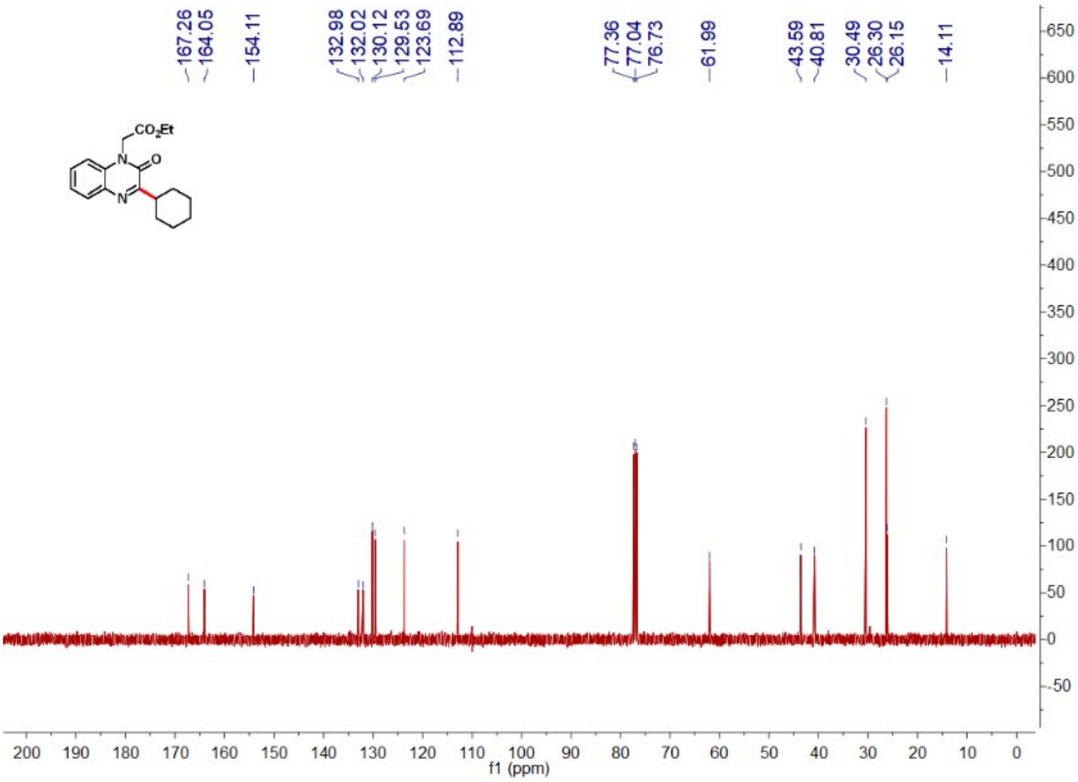
^{13}C NMR spectrum of compound **18**



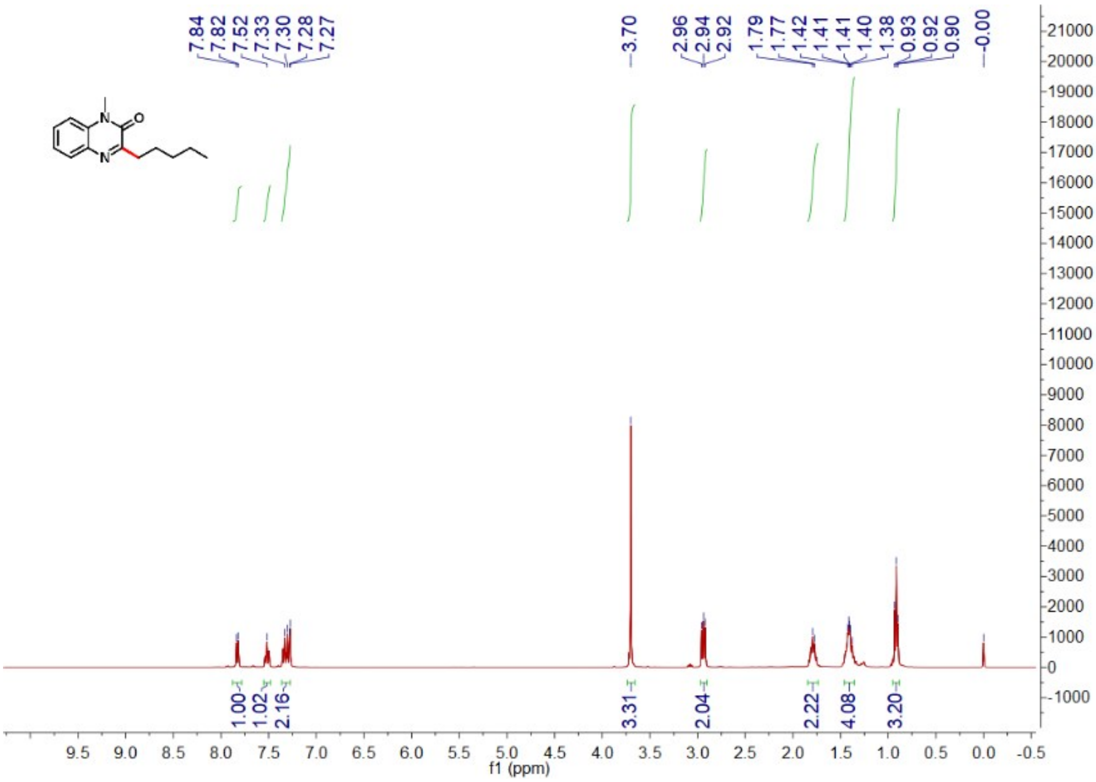
^{1}H NMR spectrum of compound **19**



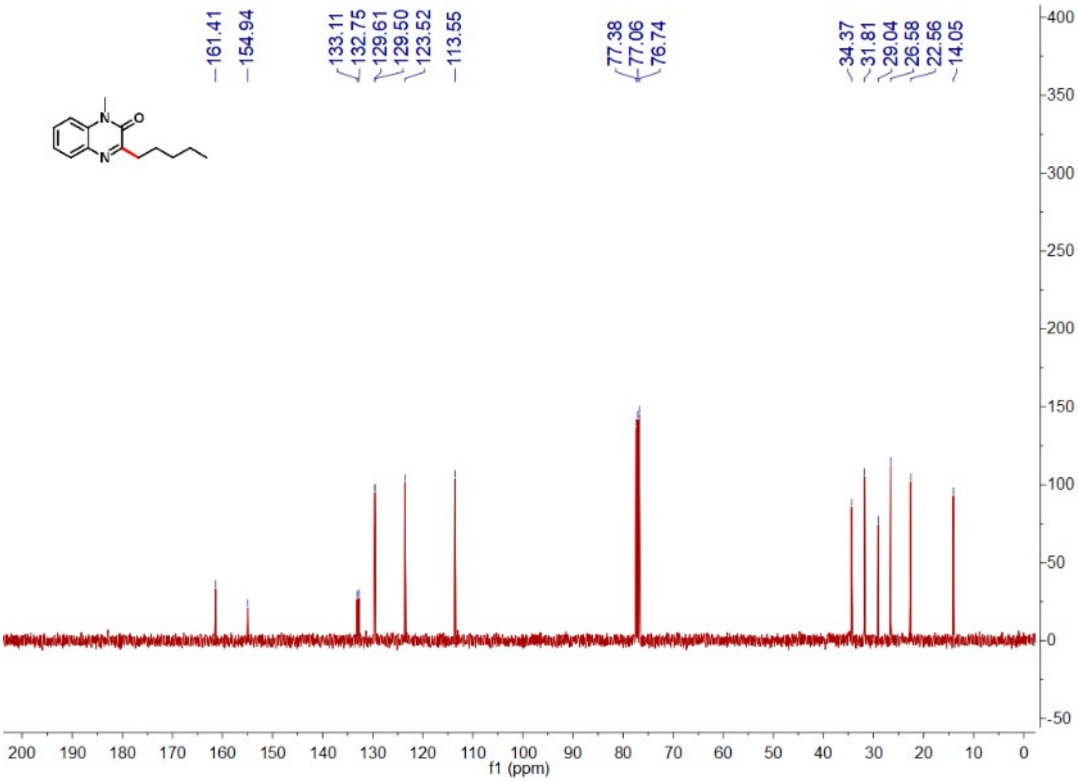
¹³C NMR spectrum of compound **19**



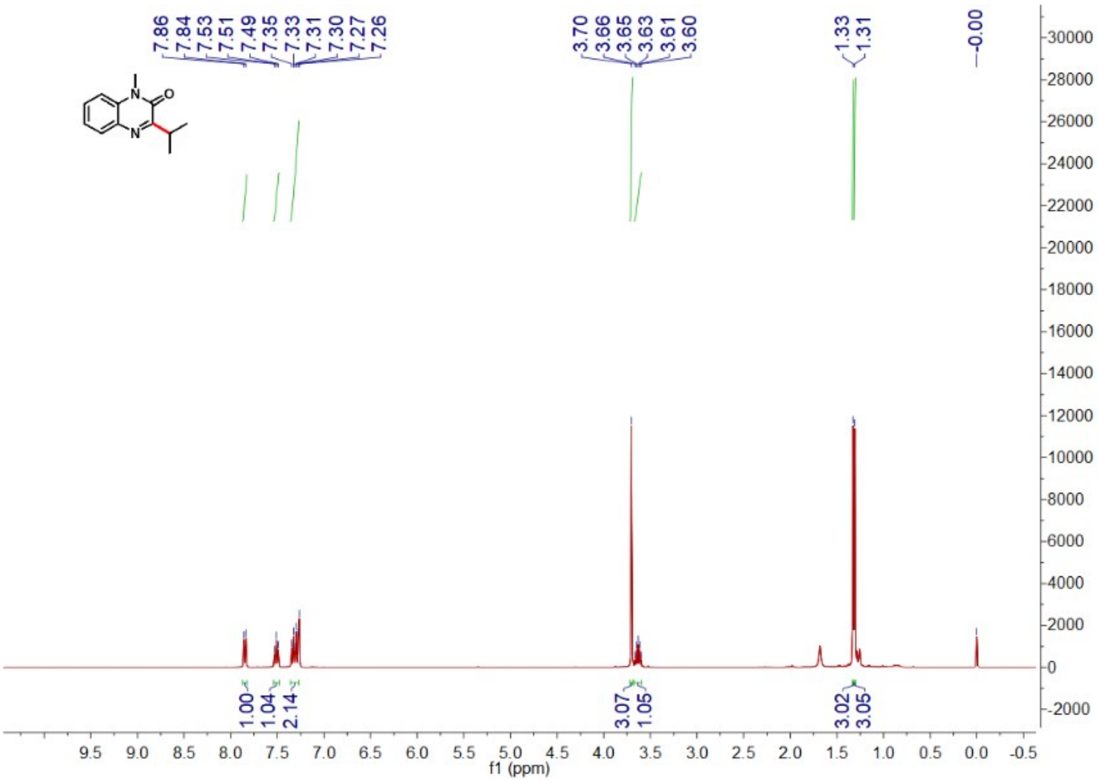
¹H NMR spectrum of compound **20**



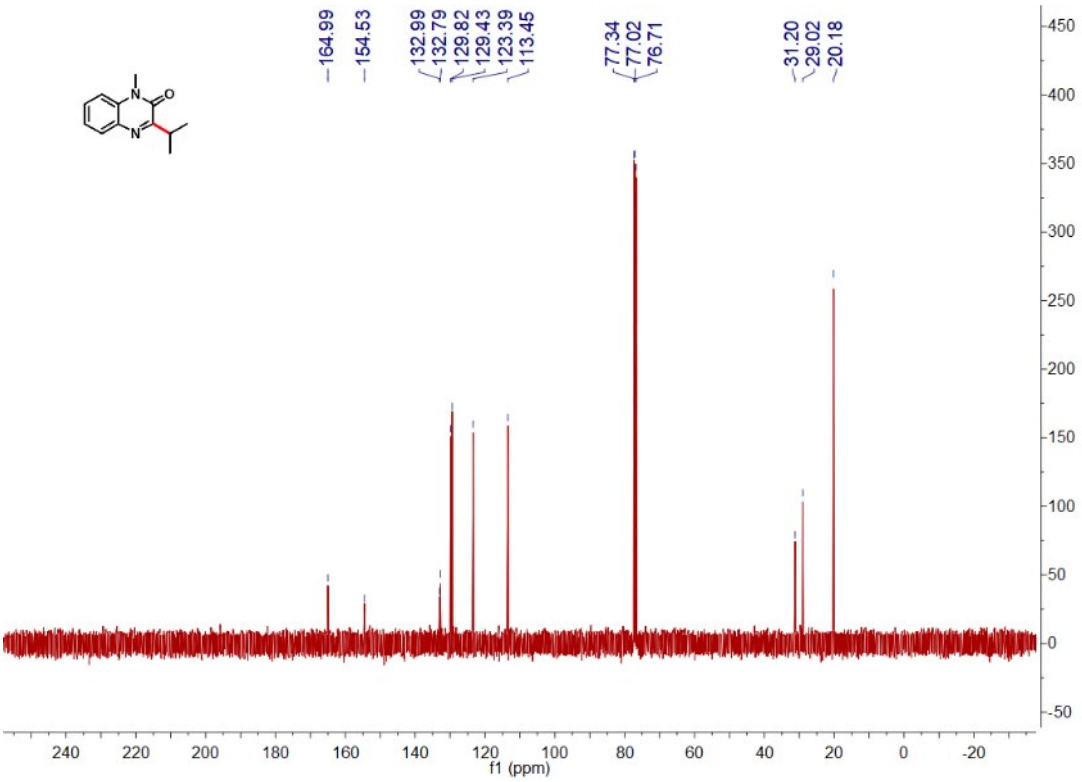
^{13}C NMR spectrum of compound **20**



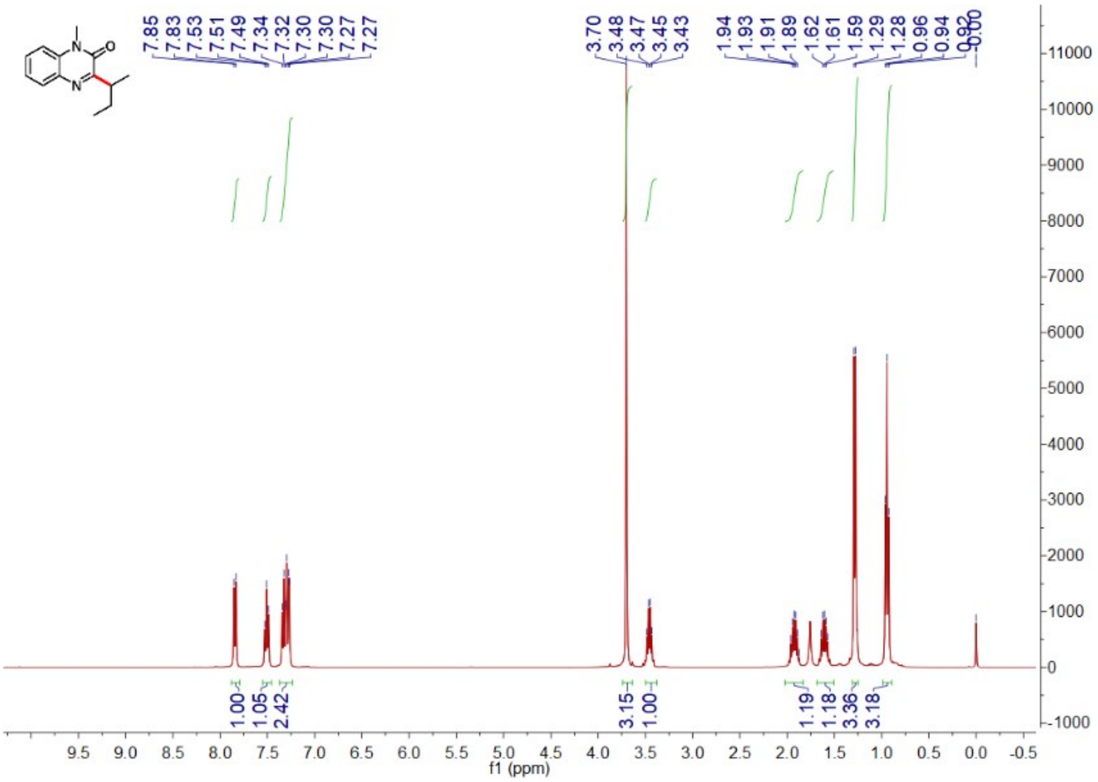
^{1}H NMR spectrum of compound **21**



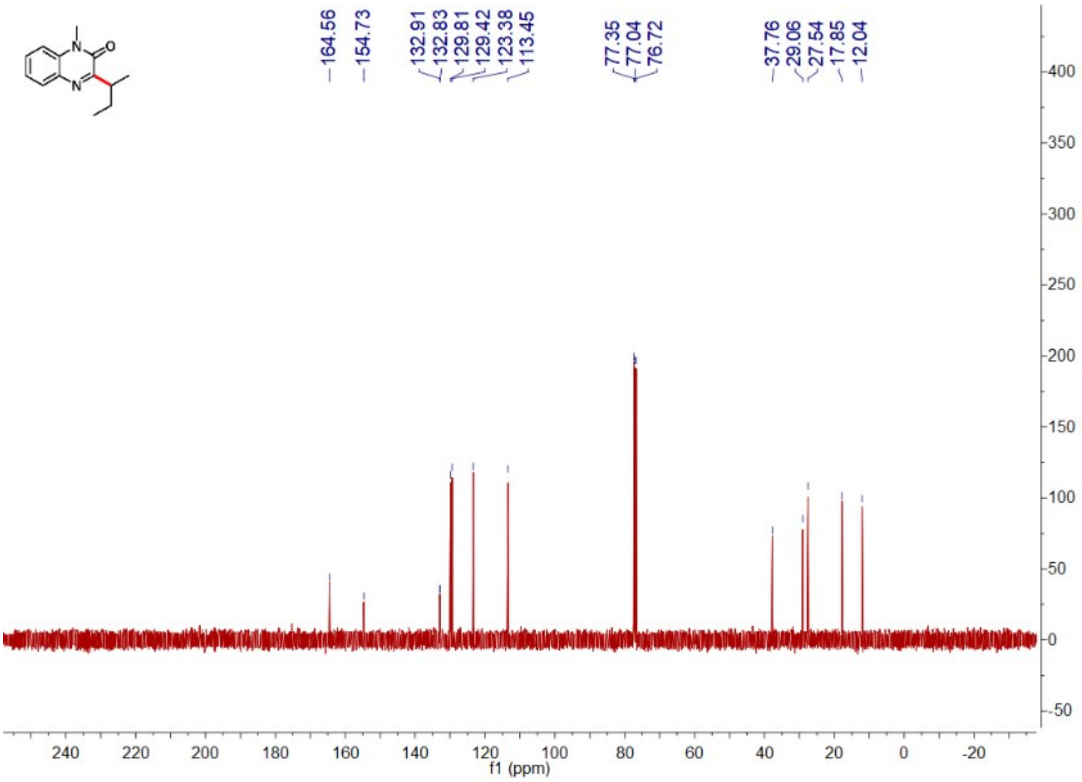
^{13}C NMR spectrum of compound **21**



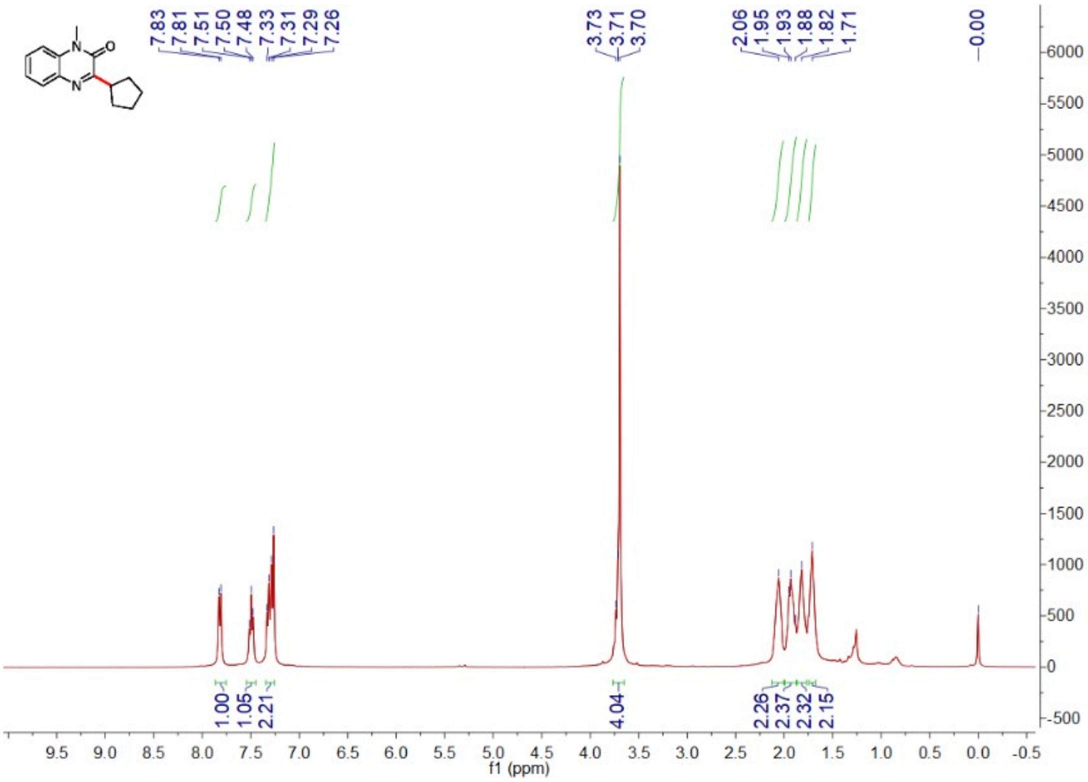
^{1}H NMR spectrum of compound **22**



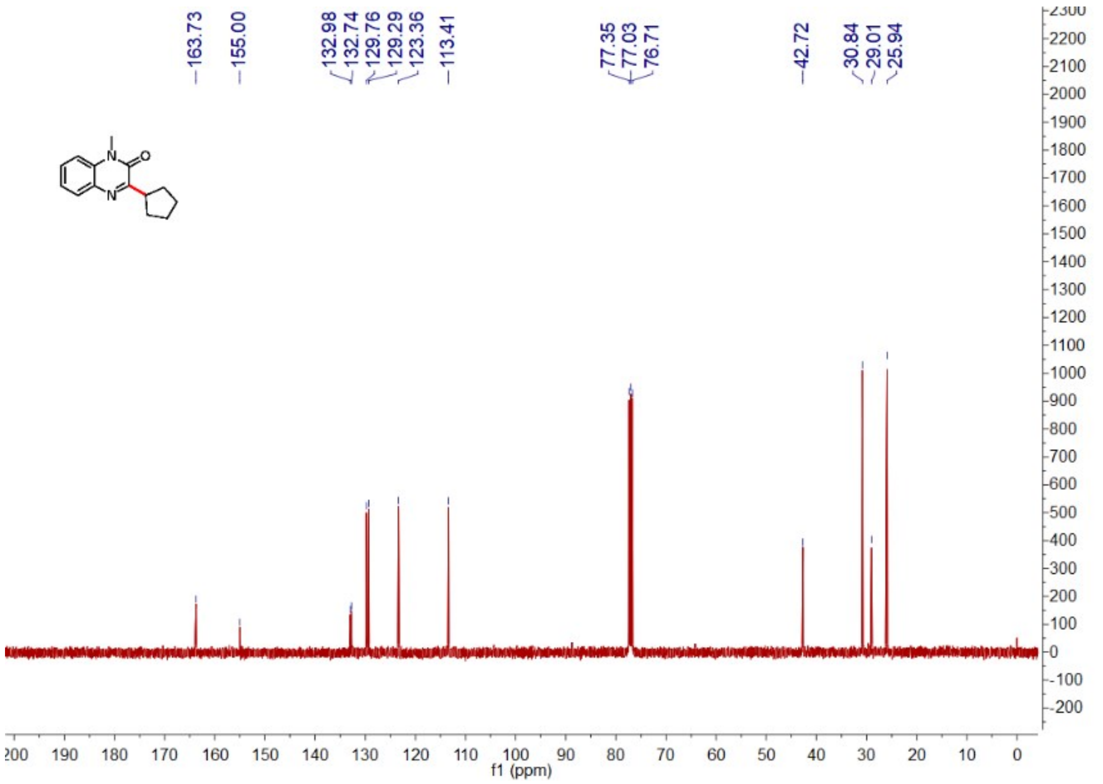
^{13}C NMR spectrum of compound **22**



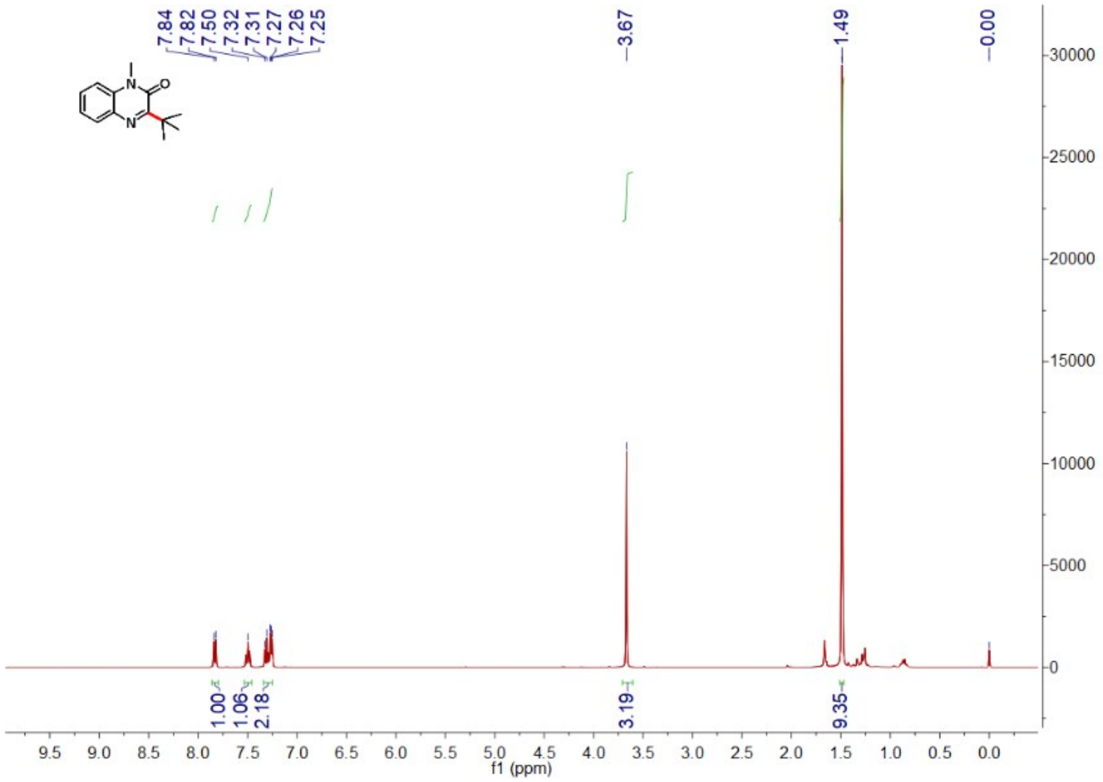
^{1}H NMR spectrum of compound **23**



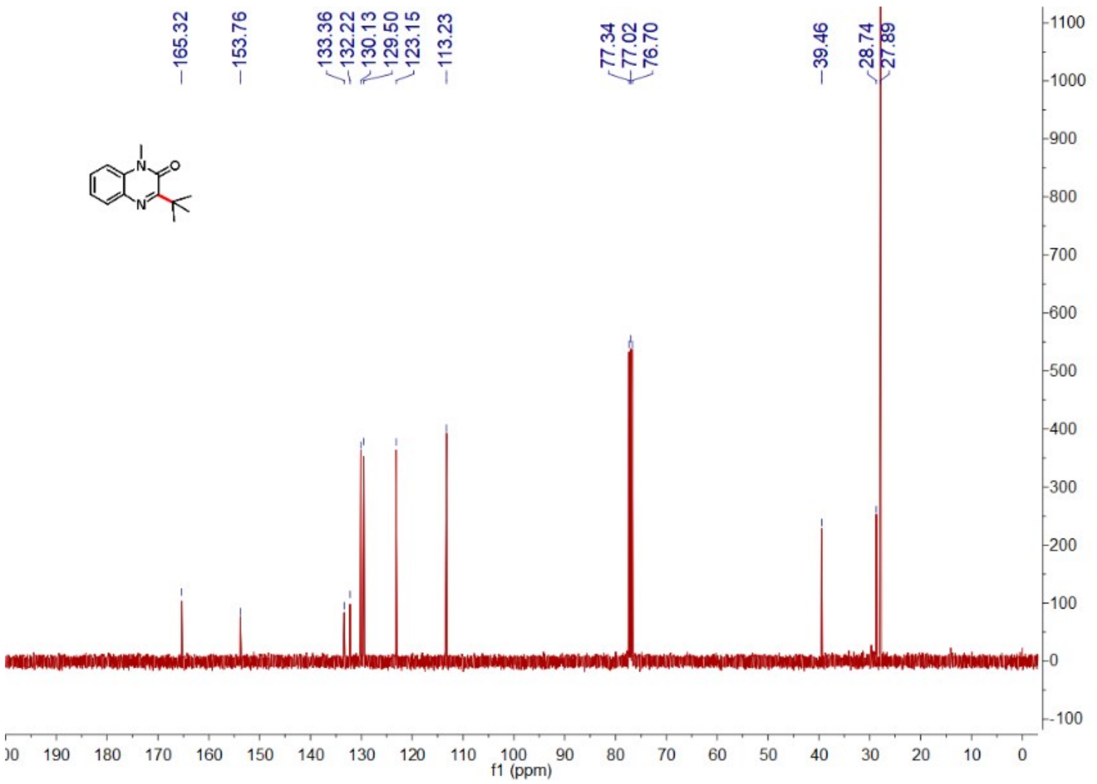
^{13}C NMR spectrum of compound **23**



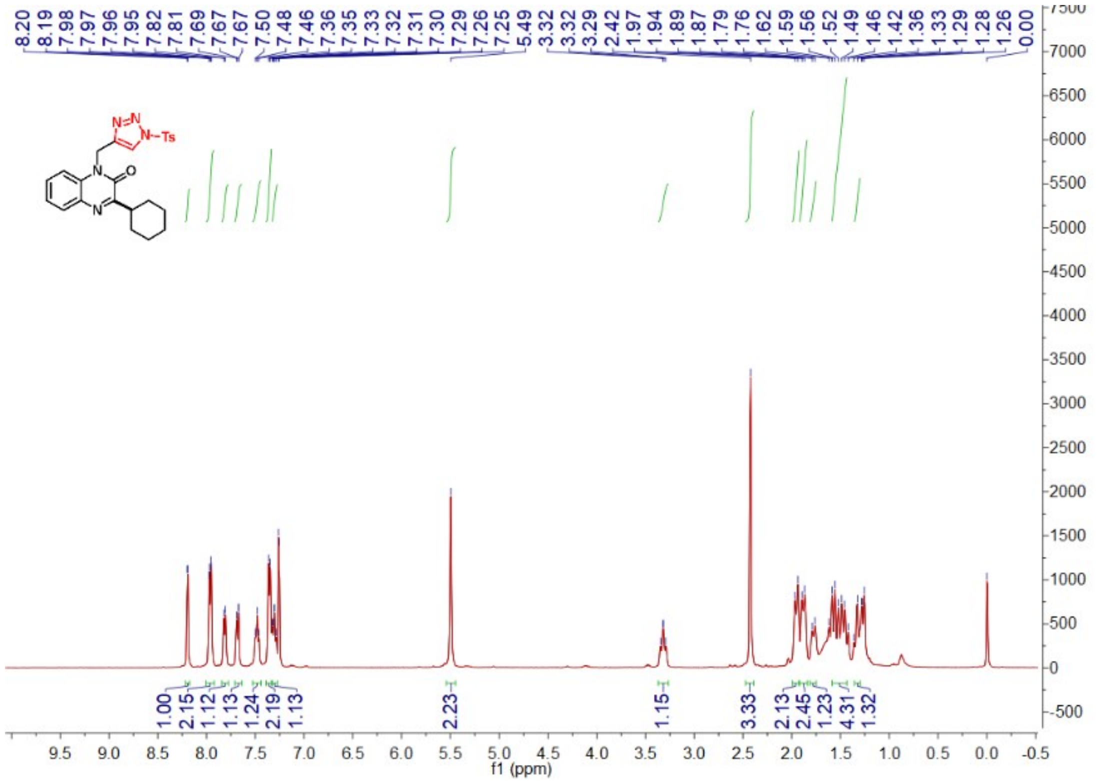
^{1}H NMR spectrum of compound **24**



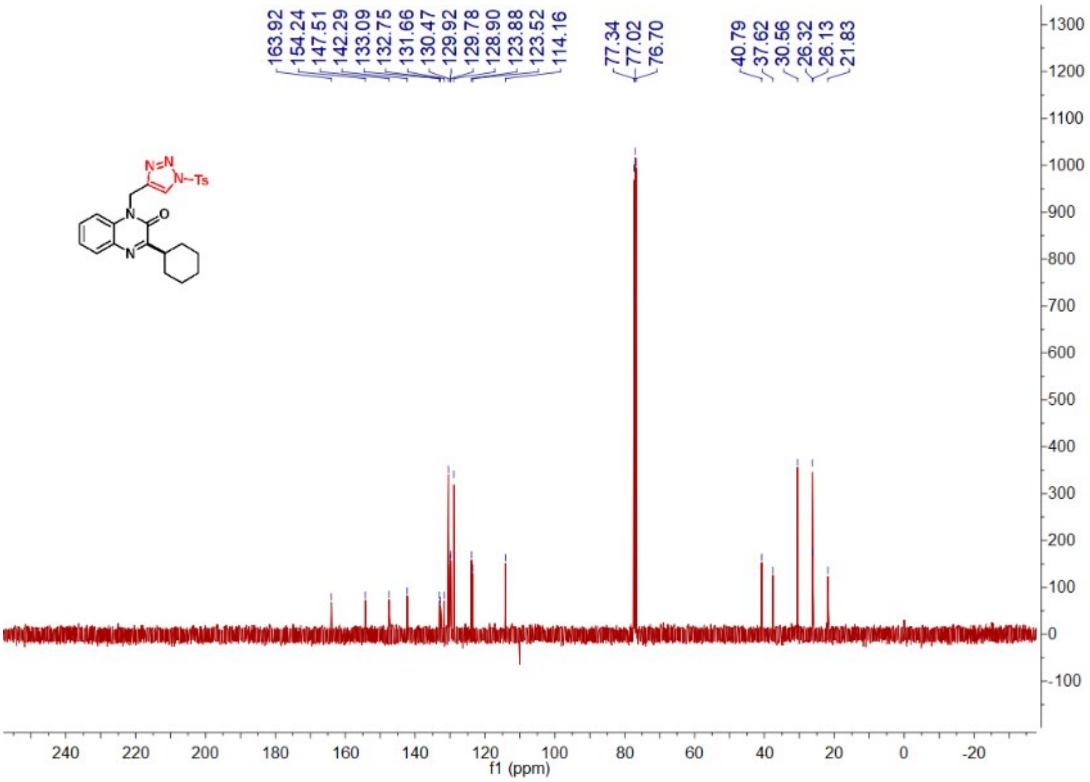
^{13}C NMR spectrum of compound **24**



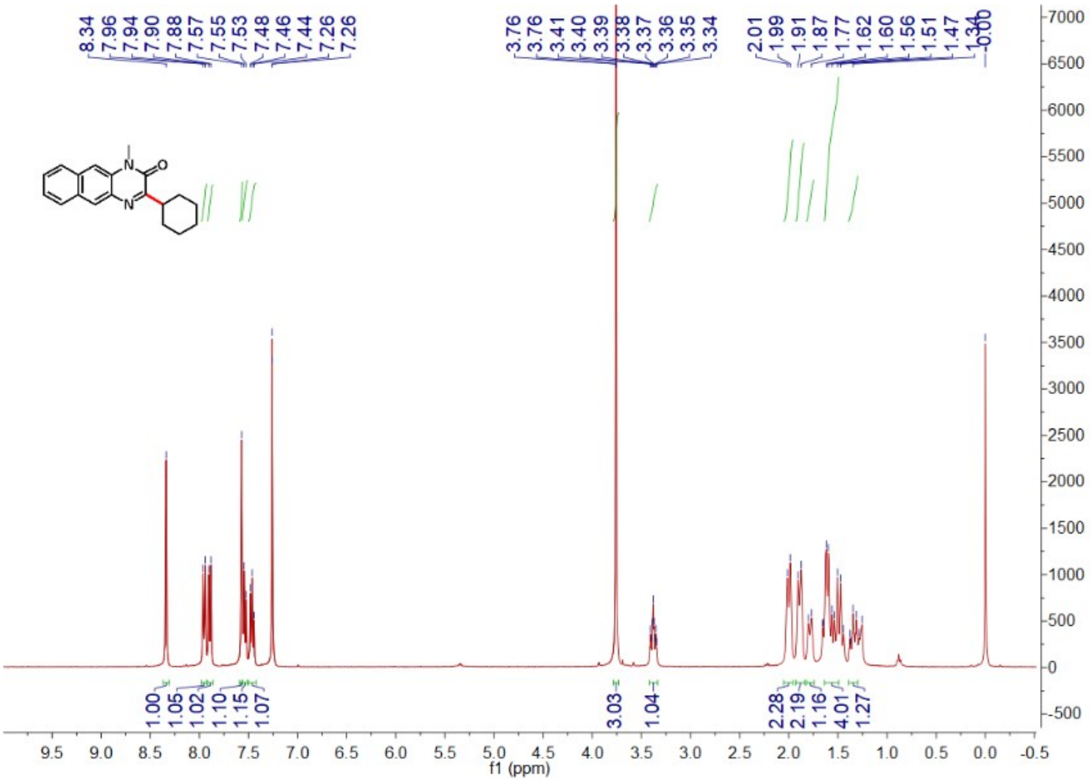
^{1}H NMR spectrum of compound **25**



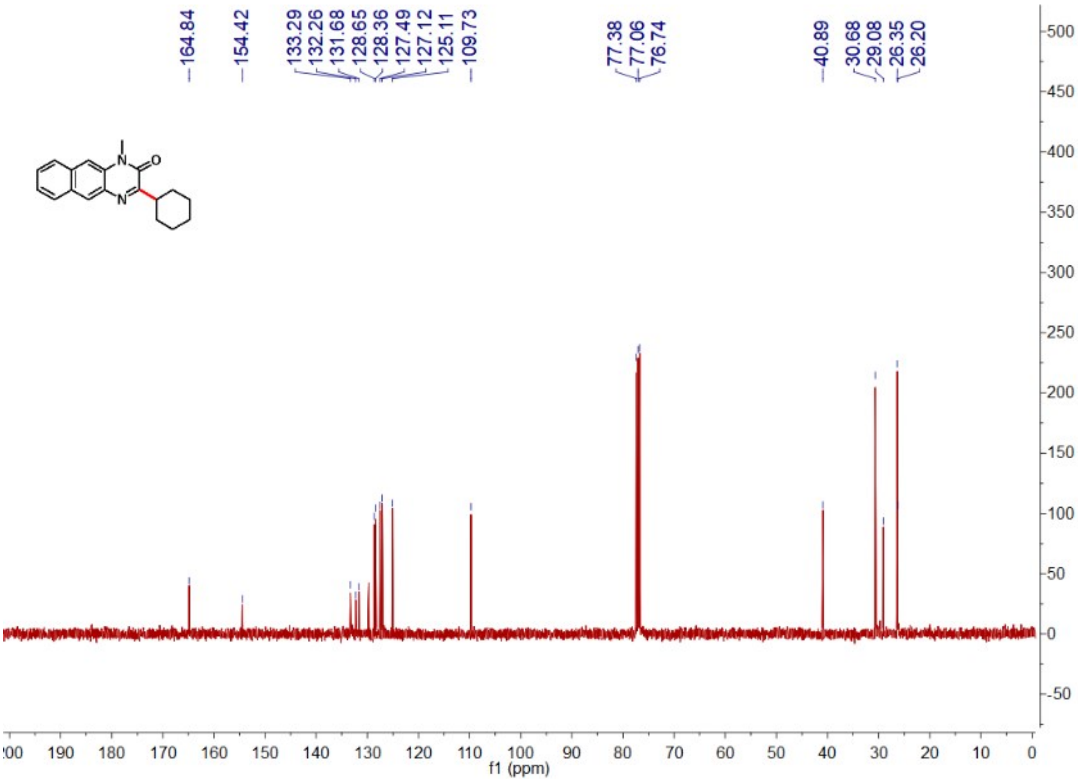
^{13}C NMR spectrum of compound **25**



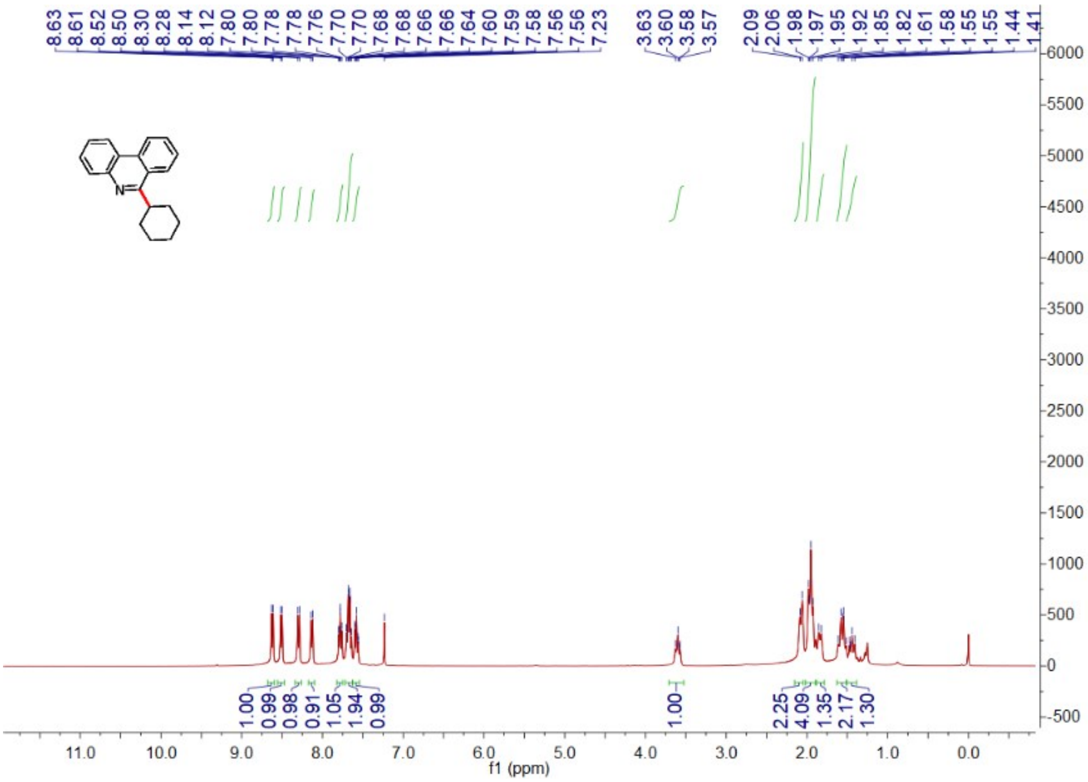
^{1}H NMR spectrum of compound **26**



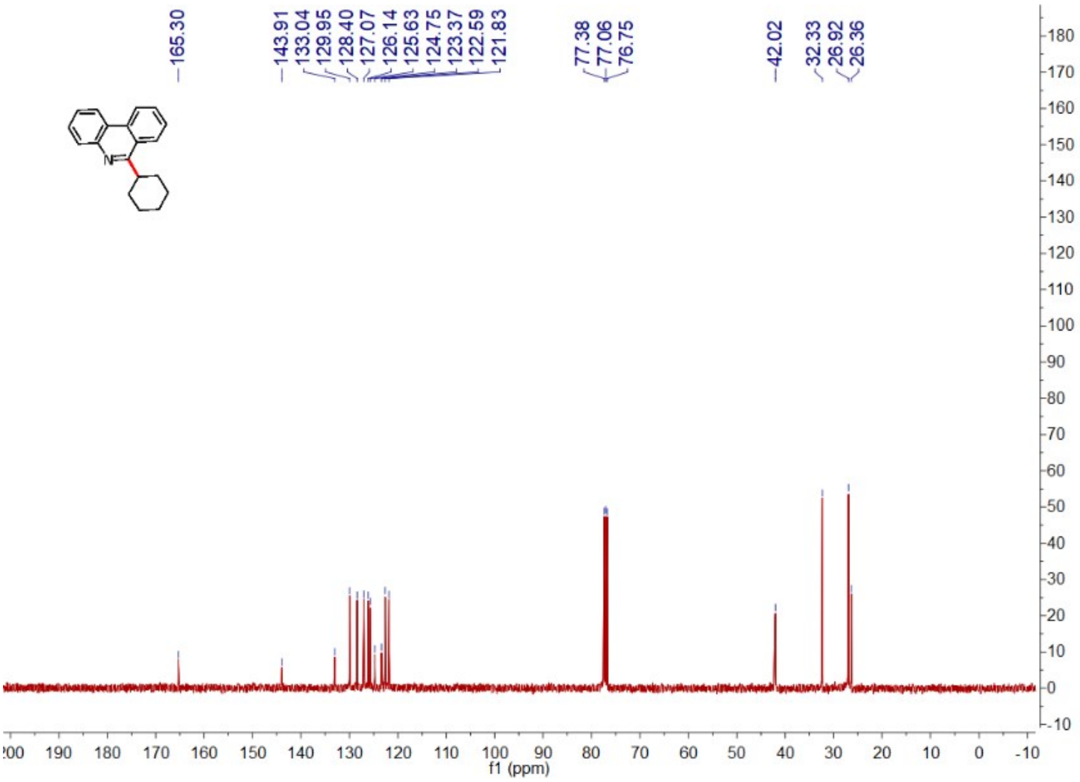
^{13}C NMR spectrum of compound **26**



^{1}H NMR spectrum of compound **27**



^{13}C NMR spectrum of compound **27**



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