

# Supporting Information

## Decatungstate-Photocatalyzed Direct Coupling of Inert Alkanes and Quinoxalin-2(1H)-ones with H<sub>2</sub> Evolution

Xiao-Ya Yuan,<sup>a</sup> Ya-Feng Si,<sup>a</sup> Xiang Li,<sup>a</sup> Shao-Jie Wu,<sup>a</sup> Fan-Lin Zeng,<sup>a</sup> Qi-Yan Lv,<sup>\*a</sup> Bing Yu<sup>\*a</sup>

<sup>a</sup> Green Catalysis Center, College of Chemistry, School of Life Sciences, Zhengzhou University, Zhengzhou 450001, China. E-mail: qiyanlv@zzu.edu.cn; bingyu@zzu.edu.cn.

### Contents

1. General Information.....	2
1.1 Materials and instruments .....	2
1.2 Spectrum of our lamp .....	2
2. Experimental procedures .....	2
2.1 Preparation of TBADT .....	2
2.2 Absorption spectrum of TBADT.....	3
2.3 Preparation of quinoxalin-2 (1H)-ones <sup>2</sup> .....	3
2.4 Optimization of reaction conditions .....	4
2.4 General experimental procedures for the synthesis of 3-alkyl quinoxalin-2(1H)-ones .....	5
2.5 Control experiment.....	6
2.6 Detection of H <sub>2</sub> Generation under Standard condition.....	7
2.7 Catalyst recycling experiments .....	7
3. Characterization of products.....	8
3.1 Data for Products.....	8
3.2 NMR Spectra for Products .....	26

## 1. General Information

### 1.1 Materials and instruments

All the chemicals were purchased from commercial distributors and used without further purification. All reactions were monitored by Thin Layer Chromatography (TLC) using UV light (254/365 nm) for detection. Products were purified by column chromatography using 200-300 mesh silica gel as the stationary phase. All the  $^1\text{H}$ ,  $^{13}\text{C}$  and  $^{19}\text{F}$  NMR spectra were recorded on a Bruker Ascend 400 spectrometer. All NMR spectra were recorded in  $\text{CDCl}_3$  at room temperature ( $20 \pm 2$  °C). Proton chemical shifts  $\delta$  were given in ppm using TMS as the internal standard. High-resolution mass spectra (HRMS) were obtained with 3000-mass spectrometer, using Waters Q-ToF MS/MS system using the ESI technique.  $\text{H}_2$  analysis was performed on Agilent 8860 (G2790A) GC system. Absorption spectra were recorded on a JASCO V-750 UV-Vis spectrometer with 1 cm quartz cell. EPR spectrum was recorded by Bruker EMX10/12 EPR spectrometer. The quinoxalin-2(1H)-ones and tetrabutylammonium decatungstate (TBADT) catalyst were synthesized according to the reported procedure<sup>1</sup>.

### 1.2 Spectrum of our lamp

All reactions have been studied in borosilicate glass vessels irradiated by white light from a photoreactor manufactured by Beijing Roger Technology Co., Ltd. without using filters.

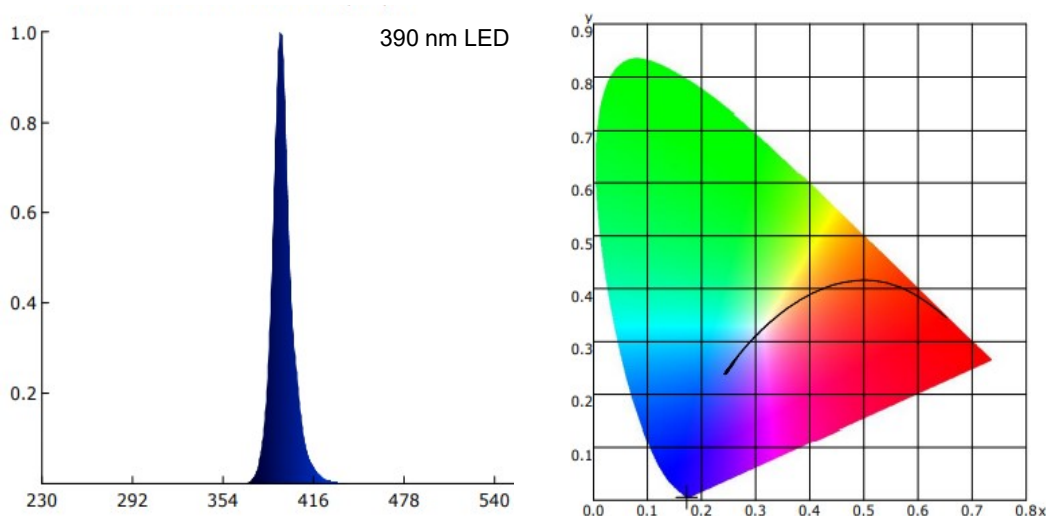


Figure S1. The spectrum of our lamp (390 nm LED)

## 2. Experimental procedures

### 2.1 Preparation of TBADT

Sodium tungstate dihydrate (16 g, about 48.5 mmol) was dissolved in 100 mL of deionized water in 250 mL round-bottomed flask, then add 3.0 M of HCl dropwise (about 30 mL) under vigorous stirring in order to adjust the pH at 2 (the tungstate solution assumed a slightly green color). Subsequently, keep the flask at 85 °C (oil bath) for 20 min. Tetrabutylammonium bromide (6 g) was dissolved in 10 mL of deionized water, and then slowly add to the flask maintained at 85 °C (oil bath) for 30 min under stirring. A white suspension of TBADT was formed, cooled to room temperature, and

filtered. The solid phase was washed with water, ethanol and then dried in vacuum oven at 60 °C for 12 h, affording a slight yellow solid, that can use directly without further purification.

## 2.2 Absorption spectrum of TBADT

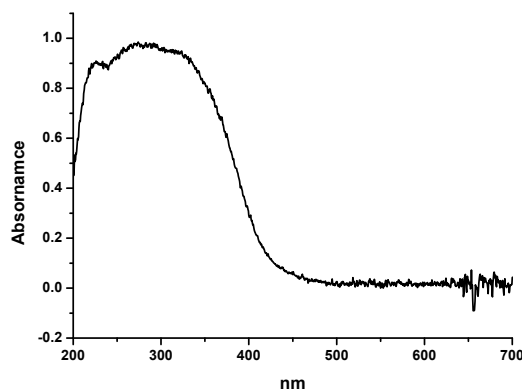
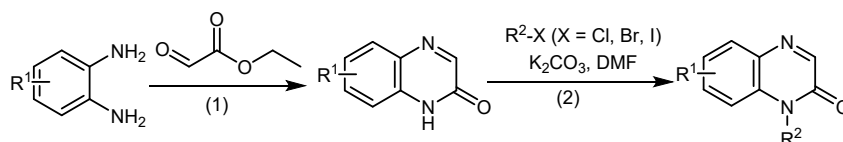


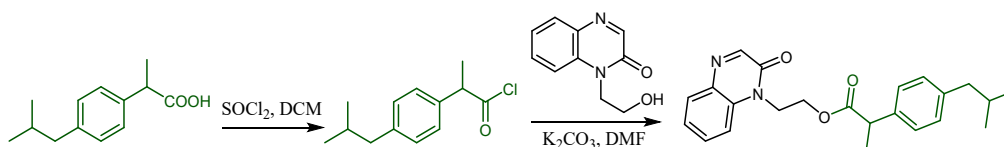
Figure S2. Absorbance spectra of TBADT

## 2.3 Preparation of quinoxalin-2(1H)-ones<sup>2</sup>



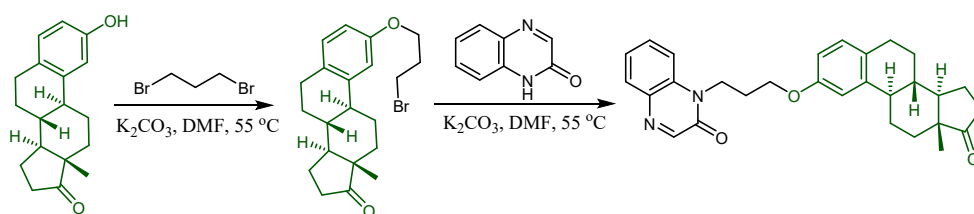
(1) To a 150 ml over dried round-bottom flask charged with a magnetic stirring bar, o-phenylenediamine (20 mmol), ethyl-2-oxoacetate (24 mmol), and ethanol (80 mL) were added respectively, the mixture was stirred at 70 °C for 4 h, cold to room temperature. Then the resulting precipitate was filtered, washed with water, and dried under vacuum to afford quinoxalin-2 (1H) -ones.

(2) Quinoxalin-2(1H)-ones (5 mmol), K<sub>2</sub>CO<sub>3</sub> (7.5 mmol, 1.5 equiv) and DMF (15 ml) were added to a 50 ml oven-dried round-bottom flask, the mixture was stirred and heated to 55 °C for 10 min, after then added corresponding haloalkane (1.6 equiv). The resulting mixture was stirred for 1-6 h. Then the reaction mixture was diluted with ethyl acetate, washed with brine, and the organic layer was separated and dried over anhydrous MgSO<sub>4</sub>. The solvent was concentrated under reduced pressure and the residue was purified by column chromatography on silica gel to afford the desired substrates.



To a 25 mL round-bottomed flask with a stir bar was added ibuprofen (2.0 mmol), dry DCM (10 mL), then was added SOCl<sub>2</sub> (4.0 mmol) dropwise. The reaction mixture was then refluxed until ibuprofen all transformed (monitored by TLC). The solvent was then removed under reduced pressure. And then added the quinoxalin-2(1H)-one (1.0 mmol), K<sub>2</sub>CO<sub>3</sub> (1.5 mmol) and DMF (5.0 ml) to the resulting mixture, which was stirred at room temperature for 3 - 5 h, poured into brine and extracted with EtOAc. The combined extracts were dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and evaporated. The residue was

purified by column chromatography to afford the desired product.



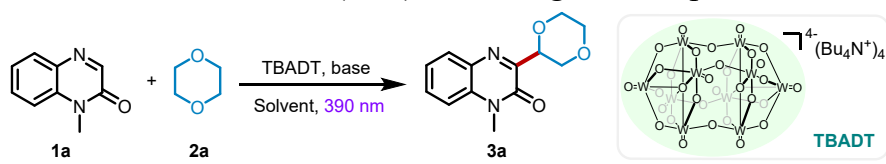
Estrone (5.0 mmol),  $K_2CO_3$  (7.5 mmol, 1.5 equiv), and DMF (15 ml) were added to a round-bottomed flask, the mixture was heated to 55 °C for 10 mins, then 1,3-dibromopropane (1.6 equiv) was added dropwise, stirred at 55 °C for 12 h, the accomplished reaction was cold to room temperature, brine and was added to the mixture and the organic phase was extracted by three portions of ethyl acetate. The combined organic layer was dried over magnesium sulfate and the solvent evaporated in vacuo. Further purification was carried out by silica gel column chromatography.

To a 50 ml round-bottomed flask with a stirring bar was added quinoxalin-2(1*H*)-one,  $K_2CO_3$  (1.5 equiv) and DMF, the mixture was heated to 55 °C for 10 mins, then the above product was added in 1.6 equiv, stirred at 55 °C for 12 h, the accomplished reaction was cold to room temperature, brine and was added to the mixture and the organic phase was extracted by three portions of ethyl acetate. The combined organic layer was dried over magnesium sulfate and the solvent evaporated in vacuo. Further purification was carried out by silica gel column chromatography.

#### 2.4 Optimization of reaction conditions

We commenced the experimental study from the model reaction of *N*-methylquinoxalin-2(1*H*)-one (**1a**) and 1,4-dioxane (**2a**) under light irradiation (Table S1). Initially, when the reaction was performed under  $N_2$  atmosphere in  $CH_3CN$  by employing TBADT as a photocatalyst under the irradiation of UV-light (390 nm) for 8 h, the desired alkylation product **3a** was successfully isolated in 52% yield (entry 1). Encouraged by this result, various solvents, including DMC, DMSO, DMA, PEG<sub>200</sub>, and acetone, were surveyed in the presence of TBADT (entries 2-6). Among them, acetone provided the best performance giving the product **3a** in 37% yield (entry 6), which is still lower than that in  $CH_3CN$ . To further improve the reaction efficiency, we added 1.2 equivalents amount of  $KHCO_3$  to the reaction system, gratifying, a satisfactory yield of **3a** was obtained (entry 7). Subsequently, various inorganic and organic bases were tested, the results showed that  $KHCO_3$  was the optimal base (entries 7-12). Then investigation of the dosages of the base indicated that 1.2 equivalent of  $KHCO_3$  was the best amount for this transformation (entries 13-15). When prolonging the reaction time to 10 h, a slight decrease in the yield was observed (entry 16). Notably, no desired product was observed in the absence of either TBADT or visible light, demonstrating that the photocatalyst and light irradiation were essential in this protocol (entries 17 and 18). When decreasing the amount of TBADT or **2a**, the yield of **3a** decreased significantly (entries 19-20). The optimal reaction conditions were thus established as follows: **1a** (0.2 mmol), **2a** (4 mmol), and  $KHCO_3$  (1.2 equiv) in  $CH_3CN$  (2 mL) at room temperature under

the irradiation of a 390 nm LED (8 W) in a nitrogen atmosphere for 8 h.

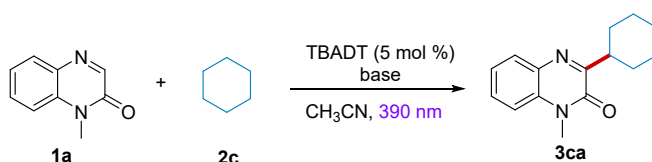


Entry	Solvent	Base (equiv)	Yield <sup>b</sup> (%)
1	CH <sub>3</sub> CN	-	52
2	DMC	-	Trace
3	DMSO	-	n.d.
4	DMA	-	n.d.
5	PEG <sub>200</sub>	-	n.d.
6	Acetone	-	37
7	CH <sub>3</sub> CN	KHCO <sub>3</sub> (1.2)	83
8	CH <sub>3</sub> CN	NaHCO <sub>3</sub> (1.2)	68
9	CH <sub>3</sub> CN	K <sub>3</sub> PO <sub>4</sub> (1.2)	40
10	CH <sub>3</sub> CN	DABCO (1.2)	51
11	CH <sub>3</sub> CN	K <sub>2</sub> HPO <sub>4</sub> (1.2)	43
12	CH <sub>3</sub> CN	DBU (1.2)	n.d.
13	CH <sub>3</sub> CN	KHCO <sub>3</sub> (1.0)	60
14	CH <sub>3</sub> CN	KHCO <sub>3</sub> (1.5)	69
15	CH <sub>3</sub> CN	KHCO <sub>3</sub> (2.0)	58
16 <sup>c</sup>	CH <sub>3</sub> CN	KHCO <sub>3</sub> (1.2)	74
17 <sup>d</sup>	CH <sub>3</sub> CN	KHCO <sub>3</sub> (1.2)	n.d.
18 <sup>e</sup>	CH <sub>3</sub> CN	KHCO <sub>3</sub> (1.2)	n.d.
19 <sup>f</sup>	CH <sub>3</sub> CN	KHCO <sub>3</sub> (1.2)	71
20 <sup>g</sup>	CH <sub>3</sub> CN	KHCO <sub>3</sub> (1.2)	52

<sup>a</sup>Reaction conditions: **1a** (0.2 mmol), **2a** (20 equiv), TBADT (5 mol %), CH<sub>3</sub>CN (2 mL) in a N<sub>2</sub> atmosphere for 8 h. DMC = dimethyl carbonate, DMA = dimethylacetamide. n.d. = not detected. <sup>b</sup>Isolated yields. <sup>c</sup>10 h. <sup>d</sup>In the darkness. <sup>e</sup>Without TBADT. <sup>f</sup>TBADT (2 mol%) as catalyst. <sup>g</sup>**2a** (10 equiv).

**Table S1** Optimization of the reaction conditions<sup>a</sup>

Based on the system of 1,4-dioxane, we began to select the optimal base towards 3-alkyl quinoxalin-2(1*H*)-ones using the model reaction of *N*-methylquinoxalin-2(1*H*)-one (**1a**) and cyclohexane (**2b**) in the presence of TBADT in CH<sub>3</sub>CN under irradiation of 8 W 390 nm LEDs at room temperature for 12 h. The results can be seen in Table S2.

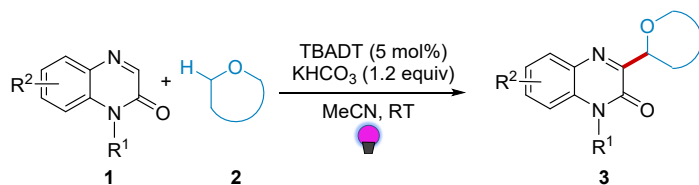


Entry	Base (1.2 equiv)	Yield <sup>b</sup> (%)
1	KHCO <sub>3</sub>	64
2	NaHCO <sub>3</sub>	81
3	K <sub>2</sub> CO <sub>3</sub>	42
4	DABCO	46
5	K <sub>2</sub> HPO <sub>4</sub>	41

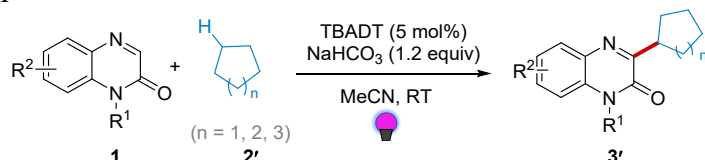
<sup>a</sup> Reaction conditions: **1a** (0.2 mmol), **2c** (10 equiv), TBADT (5 mol%), CH<sub>3</sub>CN (2 mL) in a N<sub>2</sub> atmosphere for 12 h. <sup>b</sup>Isolated yield.

**Table S2** Optimization of the reaction conditions<sup>a</sup>

## 2.4 General experimental procedures for the synthesis of 3-alkyl quinoxalin-2(1*H*)-ones



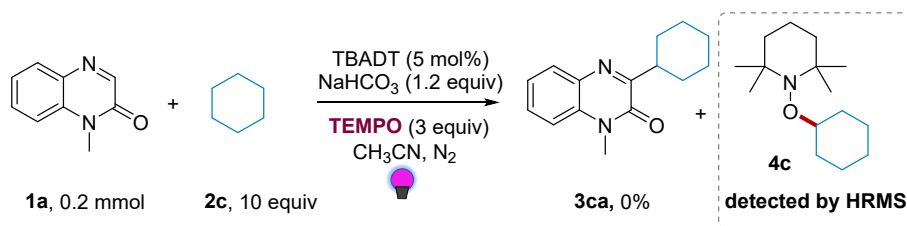
The mixture of quinoxalin-2(1H)-ones **1** (0.2 mmol), **2** (20 equiv),  $\text{KHCO}_3$  (1.2 equiv), TBADT (5 mol%) and  $\text{CH}_3\text{CN}$  (2 mL, 0.1 M) were sequentially added in 15-mL reaction vial. The vessel was then evacuated and filled with  $\text{N}_2$  through Freeze-Pump-Thaw degassing procedure. After being stirred for 8 hours at room temperature under 390 nm LEDs. After reaction, the residue was quenched with water (5 mL), and the ethyl acetate (5 mL) was added three times for extraction. The combined organic layers were dried over anhydrous  $\text{Na}_2\text{SO}_4$ , the solvent was removed under vacuum and the residue was purified by flash chromatography ( $\text{SiO}_2$ , petroleum ether/ethyl acetate = 2:1) to provide product **3**.



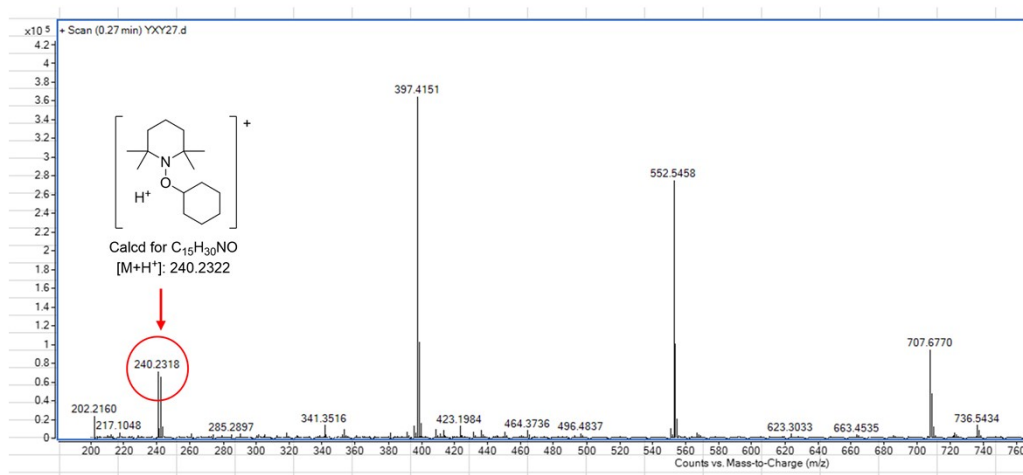
The mixture of quinoxalin-2(1H)-ones **1** (0.2 mmol), **2'** (10 equiv),  $\text{NaHCO}_3$  (1.2 equiv), TBADT (5 mol%) and  $\text{CH}_3\text{CN}$  (2 mL, 0.1 M) were sequentially added in a 10-mL reaction vial. The vessel was then evacuated and filled with  $\text{N}_2$  through Freeze-Pump-Thaw degassing procedure. After being stirred for 12 hours at room temperature under 390 nm LEDs. After reaction, the residue was quenched with water (5 mL), and the ethyl acetate (15 mL) was added three times for extraction. The combined organic layers were dried over anhydrous  $\text{Na}_2\text{SO}_4$ , the solvent was removed under vacuum and the residue was purified by flash chromatography ( $\text{SiO}_2$ , petroleum ether/ethyl acetate = 10:1) to provide product **3'**.

## 2.5 Control experiment

Radical capture experiment by TEMPO



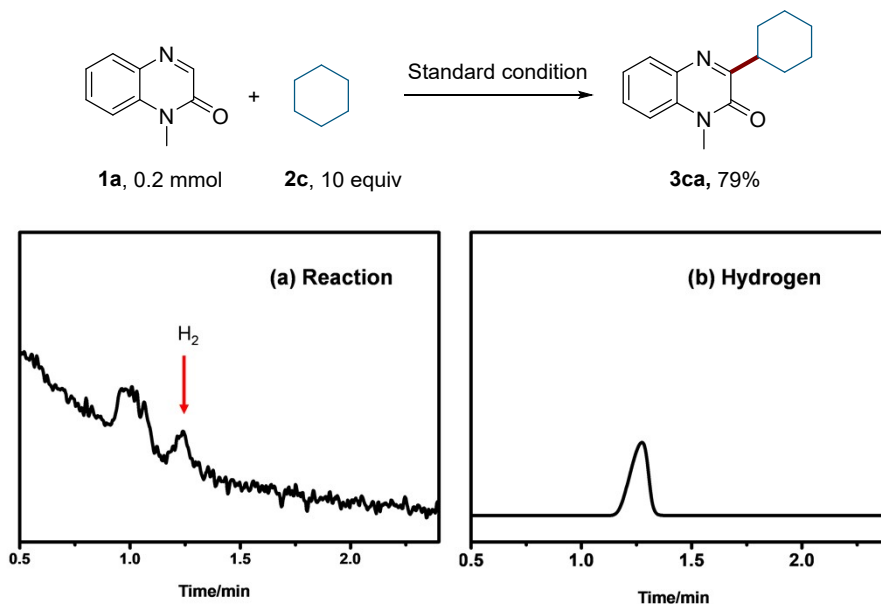
A mixture of **1a** (0.2 mmol, 1 equiv), **2ca** (2 mmol, 10 equiv),  $\text{NaHCO}_3$  (1.2 equiv), TBADT (5 mol%) and TEMPO (3 equiv) were sequentially added in a 10-mL reaction vial. Then, The vessel was then evacuated and filled with  $\text{N}_2$  through Freeze-Pump-Thaw degassing procedure. After being stirred for 12 hours at room temperature under 390 nm LEDs. After the reaction, the reaction mixture was analyzed by HRMS, and product **4c** was successfully detected (**Figure S3**). HRMS calc. for  $\text{C}_{15}\text{H}_{30}\text{NO}^+$  (**4c**)  $m/z = 240.2322$  found 240.2318.



**Figure S3** HRMS spectrum of compound **4c**

## 2.6 Detection of H<sub>2</sub> Generation under Standard condition

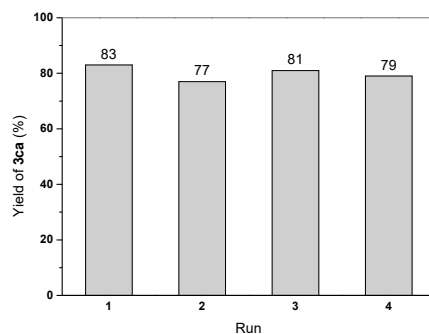
In order to verify the release of H<sub>2</sub> in our reaction system, *N*-methylquinoxalin-2(1H)-one **1a** (0.2 mmol), *N*-phenylglycine **2c** (1 mmol) and TBADT (5 mol %) were dissolved in CH<sub>3</sub>CN (2 mL) under N<sub>2</sub> through the Freeze-Pump-Thaw degassing procedure. After reaction, the gas phase was collected and detected by gas chromatography equipped with a TCD detector. As shown below in Figure S5, the signal of H<sub>2</sub> (retention time = 1.25 min) was obviously observed, providing a strong evidence for the release of H<sub>2</sub>. The product yield could be measured by flash chromatography (SiO<sub>2</sub>, petroleum ether/ethyl acetate = 10:1) to provide product **3ca**.



## 2.7 Catalyst recycling experiments

The mixture of quinoxalin-2(1H)-ones **1a** (0.2 mmol), **2c** (10 equiv), NaHCO<sub>3</sub> (1.2 equiv), TBADT (10 mol%) and CH<sub>3</sub>CN (2 mL) were sequentially added in a 20-mL reaction vial. The vessel was then evacuated and filled with N<sub>2</sub> through Freeze-Pump-Thaw degassing procedure. After being stirred for 12 hours at room temperature under irradiation of 390 nm (Kessil, 10 W). After the reaction, 15 mL diethyl ether was added

to the reaction mixture to ensure that TBADT is completely precipitated from the reaction solution. The resulting suspension was filtered through celite, and then washed with EA (10 mL). The residue was purified by flash chromatography (SiO<sub>2</sub>, petroleum ether/ethyl acetate = 10:1) to provide product **3ca** in 83% yield. Then the residual TBADT in celite was washed by CH<sub>3</sub>CN (10 mL) to give a TBADT solution as filtrate, which was evaporated to afford the recycled TBADT. Then, the recycled TBADT can be directly used for the next run. The results are shown in Figure S4.

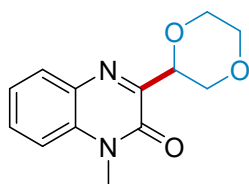


**Figure S4.** Catalyst recycling experiments.

### 3. Characterization of products

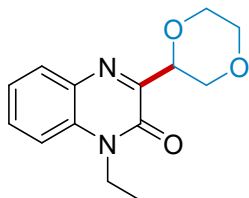
#### 3.1 Data for Products

*3-(1,4-dioxan-2-yl)-1-methylquinoxalin-2(1H)-one (3aa)*<sup>3</sup>



Purification by flash column chromatography (PE:EA, 2:1 v/v) to provide **3aa**. White solid (41 mg, 83% yield); <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 8.01 (d, *J* = 8.9 Hz, 1H), 7.57 (d, *J* = 8.4 Hz, 1H), 7.39 – 7.29 (m, 2H), 5.28 (dd, *J* = 9.5, 2.5 Hz, 1H), 4.26 (dd, *J* = 11.2, 2.4 Hz, 1H), 4.10 (d, *J* = 11.5 Hz, 1H), 4.02 – 3.92 (m, 1H), 3.82 (d, *J* = 5.8 Hz, 2H), 3.69 (s, 3H), 3.63 (dd, *J* = 11.0, 9.7 Hz, 1H). <sup>13</sup>C{<sup>1</sup>H} NMR (101 MHz, Chloroform-*d*) δ 155.1, 153.6, 133.1, 132.6, 130.8, 130.7, 123.9, 113.6, 74.6, 69.4, 67.5, 66.3, 29.0.

*3-(1,4-dioxan-2-yl)-1-ethylquinoxalin-2(1H)-one (3ab)*

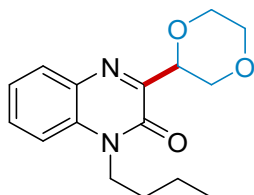


Purification by flash column chromatography (PE:EA, 2:1 v/v) to provide **3ab**. Yellow solid (48 mg, 92% yield); m.p. 153.6 – 154.8 °C; <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 8.02 (d, *J* = 7.8 Hz, 1H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.33 (d, *J* = 7.4 Hz, 2H), 5.29 (d, *J* = 9.1 Hz, 1H), 4.27 (dd, *J* = 21.1, 9.1 Hz, 3H), 4.09 (d, *J* = 11.4 Hz, 1H), 4.00 – 3.92 (m, 1H), 3.81 (d, *J* = 5.6 Hz, 2H), 3.65 (t, *J* = 10.3 Hz, 1H), 1.36 (t, *J* = 6.7 Hz, 3H). <sup>13</sup>C{<sup>1</sup>H} NMR (101 MHz, Chloroform-*d*) δ 155.1, 153.1, 132.9, 132.0, 130.9, 130.8,



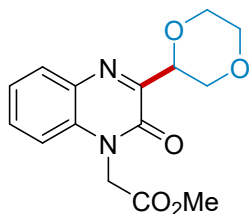
123.6, 113.5, 74.5, 69.4, 67.5, 66.3, 37.3, 12.4. HRMS (ESI)  $m/z$ : calcd for  $C_{14}H_{17}N_2O_3$  [ $M + H$ ] $^+$ , 261.1234; found, 261.1234.

*1-butyl-3-(1,4-dioxan-2-yl)quinoxalin-2(1H)-one (3ac)*



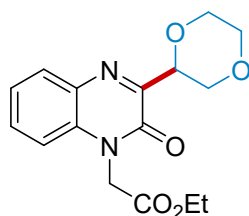
Purification by flash column chromatography (PE:EA, 3:1 v/v) to provide **3ac**. White solid (36 mg, 62% yield); m.p. 67.6 – 69.7 °C;  $^1H$  NMR (400 MHz, Chloroform-*d*)  $\delta$  8.04 (d,  $J = 8.0$  Hz, 1H), 7.58 (t,  $J = 7.4$  Hz, 1H), 7.39 – 7.31 (m, 2H), 5.31 (dd,  $J = 9.5, 2.5$  Hz, 1H), 4.31 – 4.19 (m, 3H), 4.12 (d,  $J = 11.6$  Hz, 1H), 4.05 – 3.95 (m, 1H), 3.84 (dd,  $J = 7.9, 2.1$  Hz, 2H), 3.72 – 3.62 (m, 1H), 1.76 – 1.69 (m, 2H), 1.52 – 1.42 (m, 2H), 1.01 (t,  $J = 7.3$  Hz, 3H).  $^{13}C$  { $^1H$ } NMR (101 MHz, Chloroform-*d*)  $\delta$  155.1, 153.4, 132.8, 132.3, 130.9, 130.7, 123.6, 113.6, 74.6, 69.4, 67.5, 66.3, 42.1, 29.3, 20.3, 13.7. HRMS (ESI)  $m/z$ : calcd for  $C_{16}H_{21}N_2O_3$  [ $M + H$ ] $^+$ , 289.1547; found, 289.1552.

*Methyl-2-(3-(1,4-dioxan-2-yl)-2-oxoquinoxalin-1(2H)-yl)acetate (3ad)*



Purification by flash column chromatography (PE:EA, 2:1 v/v) to provide **3ad**. White solid (41 mg, 68% yield); m.p. 126.7 – 128.1 °C;  $^1H$  NMR (400 MHz, Chloroform-*d*)  $\delta$  8.05 (dd,  $J = 8.0, 1.5$  Hz, 1H), 7.58 – 7.53 (m, 1H), 7.41 – 7.35 (m, 1H), 7.09 (d,  $J = 8.3$  Hz, 1H), 5.29 (dd,  $J = 9.5, 2.7$  Hz, 1H), 5.04 (d,  $J = 2.0$  Hz, 2H), 4.26 (dd,  $J = 11.2, 2.7$  Hz, 1H), 4.10 (s, 1H), 4.02 – 3.94 (m, 1H), 3.84 (dd,  $J = 7.7, 2.0$  Hz, 2H), 3.78 (s, 3H), 3.68 (dd,  $J = 11.2, 9.6$  Hz, 1H).  $^{13}C$  { $^1H$ } NMR (101 MHz, Chloroform-*d*)  $\delta$  167.3, 154.9, 153.2, 132.6, 132.2, 131.1, 131.0, 124.2, 113.1, 74.5, 69.4, 67.5, 66.3, 52.9, 43.3. HRMS (ESI)  $m/z$ : calcd for  $C_{15}H_{17}N_2O_5$  [ $M + H$ ] $^+$ , 305.1132; found, 305.1140.

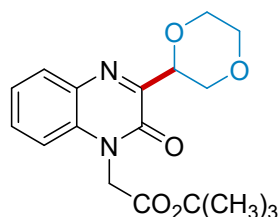
*Ethyl-2-(3-(1,4-dioxan-2-yl)-2-oxoquinoxalin-1(2H)-yl)acetate (3ae)*<sup>3</sup>



Purification by flash column chromatography (PE:EA, 2:1 v/v) to provide **3ae**. White solid (49 mg, 77% yield);  $^1H$  NMR (400 MHz, Chloroform-*d*)  $\delta$  8.01 (dd,  $J = 8.0, 1.2$  Hz, 1H), 7.50 (d,  $J = 8.5$  Hz, 1H), 7.33 (t,  $J = 7.6$  Hz, 1H), 7.06 (d,  $J = 8.3$  Hz, 1H), 5.25 (dd,  $J = 9.5, 2.7$  Hz, 1H), 4.99 (d,  $J = 1.5$  Hz, 2H), 4.26 – 4.19 (m, 3H), 4.08 (d,  $J = 11.5$  Hz, 1H), 3.99 – 3.89 (m, 1H), 3.82 – 3.76 (m, 2H), 3.64 (dd,  $J =$

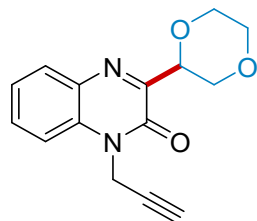
11.1, 9.6 Hz, 1H), 1.2 (t,  $J = 7.2$  Hz, 3H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (101 MHz, Chloroform- $d$ )  $\delta$  166.8, 154.9, 153.2, 132.6, 132.2, 131.0, 130.9, 124.1, 113.1, 74.5, 69.4, 67.5, 66.3, 62.1, 43.4, 14.1.

*tert*-butyl-2-(3-(1,4-dioxan-2-yl)-2-oxoquinoxalin-1(2H)-yl)acetate (**3af**)



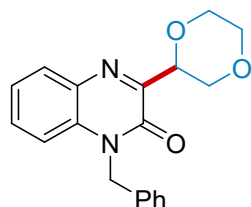
Purification by flash column chromatography (PE:EA, 2:1 v/v) to provide **3af**. Yellow solid (45 mg, 65% yield); m.p. 135.2 – 136.5 °C;  $^1\text{H}$  NMR (400 MHz, Chloroform- $d$ )  $\delta$  7.99 (d,  $J = 8.0$  Hz, 1H), 7.50 (t,  $J = 7.8$  Hz, 1H), 7.34 – 7.28 (m, 1H), 7.04 (d,  $J = 8.4$  Hz, 1H), 5.25 (dd,  $J = 9.5, 2.4$  Hz, 1H), 4.95 – 4.81 (m, 2H), 4.22 (dd,  $J = 11.2, 2.3$  Hz, 1H), 4.07 (d,  $J = 11.5$  Hz, 1H), 3.98 – 3.89 (m, 1H), 3.78 (d,  $J = 5.9$  Hz, 2H), 3.67 – 3.58 (m, 1H), 1.41 (s, 9H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (101 MHz, Chloroform- $d$ )  $\delta$  165.8, 154.9, 153.2, 132.6, 132.3, 130.9, 124.0, 113.1, 83.3, 74.5, 69.4, 67.5, 66.3, 44.1, 27.9. HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{18}\text{H}_{23}\text{N}_2\text{O}_5$   $[\text{M} + \text{H}]^+$ , 347.1601; found, 347.1612.

3-(1,4-dioxan-2-yl)-1-(prop-2-yn-1-yl)quinoxalin-2(1H)-one (**3ag**)



Purification by flash column chromatography (PE:EA, 3:1 v/v) to provide **3ag**. Yellow solid (37 mg, 68% yield); m.p. 128.9 – 131.1 °C;  $^1\text{H}$  NMR (400 MHz, Chloroform- $d$ )  $\delta$  8.03 (dd,  $J = 8.0, 1.5$  Hz, 1H), 7.65 – 7.59 (m, 1H), 7.49 (d,  $J = 8.4$  Hz, 1H), 7.42 – 7.36 (m, 1H), 5.28 (dd,  $J = 9.5, 2.6$  Hz, 1H), 5.12 – 4.98 (m, 2H), 4.26 (dd,  $J = 11.2, 2.5$  Hz, 1H), 4.11 (d,  $J = 11.6$  Hz, 1H), 4.01 – 3.94 (m, 1H), 3.82 (d,  $J = 5.6$  Hz, 2H), 3.67 (dd,  $J = 11.2, 9.6$  Hz, 1H), 2.34 (t,  $J = 2.5$  Hz, 1H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (101 MHz, Chloroform- $d$ )  $\delta$  155.0, 152.6, 132.7, 131.5, 130.9, 130.8, 124.2, 114.2, 76.5, 74.5, 73.5, 69.3, 67.4, 66.3, 31.4. HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{15}\text{H}_{15}\text{N}_2\text{O}_3$   $[\text{M} + \text{H}]^+$ , 271.1077; found, 271.1084.

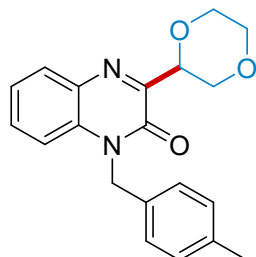
1-benzyl-3-(1,4-dioxan-2-yl)quinoxalin-2(1H)-one (**3ah**)<sup>3</sup>



Purification by flash column chromatography (PE:EA, 2:1 v/v) to provide **3ah**. Yellow solid (57 mg, 88% yield);  $^1\text{H}$  NMR (400 MHz, Chloroform- $d$ )  $\delta$  8.04 (d,  $J = 7.9$  Hz, 1H), 7.45 (t,  $J = 7.7$  Hz, 1H), 7.35 – 7.20 (m, 7H), 5.59 – 5.41 (m, 2H), 5.37 (dd,  $J = 9.5, 2.4$  Hz, 1H), 4.31 (dd,  $J = 11.2, 2.3$  Hz, 1H), 4.14 (d,  $J = 11.5$  Hz, 1H), 4.04 – 3.98 (m, 1H), 3.90 – 3.83 (m, 2H), 3.75 – 3.68 (dd,  $J = 9.8, 1.0$  Hz, 1H).

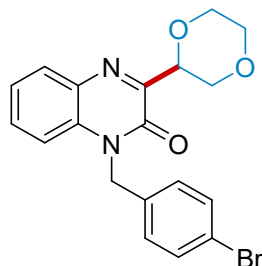
$^{13}\text{C}\{^1\text{H}\}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  155.3, 153.8, 134.9, 132.9, 132.5, 130.84, 130.82, 129.0, 127.8, 126.8, 123.9, 114.5, 74.6, 69.5, 67.6, 66.4, 45.9.

*3-(1,4-dioxan-2-yl)-1-(4-methylbenzyl)quinoxalin-2(1H)-one (3ai)*



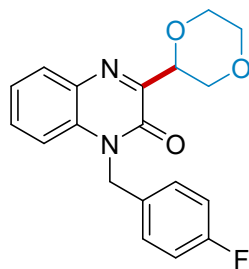
Purification by flash column chromatography (PE:EA, 3:1 v/v) to provide **3ai**. Yellow solid (42 mg, 62% yield); m.p. 149.4 – 151.1 °C;  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  8.04 (d,  $J$  = 7.7 Hz, 1H), 7.46 (t,  $J$  = 2.0 Hz, 1H), 7.31 (dd,  $J$  = 11.4, 8.1 Hz, 2H), 7.12 (s, 4H), 5.55 – 5.35 (m, 3H), 4.32 (dd,  $J$  = 11.1, 2.3 Hz, 1H), 4.14 (d,  $J$  = 11.5 Hz, 1H), 4.05 – 3.96 (m, 1H), 3.90 – 3.84 (m, 2H), 2.71 (t,  $J$  = 2.5 Hz, 1H), 2.30 (s, 3H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  155.2, 153.8, 137.5, 132.8, 132.5, 131.9, 130.81, 130.76, 129.6, 126.9, 123.9, 114.5, 74.6, 69.5, 67.6, 66.3, 45.7, 21.1. HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{20}\text{H}_{21}\text{N}_2\text{O}_3$  [ $\text{M} + \text{H}$ ] $^+$ , 337.1547; found, 337.1555.

*1-(4-bromobenzyl)-3-(1,4-dioxan-2-yl)quinoxalin-2(1H)-one (3aj)*



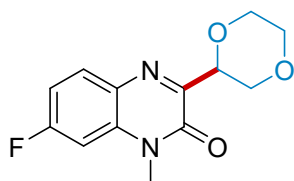
Purification by flash column chromatography (PE:EA, 3:1 v/v) to provide **3aj**. Yellow solid (58 mg, 72% yield); m.p. 160.6 – 163.2 °C;  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  8.05 (dd,  $J$  = 8.0, 1.3 Hz, 1H), 7.45 (dd,  $J$  = 10.7, 7.9 Hz, 3H), 7.37 – 7.31 (m, 1H), 7.21 (d,  $J$  = 8.3 Hz, 1H), 7.11 (d,  $J$  = 8.4 Hz, 2H), 5.50 (d,  $J$  = 15.7 Hz, 1H), 5.41 – 5.34 (m, 2H), 4.29 (dd,  $J$  = 11.2, 2.5 Hz, 1H), 4.13 (d,  $J$  = 11.5 Hz, 1H), 4.05 – 3.97 (m, 1H), 3.85 (dd,  $J$  = 7.9, 2.1 Hz, 2H), 3.71 (dd,  $J$  = 11.1, 9.6 Hz, 1H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  155.2, 153.7, 134.0, 132.8, 132.2, 132.1, 131.0, 128.6, 124.1, 121.8, 114.2, 74.6, 69.4, 67.6, 66.3, 45.3. HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{19}\text{H}_{18}^{79}\text{BrN}_2\text{O}_3$  [ $\text{M} + \text{H}$ ] $^+$ , 401.0495; found, 401.0500.

*3-(1,4-dioxan-2-yl)-1-(4-fluorobenzyl)quinoxalin-2(1H)-one (3ak)*



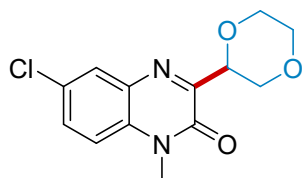
Purification by flash column chromatography (PE:EA, 3:1 v/v) to provide **3ak**. Yellow solid (50 mg, 73% yield); m.p. 191.3 – 192.7 °C; <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 8.04 (dd, *J* = 8.0, 1.3 Hz, 1H), 7.50 – 7.44 (m, 1H), 7.36 – 7.30 (m, 1H), 7.27 – 7.19 (m, 3H), 6.99 (t, *J* = 8.6 Hz, 2H), 5.51 (d, *J* = 15.5 Hz, 1H), 5.42 – 5.32 (m, 2H), 4.29 (dd, *J* = 11.2, 2.5 Hz, 1H), 4.13 (d, *J* = 11.5 Hz, 1H), 4.03 – 3.97 (m, 1H), 3.84 (dd, *J* = 8.1, 2.2 Hz, 2H), 3.71 (dd, *J* = 11.2, 9.6 Hz, 1H). <sup>13</sup>C{<sup>1</sup>H} NMR (101 MHz, Chloroform-*d*) δ 162.2 (d, *J* = 246.9 Hz), 155.2, 153.7, 132.8, 132.3, 130.9 (d, *J* = 1.8 Hz), 130.7 (d, *J* = 3.2 Hz), 128.8, 128.7, 124.0, 115.9 (d, *J* = 21.7 Hz), 114.2, 74.6, 69.4, 67.5, 66.3, 45.2. <sup>19</sup>F NMR (376 MHz, Chloroform-*d*) δ -114.08. HRMS (ESI) *m/z*: calcd for C<sub>19</sub>H<sub>18</sub>FN<sub>2</sub>O<sub>3</sub> [M + H]<sup>+</sup>, 341.1296; found, 341.1295.

*3-(1,4-dioxan-2-yl)-7-fluoro-1-methylquinoxalin-2(1H)-one (3al)*<sup>3</sup>



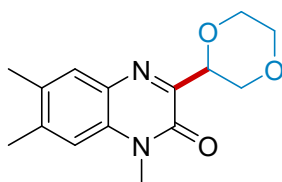
Purification by flash column chromatography (PE:EA, 2:1 v/v) to provide **3al**. White solid (24 mg, 46% yield); <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.72 (dd, *J* = 8.8, 2.7 Hz, 1H), 7.34 – 7.26 (m, 2H), 5.27 (dd, *J* = 9.5, 2.7 Hz, 1H), 4.25 (dd, *J* = 11.1, 2.6 Hz, 1H), 4.10 (d, *J* = 11.6 Hz, 1H), 4.01 – 3.92 (m, 1H), 3.81 (dd, *J* = 6.0, 1.8 Hz, 2H), 3.69 (s, 3H), 3.60 (dd, *J* = 11.2, 9.5 Hz, 1H). <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, Chloroform-*d*) δ 158.7 (d, *J* = 244.3 Hz), 156.7, 153.3, 133.1 (d, *J* = 11.4 Hz), 129.8 (d, *J* = 2.1 Hz), 118.6 (d, *J* = 24.1 Hz), 116.0 (d, *J* = 22.9 Hz), 114.8 (d, *J* = 8.7 Hz), 74.7, 69.4, 67.5, 66.3, 29.3. <sup>19</sup>F NMR (376 MHz, Chloroform-*d*) δ -118.23.

*6-chloro-3-(1,4-dioxan-2-yl)-1-methylquinoxalin-2(1H)-one (3am)*



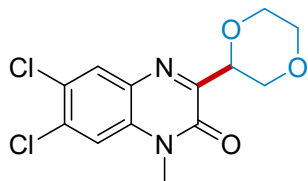
Purification by flash column chromatography (PE:EA, 2:1 v/v) to provide **3am**. Yellow solid (23 mg, 41% yield); m.p. 171.9 – 173.8 °C; <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 8.03 (d, *J* = 2.3 Hz, 1H), 7.53 (dd, *J* = 8.9, 2.4 Hz, 1H), 7.26 (d, *J* = 8.9 Hz, 1H), 5.28 (dd, *J* = 9.4, 2.6 Hz, 1H), 4.24 (dd, *J* = 11.2, 2.5 Hz, 1H), 4.10 (s, 1H), 4.01 – 3.95 (m, 1H), 3.82 (dd, *J* = 8.3, 2.2 Hz, 2H), 3.69 (s, 3H), 3.66 – 3.59 (m, 1H). <sup>13</sup>C{<sup>1</sup>H} NMR (101 MHz, Chloroform-*d*) δ 156.6, 153.3, 133.0, 131.8, 130.8, 130.0, 129.2, 114.8, 74.6, 69.3, 67.4, 66.3, 29.2. HRMS (ESI) *m/z*: calcd for C<sub>13</sub>H<sub>14</sub>ClN<sub>2</sub>O<sub>3</sub> [M + H]<sup>+</sup>, 281.0687; found, 281.0683.

*3-(1,4-dioxan-2-yl)-1,6,7-trimethylquinoxalin-2(1H)-one (3an)*<sup>3</sup>



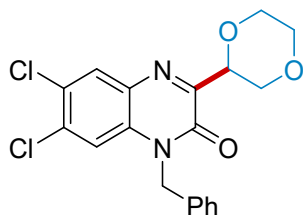
Purification by flash column chromatography (PE:EA, 2:1 v/v) to provide **3an**. Yellow solid (14 mg, 26% yield);  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.80 (s, 1H), 7.09 (s, 1H), 5.29 (dd,  $J = 9.6, 2.7$  Hz, 1H), 4.27 (dd,  $J = 11.2, 2.6$  Hz, 1H), 4.11 (d,  $J = 11.6$  Hz, 1H), 4.03 – 3.96 (m, 1H), 3.84 (dd,  $J = 8.2, 2.3$  Hz, 2H), 3.69 (s, 3H), 2.43 (s, 3H), 2.35 (s, 3H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  153.8, 153.7, 140.8, 132.9, 131.11, 131.05, 130.7, 114.1, 74.6, 69.5, 67.5, 66.3, 29.0, 20.6, 19.2.

*6,7-dichloro-3-(1,4-dioxan-2-yl)-1-methylquinoxalin-2(1H)-one (3ao)*



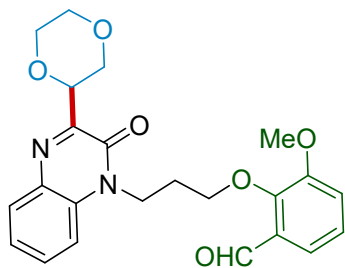
Purification by flash column chromatography (PE:EA, 2:1 v/v) to provide **3ao**. Yellow solid (29 mg, 46% yield); m.p. 207.1 – 208.6 °C;  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  8.09 (s, 1H), 7.40 (s, 1H), 5.24 (dd,  $J = 9.4, 2.7$  Hz, 1H), 4.23 (dd,  $J = 11.2, 2.7$  Hz, 1H), 4.09 (d,  $J = 11.5$  Hz, 1H), 4.00 – 3.91 (m, 1H), 3.85 – 3.77 (m, 2H), 3.65 (s, 3H), 3.60 (dd,  $J = 11.1, 9.5$  Hz, 1H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  156.7, 153.0, 135.0, 132.4, 131.5, 131.4, 127.8, 115.2, 74.5, 69.3, 67.4, 66.3, 29.3. HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{13}\text{H}_{13}\text{Cl}_2\text{N}_2\text{O}_3$   $[\text{M} + \text{H}]^+$ , 315.0298; found, 315.0295.

*1-benzyl-6,7-dichloro-3-(1,4-dioxan-2-yl)quinoxalin-2(1H)-one (3ap)*



Purification by flash column chromatography (PE:EA, 3:1 v/v) to provide **3ap**. White solid (30 mg, 39% yield); m.p. 211.2 – 213.2 °C;  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  8.13 (s, 1H), 7.37 (s, 1H), 7.36 – 7.29 (m, 3H), 7.21 (d,  $J = 6.8$  Hz, 2H), 5.50 (d,  $J = 15.7$  Hz, 1H), 5.39 – 5.31 (m, 2H), 4.29 (dd,  $J = 11.2, 2.6$  Hz, 1H), 4.12 (d,  $J = 11.7$  Hz, 1H), 4.03 – 3.97 (m, 1H), 3.85 (dd,  $J = 6.0, 2.5$  Hz, 2H), 3.69 (dd,  $J = 11.2, 9.5$  Hz, 1H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  156.8, 153.2, 135.1, 134.1, 131.82, 131.80, 131.5, 129.2, 128.2, 127.9, 126.8, 115.9, 74.5, 69.3, 67.5, 66.3, 46.2. HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{19}\text{H}_{17}\text{Cl}_2\text{N}_2\text{O}_3$   $[\text{M} + \text{H}]^+$ , 391.0611; found, 391.0602.

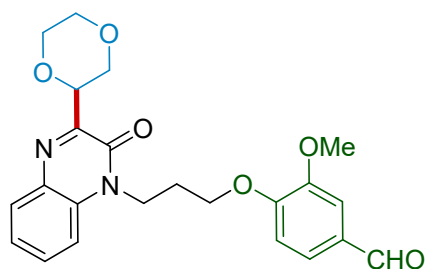
*2-(3-(3-(1,4-dioxan-2-yl)-2-oxoquinoxalin-1(2H)-yl)propoxy)-3-methoxybenzaldehyde (3aq)*



Purification by flash column chromatography (PE:EA, 2:1 v/v) to provide **3aq**. White solid (53 mg, 62% yield); m.p. 71.7 – 73.2 °C;  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  10.49 (s, 1H), 8.15 (d,  $J = 8.0$  Hz, 1H),

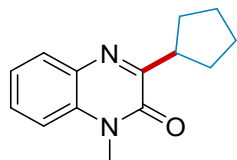
7.83 (d,  $J = 7.8$  Hz, 1H), 7.66 (t,  $J = 7.6$  Hz, 1H), 7.56 (d,  $J = 7.1$  Hz, 1H), 7.46 – 7.38 (m, 1H), 7.13 (d,  $J = 4.1$  Hz, 2H), 5.22 (dd,  $J = 9.7, 2.4$  Hz, 1H), 4.82 – 4.76 (m, 2H), 4.36 (t,  $J = 6.1$  Hz, 2H), 4.19 – 4.08 (m, 2H), 4.03 – 3.93 (m, 1H), 3.90 (dd,  $J = 11.5, 2.4$  Hz, 2H), 3.83 (s, 3H), 3.76 – 3.71 (m, 1H), 2.39 (p,  $J = 6.2$  Hz, 2H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (101 MHz, Chloroform- $d$ )  $\delta$  190.0, 154.6, 152.9, 151.5, 145.0, 140.1, 138.5, 130.1, 129.9, 129.2, 126.9, 126.8, 124.2, 119.3, 118.0, 74.2, 71.4, 69.5, 67.6, 66.3, 63.5, 56.0, 29.6. HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{23}\text{H}_{25}\text{N}_2\text{O}_6$   $[\text{M} + \text{H}]^+$ , 425.1707; found, 425.1701.

4-(3-(3-(1,4-dioxan-2-yl)-2-oxoquinoxalin-1(2H)-yl)propoxy)-3-methoxybenzaldehyde (**3ar**)



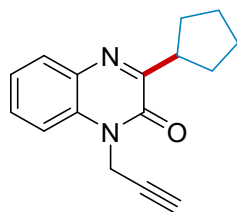
Purification by flash column chromatography (PE:EA, 2:1 v/v) to provide **3ar**. White solid (64 mg, 75% yield); m.p. 145.4 – 147.7 °C;  $^1\text{H}$  NMR (400 MHz, Chloroform- $d$ )  $\delta$  9.85 (s, 1H), 8.14 (d,  $J = 7.9$  Hz, 1H), 7.79 (d,  $J = 7.7$  Hz, 1H), 7.69 – 7.62 (m, 1H), 7.60 – 7.54 (m, 1H), 7.48 – 7.39 (m, 2H), 7.02 (d,  $J = 7.8$  Hz, 1H), 5.20 (d,  $J = 8.8$  Hz, 1H), 4.77 (t,  $J = 6.3$  Hz, 2H), 4.35 (t,  $J = 6.4$  Hz, 2H), 4.12 (t,  $J = 10.2$  Hz, 2H), 3.91 (s, 3H), 3.76 (t,  $J = 10.6$  Hz, 1H), 2.53 – 2.42 (m, 2H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (101 MHz, Chloroform- $d$ )  $\delta$  190.9, 154.5, 153.7, 149.8, 144.9, 140.1, 138.5, 130.2, 130.1, 129.2, 126.9, 126.8, 126.7, 111.5, 109.2, 74.2, 69.5, 67.6, 66.3, 65.8, 63.4, 55.9, 28.5. HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{23}\text{H}_{25}\text{N}_2\text{O}_6$   $[\text{M} + \text{H}]^+$ , 425.1707; found, 425.1723.

3-cyclopentyl-1-methylquinoxalin-2(1H)-one (**3ba**)<sup>4</sup>



Purification by flash column chromatography (PE:EA, 10:1 v/v) to provide **3ba**. White solid (36 mg, 79% yield);  $^1\text{H}$  NMR (400 MHz, Chloroform- $d$ )  $\delta$  7.84 (dd,  $J = 8.0, 1.4$  Hz, 1H), 7.56 – 7.49 (m, 1H), 7.38 – 7.28 (m, 2H), 3.73 (s, 3H), 3.76 – 3.68 (m, 1H), 2.11 – 2.05 (m, 2H), 2.00 – 1.70 (m, 6H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (101 MHz, Chloroform- $d$ )  $\delta$  163.7, 155.0, 133.0, 132.7, 129.6, 129.3, 123.4, 113.4, 42.7, 30.8, 29.0, 25.9.

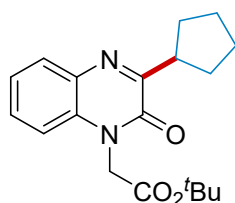
3-cyclopentyl-1-(prop-2-yn-1-yl)quinoxalin-2(1H)-one (**3bb**)



Purification by flash column chromatography (PE:EA, 10:1 v/v) to provide **3bb**. White solid (30 mg, 60% yield). m.p. 138.2 – 139.6 °C;  $^1\text{H}$  NMR (400 MHz, Chloroform- $d$ )  $\delta$  7.85 (dd,  $J = 8.0, 1.5$  Hz, 1H),

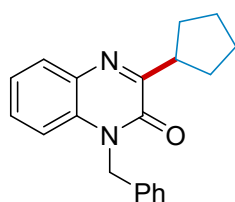
7.57 – 7.53 (m, 1H), 7.45 (dd,  $J = 8.3, 1.0$  Hz, 1H), 7.39 – 7.33 (m, 1H), 5.07 (d,  $J = 2.5$  Hz, 2H), 3.78 – 3.66 (m, 1H), 2.31 (t,  $J = 2.5$  Hz, 1H), 2.12 – 2.05 (m, 2H), 1.99 – 1.70 (m, 6H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform- $d$ )  $\delta$  163.7, 153.9, 132.9, 131.4, 129.9, 129.4, 123.8, 113.9, 73.1, 42.7, 31.4, 30.9, 25.9. HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{16}\text{H}_{17}\text{N}_2\text{O}$   $[\text{M} + \text{H}]^+$ , 253.1335; found, 253.1345.

*Tert-butyl 2-(3-cyclopentyl-2-oxoquinoxalin-1(2H)-yl)acetate (3bc)*



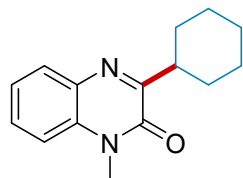
Purification by flash column chromatography (PE:EA, 10:1 v/v) to provide **3bc**. White solid (27 mg, 41% yield). m.p. 66.4 – 67.9 °C;  $^1\text{H}$  NMR (400 MHz, Chloroform- $d$ )  $\delta$  7.84 (dd,  $J = 8.0, 1.3$  Hz, 1H), 7.54 – 7.43 (m, 1H), 7.31 (dd,  $J = 15.1, 7.7$  Hz, 1H), 7.05 (d,  $J = 8.3$  Hz, 1H), 4.95 (s, 2H), 3.78 – 3.65 (m, 1H), 2.14 – 2.04 (m, 2H), 2.00 – 1.90 (m, 2H), 1.85 – 1.69 (m, 4H), 1.47 (s, 6H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform- $d$ )  $\delta$  166.3, 163.6, 154.5, 132.7, 132.2, 130.0, 129.4, 123.6, 112.9, 83.0, 44.2, 42.7, 30.8, 28.0, 25.9. HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{19}\text{H}_{25}\text{N}_2\text{O}_3$   $[\text{M} + \text{H}]^+$ , 329.1860; found, 329.1866.

*1-benzyl-3-cyclopentylquinoxalin-2(1H)-one (3be)*



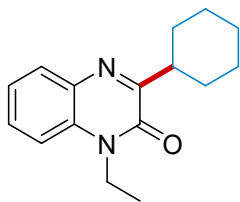
Purification by flash column chromatography (PE:EA, 10:1 v/v) to provide **3bd**. White solid (31 mg, 51% yield). m.p. 82.6 – 84.1 °C;  $^1\text{H}$  NMR (400 MHz, Chloroform- $d$ )  $\delta$  7.86 (dd,  $J = 7.9, 1.6$  Hz, 1H), 7.42 – 7.25 (m, 8H), 5.53 (s, 2H), 3.81 (p,  $J = 8.2$  Hz, 1H), 2.18 – 2.10 (m, 2H), 2.06 – 1.67 (m, 6H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform- $d$ )  $\delta$  163.9, 155.0, 135.5, 133.0, 132.3, 129.9, 129.3, 128.9, 127.6, 126.9, 123.4, 114.3, 45.9, 42.7, 30.9, 26.0. HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{20}\text{H}_{21}\text{N}_2\text{O}$   $[\text{M} + \text{H}]^+$ , 305.1648; found, 305.1660.

*3-cyclohexyl-1-methylquinoxalin-2(1H)-one (3ca)<sup>3, 5</sup>*



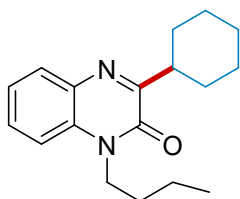
Purification by flash column chromatography (PE:EA, 10:1 v/v) to provide **3ca**. White solid (39 mg, 81% yield);  $^1\text{H}$  NMR (400 MHz, Chloroform- $d$ )  $\delta$  7.85 (d,  $J = 8.9$  Hz, 1H), 7.55 – 7.46 (m, 1H), 7.37 – 7.26 (m, 2H), 3.71 (s, 3H), 3.39 – 3.32 (m, 1H), 1.97 (d,  $J = 11.5$  Hz, 2H), 1.88 (d,  $J = 12.6$  Hz, 2H), 1.78 (d,  $J = 10.3$  Hz, 1H), 1.65 – 1.42 (m, 4H), 1.38 – 1.28 (m, 1H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (101 MHz, Chloroform- $d$ )  $\delta$  164.3, 154.5, 132.89, 132.85, 129.8, 129.4, 123.4, 113.4, 40.8, 30.5, 29.1, 26.3, 26.2.

*3-cyclohexyl-1-ethylquinoxalin-2(1H)-one (3cb)*



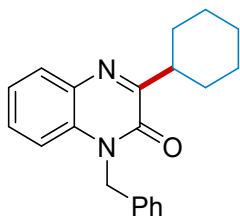
Purification by flash column chromatography (PE:EA, 10:1 v/v) to provide **3cb**. White solid (35 mg, 68% yield); m.p. 67.9 – 68.2 °C; <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.85 (dd, *J* = 8.3, 1.6 Hz, 1H), 7.52 – 7.48 (m, 1H), 7.34 – 7.28 (m, 2H), 4.32 (q, *J* = 7.2 Hz, 2H), 3.41 – 3.28 (m, 1H), δ 2.01 – 1.73 (m, 5H), 1.66 – 1.44 (m, 4H), 1.38 (t, *J* = 7.2 Hz, 3H), 1.34 – 1.25 (m, 1H). <sup>13</sup>C{<sup>1</sup>H} NMR (101 MHz, Chloroform-*d*) δ 164.3, 154.0, 133.2, 131.7, 130.0, 129.3, 123.1, 113.3, 40.6, 37.3, 30.6, 26.3, 26.2, 12.4. HRMS (ESI) *m/z*: calcd for C<sub>16</sub>H<sub>21</sub>N<sub>2</sub>O [M + H]<sup>+</sup>, 257.1648; found, 257.1643

*1-butyl-3-cyclohexylquinoxalin-2(1H)-one (3cc)*



Purification by flash column chromatography (PE:EA, 15:1 v/v) to provide **3cc**. colorless liquid (44 mg, 78% yield); <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.85 (dd, *J* = 7.9, 1.3 Hz, 1H), 7.53 – 7.45 (m, 1H), 7.35 – 7.24 (m, 2H), 4.25 (t, *J* = 8 Hz, 2H), 3.45 – 3.29 (m, 1H), 1.97 (d, *J* = 11.1 Hz, 2H), 1.88 (d, *J* = 12.7 Hz, 2H), 1.82 – 1.72 (m, 3H), 1.64 – 1.43 (m, 6H), 1.35 – 1.24 (m, 1H), 1.01 (t, *J* = 7.4 Hz, 3H). <sup>13</sup>C{<sup>1</sup>H} NMR (101 MHz, Chloroform-*d*) δ 164.2, 154.2, 133.2, 132.0, 130.0, 129.3, 123.1, 113.4, 42.1, 40.7, 30.5, 29.3, 26.4, 26.2, 20.3, 13.8. HRMS (ESI) *m/z*: calcd for C<sub>18</sub>H<sub>25</sub>N<sub>2</sub>O [M + H]<sup>+</sup>, 285.1961; found, 285.1964

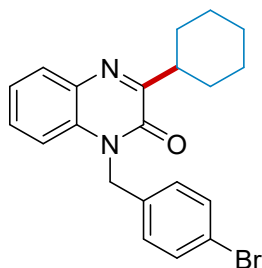
*1-benzyl-3-cyclohexylquinoxalin-2(1H)-one (3cd)*



Purification by flash column chromatography (PE:EA, 10:1 v/v) to provide **3cd**. White solid (42 mg, 66% yield); m.p. 131.8 – 133.2 °C; <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.88 (dd, *J* = 7.9, 1.4 Hz, 1H), 7.42 – 7.38 (m, 1H), 7.36 – 7.26 (m, 7H), 5.52 (s, 2H), 3.47 – 3.39 (m, 1H), 2.04 (d, *J* = 11.4 Hz, 2H), 1.91 (dt, *J* = 12.7, 3.0 Hz, 2H), 1.84 – 1.78 (m, 1H), 1.68 – 1.62 (m, 2H), 1.56 – 1.45 (m, 2H), 1.41 – 1.33 (m, 1H). <sup>13</sup>C{<sup>1</sup>H} NMR (101 MHz, Chloroform-*d*) δ 164.4, 154.6, 135.5, 133.2, 132.2, 129.9, 129.4, 128.9, 127.6, 126.9, 123.4, 114.2, 45.9, 40.8, 30.6, 26.4, 26.2. HRMS (ESI) *m/z*: calcd for C<sub>21</sub>H<sub>23</sub>N<sub>2</sub>O [M + H]<sup>+</sup>, 319.1085; found, 319.1082.

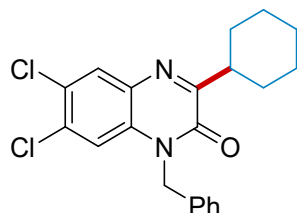
*1-(4-bromobenzyl)-3-cyclohexylquinoxalin-2(1H)-one (3ce)*





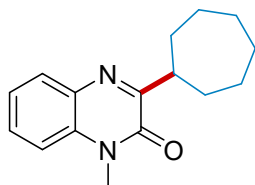
Purification by flash column chromatography (PE:EA, 10:1 v/v) to provide **3ce**. White solid (60 mg, 76% yield); m.p. 155.2 – 157.0 °C;  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.87 (dd,  $J$  = 7.9, 1.6 Hz, 1H), 7.43 (dd,  $J$  = 20.2, 7.7 Hz, 3H), 7.34 – 7.29 (m, 1H), 7.17 (dd,  $J$  = 16.3, 8.3 Hz, 3H), 5.45 (s, 2H), 3.40 (tt,  $J$  = 11.6, 3.2 Hz, 1H), 2.06 – 1.98 (m, 2H), 1.93 – 1.88 (m, 2H), 1.83 – 1.77 (m, 1H), 1.66 – 1.47 (m, 4H), 1.36 (tt,  $J$  = 12.5, 3.4 Hz, 1H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  164.4, 154.5, 134.6, 133.2, 132.0, 131.9, 130.1, 129.5, 128.7, 123.6, 121.6, 114.0, 45.4, 40.8, 30.6, 26.3, 26.2. HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{21}\text{H}_{22}^{79}\text{BrN}_2\text{O}$  [ $\text{M} + \text{H}$ ] $^+$ , 397.0910; found, 397.0915.

*1-benzyl-6,7-dichloro-3-cyclohexylquinoxalin-2(1H)-one (3cf)*



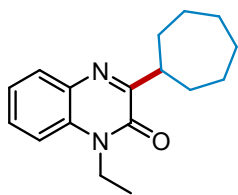
Purification by flash column chromatography (PE:EA, 10:1 v/v) to provide **3cf**. White solid (42 mg, 54% yield); m.p. 168.4 – 169.9 °C;  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.94 (s, 1H), 7.34 (dt,  $J$  = 14.7, 8.3 Hz, 4H), 7.25 (d,  $J$  = 7.0 Hz, 2H), 5.43 (s, 2H), 3.43 – 3.36 (m, 2H), 2.01 (d,  $J$  = 12.2 Hz, 2H), 1.91 (d,  $J$  = 12.5 Hz, 2H), 1.80 (d,  $J$  = 12.8 Hz, 1H), 1.61 – 1.47 (m, 4H), 1.38 – 1.31 (m, 1H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  166.0, 154.0, 134.6, 133.3, 132.3, 131.6, 130.7, 129.1, 128.0, 127.2, 126.9, 115.6, 46.2, 40.9, 30.5, 26.2, 26.1. HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{21}\text{H}_{21}\text{Cl}_2\text{N}_2\text{O}$  [ $\text{M} + \text{H}$ ] $^+$ , 387.1025; found, 387.1018.

*3-cycloheptyl-1-methylquinoxalin-2(1H)-one (3da)*



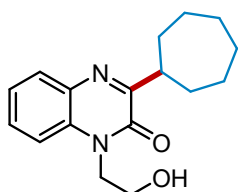
Purification by flash column chromatography (PE:EA, 15:1 v/v) to provide **3da**. White solid (35 mg, 69% yield); m.p. 101.3 – 102.4 °C;  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.84 (dd,  $J$  = 8.0, 1.3 Hz, 1H), 7.56 – 7.47 (m, 1H), 7.37 – 7.26 (m, 2H), 3.71 (s, 3H), 3.53 – 3.46 (m, 1H), 2.03 – 1.94 (m, 2H), 1.88 – 1.80 (m, 4H), 1.73 – 1.60 (m, 6H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  165.3, 154.4, 132.9, 132.8, 129.7, 129.3, 123.4, 113.4, 42.4, 32.3, 29.1, 28.2, 27.1. HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{16}\text{H}_{21}\text{N}_2\text{O}$  [ $\text{M} + \text{H}$ ] $^+$ , 257.1648; found, 257.1644.

*3-cycloheptyl-1-ethylquinoxalin-2(1H)-one (3db)*



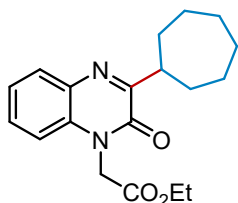
Purification by flash column chromatography (PE:EA, 15:1 v/v) to provide **3db**. colorless liquid (39 mg, 72% yield);  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.88 – 7.83 (m, 1H), 7.54 – 7.48 (m, 1H), 7.35 – 7.29 (m, 2H), 4.33 (q,  $J = 7.2$  Hz, 2H), 3.54 – 3.48 (m, 1H), 2.03 – 1.95 (m, 2H), 1.88 – 1.62 (m, 10H), 1.39 (t,  $J = 7.2$  Hz, 3H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  165.4, 153.9, 133.1, 131.8, 130.0, 129.2, 123.2, 113.3, 42.3, 37.3, 32.3, 28.2, 27.2, 12.4. HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{17}\text{H}_{23}\text{N}_2\text{O}$   $[\text{M} + \text{H}]^+$ , 271.1805; found, 271.1809.

*3-cycloheptyl-1-(2-hydroxyethyl)quinoxalin-2(1H)-one (3dc)*



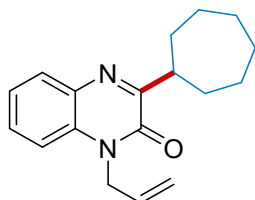
Purification by flash column chromatography (PE:EA, 5:1 v/v) to provide **3dc**. White solid (31 mg, 54% yield); m.p. 87.9 – 88.4 °C;  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.87 (dd,  $J = 8.0, 1.6$  Hz, 1H), 7.54 – 7.50 (m, 1H), 7.43 – 7.31 (m, 2H), 4.51 (t,  $J = 5.6$  Hz, 2H), 4.06 (q,  $J = 5.5$  Hz, 2H), 3.51 – 3.44 (m, 1H), 2.73 (t,  $J = 5.4$  Hz, 1H), 2.02 – 1.96 (m, 2H), 1.88 – 1.51 (m, 10H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  165.1, 155.4, 133.2, 132.2, 130.1, 129.4, 123.7, 113.6, 60.6, 45.0, 42.3, 32.3, 28.2, 27.1. HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{17}\text{H}_{23}\text{N}_2\text{O}_2$   $[\text{M} + \text{H}]^+$ , 287.1754; found, 287.1758.

*Ethyl 2-(3-cycloheptyl-2-oxoquinoxalin-1(2H)-yl)acetate (3dd)*



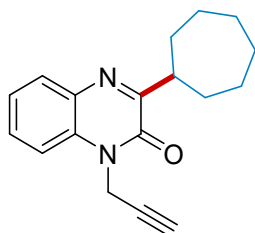
Purification by flash column chromatography (PE:EA, 10:1 v/v) to provide **3dd**. Colorless liquid (27 mg, 41% yield);  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.87 (dd,  $J = 7.9, 1.5$  Hz, 1H), 7.51 – 7.45 (m, 1H), 7.38 – 7.31 (m, 1H), 7.06 (dd,  $J = 8.4, 1.2$  Hz, 1H), 5.04 (s, 2H), 4.26 (q,  $J = 7.1$  Hz, 2H), 3.54 – 3.43 (m, 1H), 2.04 – 1.98 (m, 1H), 1.87 – 1.63 (m, 10H), 1.29 (t,  $J = 7.1$  Hz, 3H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  167.3, 165.1, 154.0, 132.8, 132.0, 130.1, 129.5, 123.7, 112.9, 62.0, 43.6, 42.4, 32.2, 28.2, 27.1, 14.1. HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{19}\text{H}_{25}\text{N}_2\text{O}_3$   $[\text{M} + \text{H}]^+$ , 329.1860; found, 329.1863.

*1-allyl-3-cycloheptylquinoxalin-2(1H)-one (3de)*



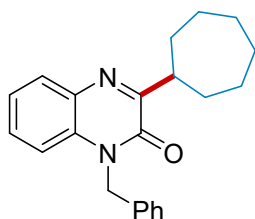
Purification by flash column chromatography (PE:EA, 15:1 v/v) to provide **3de**. White solid (27 mg, 48% yield); m.p. 84.2 – 85.9 °C; <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.92 – 7.81 (m, 1H), 7.51 – 7.48 (m, 1H), 7.35 – 7.22 (m, 2H), 6.05 – 5.91 (m, 1H), 5.35 – 5.15 (m, 2H), 4.93 (dt, *J* = 5.2, 1.8 Hz, 2H), 3.52 (tt, *J* = 9.8, 3.6 Hz, 1H), 2.01 (dq, *J* = 12.8, 4.3, 3.7 Hz, 2H), 1.91 – 1.60 (m, 10H). <sup>13</sup>C{<sup>1</sup>H} NMR (101 MHz, Chloroform-*d*) δ 165.4, 154.0, 132.9, 132.1, 130.8, 129.8, 129.2, 123.4, 117.9, 114.0, 44.6, 42.4, 32.3, 28.2, 27.2. HRMS (ESI) *m/z*: calcd for C<sub>18</sub>H<sub>23</sub>N<sub>2</sub>O [M + H]<sup>+</sup>, 283.1805; found, 283.1811.

*3-cycloheptyl-1-(prop-2-yn-1-yl)quinoxalin-2(1H)-one (3df)*



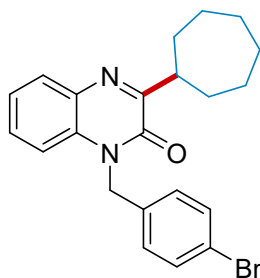
Purification by flash column chromatography (PE:EA, 15:1 v/v) to provide **3df**. White solid (38 mg, 72% yield); m.p. 108.2 – 109.7 °C; <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.86 (dd, *J* = 8.0, 1.3 Hz, 1H), 7.57 – 7.53 (m, 1H), 7.45 (d, *J* = 7.5 Hz, 1H), 7.41 – 7.32 (m, 1H), 5.07 (d, *J* = 2.5 Hz, 2H), 3.55 – 3.43 (m, 1H), 2.31 (t, *J* = 2.5 Hz, 1H), 2.02 – 1.96 (m, 2H), 1.93 – 1.54 (m, 10H). <sup>13</sup>C{<sup>1</sup>H} NMR (101 MHz, Chloroform-*d*) δ 165.2, 153.4, 133.0, 131.4, 129.9, 129.4, 123.8, 113.9, 77.0, 73.1, 42.4, 32.3, 31.5, 28.2, 27.1. HRMS (ESI) *m/z*: calcd for C<sub>18</sub>H<sub>21</sub>N<sub>2</sub>O [M + H]<sup>+</sup>, 281.1648; found, 281.1645.

*1-benzyl-3-cycloheptylquinoxalin-2(1H)-one (3dg)*



Purification by flash column chromatography (PE:EA, 15:1 v/v) to provide **3dg**. Yellow solid (42 mg, 63% yield); m.p. 115.4 – 116.2 °C; <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.86 (dd, *J* = 7.9, 1.5 Hz, 1H), 7.43 – 7.25 (m, 8H), 5.52 (s, 2H), 3.66 – 3.47 (m, 1H), 2.09 – 2.03 (m, 2H), 1.93 – 1.63 (m, 10H). <sup>13</sup>C{<sup>1</sup>H} NMR (101 MHz, Chloroform-*d*) δ 165.5, 154.5, 135.5, 133.0, 132.2, 129.9, 129.3, 128.9, 127.6, 126.9, 123.5, 114.3, 46.0, 42.5, 32.4, 28.2, 27.2. HRMS (ESI) *m/z*: calcd for C<sub>22</sub>H<sub>25</sub>N<sub>2</sub>O [M + H]<sup>+</sup>, 333.1961; found, 333.1959.

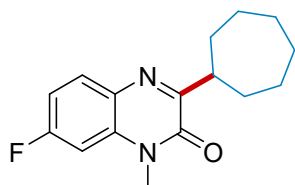
*1-(4-bromobenzyl)-3-cycloheptylquinoxalin-2(1H)-one (3dh)*



Purification by flash column chromatography (PE:EA, 15:1 v/v) to provide **3dh**. White solid (53 mg,

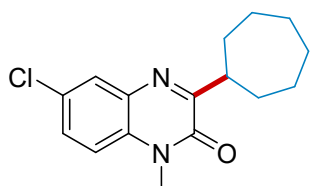
65% yield); m.p. 130.0 – 131.8 °C; <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.86 (dd, *J* = 7.9, 1.4 Hz, 1H), δ 7.44 (d, *J* = 8.4 Hz, 1H), 7.39 (d, *J* = 7.2 Hz, 0H), 7.33 – 7.28 (m, 1H), δ 7.18 (d, *J* = 7.6 Hz, 1H), 7.14 (d, *J* = 8.4 Hz, 2H), 5.45 (s, 2H), 3.59 – 3.52 (m, 1H), 2.08 – 2.00 (m, 2H), 1.90 – 1.63 (m, 10H). <sup>13</sup>C{<sup>1</sup>H} NMR (101 MHz, Chloroform-*d*) δ 165.5, 154.4, 134.5, 133.0, 132.04, 132.00, 130.0, 129.4, 128.7, 123.6, 121.6, 114.0, 45.4, 42.4, 32.4, 28.2, 27.2. HRMS (ESI) *m/z*: calcd for C<sub>22</sub>H<sub>24</sub><sup>79</sup>BrN<sub>2</sub>O [M + H]<sup>+</sup>, 397.0910; found, 397.0915.

*3-cycloheptyl-7-fluoro-1-methylquinoxalin-2(1H)-one (3di)*



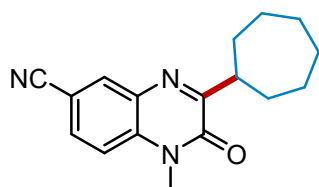
Purification by flash column chromatography (PE:EA, 15:1 v/v) to provide **3di**. White solid (88 mg, 62% yield); m.p. 119.8 – 120.2 °C; <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.59 – 7.51 (m, 1H), 7.31 – 7.23 (m, 2H), 3.71 (s, 3H), 3.54 – 3.48 (m, 1H), 2.01 – 1.94 (m, 2H), 1.87 – 1.62 (m, 10H). <sup>13</sup>C{<sup>1</sup>H} NMR (101 MHz, Chloroform-*d*) δ 166.9, 158.6 (d, *J* = 243.1 Hz), 154.1, 133.4 (d, *J* = 11.2 Hz), 129.5, 116.9 (d, *J* = 23.9 Hz), 115.3, 114.4 (d, *J* = 9.0 Hz), 42.4, 32.2, 29.3, 28.2, 27.1. <sup>19</sup>F NMR (376 MHz, Chloroform-*d*) δ -119.50. HRMS (ESI) *m/z*: calcd for C<sub>16</sub>H<sub>20</sub>FN<sub>2</sub>O [M + H]<sup>+</sup>, 275.1554; found, 275.1552.

*6-chloro-3-cycloheptyl-1-methylquinoxalin-2(1H)-one (3dj)*



Purification by flash column chromatography (PE:EA, 15:1 v/v) to provide **3dj**. Yellow solid (56 mg, 62% yield); m.p. 123.4 – 124.4 °C; <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.85 (d, *J* = 2.4 Hz, 1H), 7.46 (d, *J* = 2.4 Hz, 1H), 7.26 (d, *J* = 19.7 Hz, 1H), 3.70 (s, 3H), 3.54 – 3.47 (m, 1H), 2.04 – 1.91 (m, 2H), 1.88 – 1.60 (m, 10H). <sup>13</sup>C{<sup>1</sup>H} NMR (101 MHz, Chloroform-*d*) δ 166.8, 154.1, 133.3, 131.6, 129.2, 129.1, 128.7, 114.5, 42.3, 32.2, 29.3, 28.2, 27.0. HRMS (ESI) *m/z*: calcd for C<sub>16</sub>H<sub>20</sub>ClN<sub>2</sub>O [M + H]<sup>+</sup>, 291.1259; found, 291.1262.

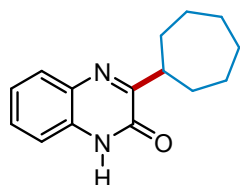
*3-cycloheptyl-1-methyl-2-oxo-1,2-dihydroquinoxaline-6-carbonitrile (3dk)*



Purification by flash column chromatography (PE:EA, 10:1 v/v) to provide **3dk**. White solid (9 mg, 16% yield); m.p. 192.8 – 193.5 °C; <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.92 (d, *J* = 8.6 Hz, 1H), 7.61 – 7.57 (m, 2H), 3.72 (s, 3H), 3.58 – 3.47 (m, 1H), 2.01 – 1.95 (m, 2H), 1.91 – 1.56 (m, 10H). <sup>13</sup>C NMR (101

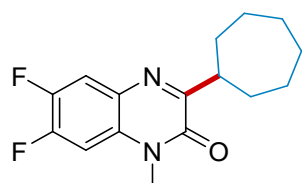
MHz, Chloroform-*d*)  $\delta$  169.0, 153.9, 135.0, 133.3, 130.6, 126.4, 118.3, 117.6, 112.4, 42.6, 32.2, 29.3, 28.2, 27.0. HRMS (ESI) *m/z*: calcd for C<sub>17</sub>H<sub>19</sub>N<sub>3</sub>O [M + H]<sup>+</sup>, 282.1601; found, 282.1618.

*3-cycloheptylquinoxalin-2(1H)-one (3cl)*<sup>6</sup>



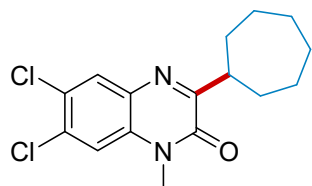
Purification by flash column chromatography (PE:EA, 5:1 v/v) to provide **3cl**. White solid (40 mg, 83% yield); <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  12.40 (s, 1H), 7.85 (d, *J* = 7.9 Hz, 1H), 7.50 (t, *J* = 7.7 Hz, 1H), 7.41 – 7.32 (m, 2H), 3.57 – 3.53 (m, 1H), 2.10 – 2.00 (m, 2H), 1.96 – 1.60 (m, 10H). <sup>13</sup>C{<sup>1</sup>H} NMR (101 MHz, Chloroform-*d*)  $\delta$  166.0, 155.9, 132.9, 130.7, 129.4, 128.8, 123.9, 115.4, 41.8, 32.3, 28.3, 27.1.

*3-cycloheptyl-6,7-difluoro-1-methylquinoxalin-2(1H)-one (3dm)*



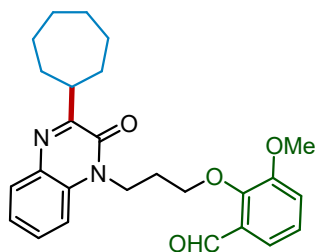
Purification by flash column chromatography (PE:EA, 10:1 v/v) to provide **3dm**. White solid (46 mg, 79% yield); m.p. 153.9 – 154.8 °C; <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.65 (dd, *J* = 10.2, 8.4 Hz, 1H), 7.09 (dd, *J* = 11.4, 7.1 Hz, 1H), 3.66 (s, 3H), 3.47 (dt, *J* = 9.7, 5.9 Hz, 1H), 2.00 – 1.91 (m, 2H), 1.87 – 1.63 (m, 10H). <sup>13</sup>C{<sup>1</sup>H} NMR (101 MHz, Chloroform-*d*)  $\delta$  165.9 (d, *J* = 3.4 Hz), 154.0, 150.8 (dd, *J* = 251.8, 14.5 Hz), 146.5 (dd, *J* = 246.3, 13.9 Hz), 130.0 (d, *J* = 8.8 Hz), 129.0 (d, *J* = 12.0 Hz), 117.3 (dd, *J* = 17.9, 2.2 Hz), 102.0 (d, *J* = 23.1 Hz), 42.3, 32.2, 29.6, 28.2, 27.0. <sup>19</sup>F NMR (376 MHz, Chloroform-*d*)  $\delta$  -132.45 (d, *J* = 22.7 Hz), -142.75 (d, *J* = 22.7 Hz). HRMS (ESI) *m/z*: calcd for C<sub>16</sub>H<sub>19</sub>F<sub>2</sub>N<sub>2</sub>O [M + H]<sup>+</sup>, 293.1460; found, 293.1455.

*6,7-dichloro-3-cycloheptyl-1-methylquinoxalin-2(1H)-one (3dn)*



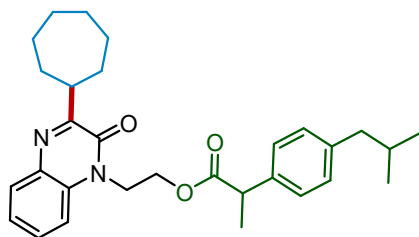
Purification by flash column chromatography (PE:EA, 10:1 v/v) to provide **3dn**. Yellow solid (34 mg, 52% yield); m.p. 136.9 – 137.4 °C; <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.91 (s, 1H), 7.37 (s, 1H), 3.66 (s, 3H), 3.52 – 3.40 (m, 1H), 1.98 – 1.93 (m, 2H), 1.89 – 1.57 (m, 10H). <sup>13</sup>C{<sup>1</sup>H} NMR (101 MHz, Chloroform-*d*)  $\delta$  166.9, 153.9, 133.2, 132.3, 131.9, 130.5, 127.1, 114.9, 42.3, 32.2, 29.3, 28.2, 27.0. HRMS (ESI) *m/z*: calcd for C<sub>16</sub>H<sub>19</sub><sup>35,37</sup>Cl<sub>2</sub>N<sub>2</sub>O [M + H]<sup>+</sup>, 325.0869; found, 325.0864.

*2-(3-(3-cycloheptyl-2-oxoquinoxalin-1(2H)-yl)propoxy)-3-methoxybenzaldehyde (3do)*



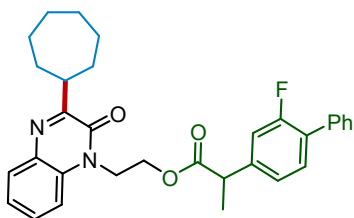
Purification by flash column chromatography (PE:EA, 10:1 v/v) to provide **3do**. White solid (26 mg, 61% yield); m.p. 64.2 – 65.6 °C; <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 10.52 (s, 1H), 7.97 (dd, *J* = 8.2, 1.5 Hz, 1H), 7.81 (dd, *J* = 8.1, 1.5 Hz, 1H), 7.63 – 7.57 (m, 1H), 7.56 – 7.50 (m, 1H), 7.48 – 7.43 (m, 1H), 7.19 – 7.14 (m, 2H), 4.77 (t, *J* = 6.2 Hz, 2H), 4.39 (t, *J* = 6.3 Hz, 2H), 3.85 (s, 3H), 3.38 – 3.31 (m, 1H), 2.41 (q, *J* = 6.2 Hz, 2H), 2.06 – 1.80 (m, 7H), 1.75 – 1.59 (m, 6H). <sup>13</sup>C{<sup>1</sup>H} NMR (101 MHz, Chloroform-*d*) δ 190.0, 155.7, 155.1, 153.0, 151.7, 139.4, 138.5, 130.0, 128.7, 128.4, 126.6, 126.2, 124.2, 119.3, 118.1, 71.8, 62.9, 56.0, 42.7, 32.5, 29.7, 28.1, 27.3. HRMS (ESI) *m/z*: calcd for C<sub>26</sub>H<sub>31</sub>N<sub>2</sub>O<sub>4</sub> [M + H]<sup>+</sup>, 435.2278; found, 435.2283.

2-(3-cycloheptyl-2-oxoquinoxalin-1(2H)-yl)ethyl 2-(4-isobutylphenyl)propanoate (**3dp**)



Purification by flash column chromatography (PE:EA, 10:1 v/v) to provide **3dp**. Colorless liquid (27 mg, 56% yield); <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.84 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.47 – 7.43 (m, 1H), 7.35 (dd, *J* = 11.7, 8.1 Hz, 2H), 7.13 – 7.03 (m, 4H), 4.60 – 4.35 (m, 4H), 3.61 (q, *J* = 7.2 Hz, 1H), 3.52 – 3.38 (m, 1H), 2.46 (d, *J* = 7.2 Hz, 2H), 2.05 – 1.96 (m, 2H), 1.92 – 1.59 (m, 11H), 1.44 (d, *J* = 7.2 Hz, 3H), 0.92 (d, *J* = 6.6 Hz, 6H). <sup>13</sup>C{<sup>1</sup>H} NMR (101 MHz, Chloroform-*d*) δ 174.7, 173.7, 165.1, 154.2, 140.7, 137.2, 132.9, 132.4, 129.9, 129.4, 127.1, 123.5, 113.6, 61.1, 45.0, 45.0, 42.3, 40.9, 32.3, 32.2, 30.2, 28.2, 27.1, 22.4, 18.3. HRMS (ESI) *m/z*: calcd for C<sub>30</sub>H<sub>39</sub>N<sub>2</sub>O<sub>3</sub> [M + H]<sup>+</sup>, 475.2955; found, 475.2958.

2-(3-cycloheptyl-2-oxoquinoxalin-1(2H)-yl)ethyl 2-(3-fluoro-[1,1'-biphenyl]-4-yl)propanoate (**3dq**)

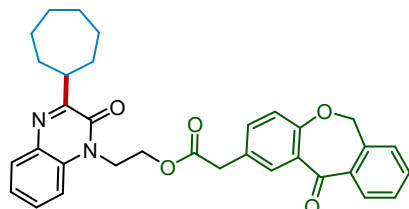


Purification by flash column chromatography (PE:EA, 10:1 v/v) to provide **3dq**. Colorless liquid (20 mg, 39% yield); <sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 7.84 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.57 – 7.54 (m, 2H), 7.49 – 7.44 (m, 3H), 7.41 – 7.30 (m, 4H), 7.03 – 7.00 (m, 2H), 4.61 – 4.45 (m, 4H), 3.66 (q, *J* = 7.2 Hz, 1H), 3.51 – 3.45 (m, 1H), 2.03 – 1.97 (m, 2H), 1.87 – 1.62 (m, 10H), 1.48 (d, *J* = 7.2 Hz, 3H). <sup>13</sup>C{<sup>1</sup>H} NMR (101 MHz, Chloroform-*d*) δ 173.9, 165.1, 159.6 (d, *J* = 248.6 Hz), 154.2, 141.2 (d, *J* = 7.7 Hz),

135.4, 132.9, 132.3, 130.8 (d,  $J = 3.9$  Hz), 130.0, 129.3, 128.9 (d,  $J = 3.0$  Hz), 128.4, 127.9 (d,  $J = 13.5$  Hz), 127.7, 123.5, 123.4 (d,  $J = 3.4$  Hz), 115.2 (d,  $J = 23.6$  Hz), 113.4, 61.5, 44.9 (d,  $J = 1.4$  Hz), 42.3, 40.9, 32.3 (d,  $J = 3.0$  Hz), 28.2, 27.1, 18.2.  $^{19}\text{F}$  NMR (376 MHz, Chloroform- $d$ )  $\delta$  -117.32. HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{32}\text{H}_{34}\text{FN}_2\text{O}_3$   $[\text{M} + \text{H}]^+$ , 513.2548; found, 513.2555.

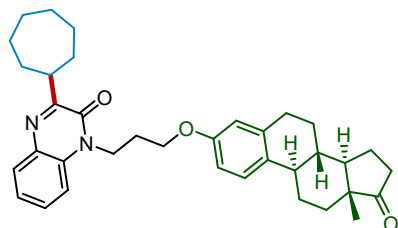
2-(3-cycloheptyl-2-oxoquinoxalin-1(2H)-yl)ethyl acetate (**3dr**)

2-(11-oxo-6,11-dihydrodibenzo[*b,e*]oxepin-2-yl)acetate (**3dr**)



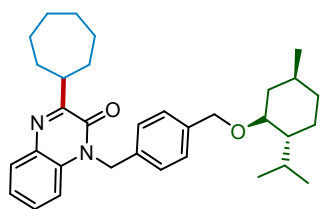
Purification by flash column chromatography (PE:EA, 2:1 v/v) to provide **3dr**. Colorless liquid (23 mg, 42% yield);  $^1\text{H}$  NMR (400 MHz, Chloroform- $d$ )  $\delta$  8.06 (d,  $J = 2.3$  Hz, 1H), 7.91 (dd,  $J = 7.7, 1.4$  Hz, 1H), 7.83 (dd,  $J = 8.0, 1.5$  Hz, 1H), 7.58 (dd,  $J = 7.5, 1.5$  Hz, 1H), 7.53 – 7.46 (m, 2H), 7.39 (dt,  $J = 7.3, 1.6$  Hz, 2H), 7.35 – 7.29 (m, 2H), 7.00 (d,  $J = 8.4$  Hz, 1H), 5.21 (s, 2H), 4.59 – 4.46 (m, 4H), 3.58 (s, 2H), 3.51 – 3.46 (m, 1H), 2.04 – 1.94 (m, 2H), 1.92 – 1.58 (m, 10H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (101 MHz, Chloroform- $d$ )  $\delta$  190.7, 171.4, 165.1, 160.5, 154.3, 140.4, 136.3, 135.5, 132.9, 132.8, 132.5, 132.3, 130.0, 129.5, 129.4, 129.3, 127.8, 127.2, 125.1, 123.5, 121.1, 113.4, 73.6, 61.3, 42.3, 40.9, 39.9, 32.3, 28.2, 27.1. HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{33}\text{H}_{33}\text{N}_2\text{O}_5$   $[\text{M} + \text{H}]^+$ , 537.2384; found, 537.2391.

3-cycloheptyl-1-(3-(((8*S*,9*R*,13*R*,14*R*)-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6*H*-cyclopenta[*a*]phenanthren-3-yl)oxy)propyl)quinoxalin-2(1*H*)-one (**3ds**)



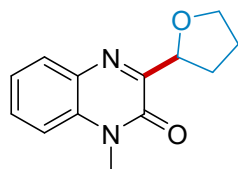
Purification by flash column chromatography (PE:EA, 15:1 v/v) to provide **3ds**. White solid (19 mg, 35% yield); m.p. 68.7 – 69.2 °C;  $^1\text{H}$  NMR (400 MHz, Chloroform- $d$ )  $\delta$  7.86 (dd,  $J = 8.0, 1.5$  Hz, 1H), 7.54 – 7.43 (m, 2H), 7.38 – 7.31 (m, 1H), 7.23 (d,  $J = 8.6$  Hz, 1H), 6.75 (dd,  $J = 8.6, 2.8$  Hz, 1H), 6.67 (d,  $J = 2.7$  Hz, 1H), 4.54 – 4.43 (m, 2H), 4.10 (t,  $J = 5.8$  Hz, 2H), 3.52 – 3.41 (m, 1H), 2.98 – 2.87 (m, 2H), 2.57 – 2.50 (m, 1H), 2.42 (dd,  $J = 8.0, 4.8$  Hz, 1H), 3.22 – 2.29 (m, 3H), 2.13 – 1.96 (m, 5H), 1.86 – 1.53 (m, 17H), 0.94 (s, 3H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (101 MHz, Chloroform- $d$ )  $\delta$  165.2, 156.6, 154.3, 137.8, 133.0, 132.4, 132.1, 130, 129.4, 126.4, 123.3, 114.6, 113.5, 112.2, 65.4, 50.4, 48.0, 44.0, 42.4, 39.8, 38.4, 35.9, 32.3, 31.6, 29.7, 28.2, 27.3, 27.2, 26.6, 25.9, 21.6, 13.9. HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{36}\text{H}_{45}\text{N}_2\text{O}_3$   $[\text{M} + \text{H}]^+$ , 553.3425; found, 553.3435.

3-cycloheptyl-1-(4-(((1*R*,2*R*,5*S*)-2-isopropyl-5-methylcyclohexyl)oxy)methyl)benzyl)quinoxalin-2(1*H*)-one (**3dt**)



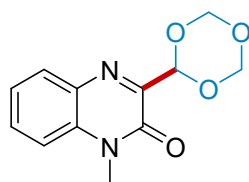
Purification by flash column chromatography (PE:EA, 15:1 v/v) to provide **3dt**. Colorless liquid (31 mg, 62% yield); m.p. 68.7 – 69.2 °C;  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.85 (dd,  $J = 7.9, 1.6$  Hz, 1H), 7.39 – 7.35 (m, 1H), 7.34 – 7.28 (m, 3H), 7.25 – 7.20 (m, 3H), 5.51 (s, 2H), 4.63 (d,  $J = 11.5$  Hz, 1H), 4.37 (d,  $J = 11.5$  Hz, 1H), 3.60 – 3.53 (m, 1H), 3.16 (td,  $J = 10.5, 4.1$  Hz, 1H), 3.23 – 2.32 (m, 1H), 2.20 – 2.15 (m, 1H), 2.09 – 2.02 (m, 2H), 1.91 – 1.83 (m, 4H), 1.78 – 1.60 (m, 8H), 1.40 – 1.23 (m, 2H), 1.02 – 0.84 (m, 9H), 0.69 (d,  $J = 6.9$  Hz, 3H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  165.5, 154.5, 138.6, 134.6, 133.0, 132.2, 129.8, 129.3, 128.4, 126.9, 123.4, 114.3, 78.9, 70.0, 48.3, 45.8, 42.5, 40.3, 34.6, 32.4, 31.6, 28.2, 27.2, 25.5, 23.2, 22.4, 21.0, 16.0. HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{33}\text{H}_{45}\text{N}_2\text{O}_2$  [ $\text{M} + \text{H}$ ] $^+$ , 501.3476; found, 501.3482.

*1-methyl-3-(tetrahydrofuran-2-yl)quinoxalin-2(1H)-one (3ea)*<sup>3, 7</sup>



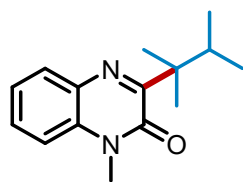
Purification by flash column chromatography (PE:EA, 5:1 v/v) to provide **3ea**. White solid (30 mg, 63% yield);  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.94 (dd,  $J = 8.0, 1.2$  Hz, 1H), 7.59 – 7.49 (m, 1H), 7.37 – 7.25 (m, 2H), 5.38 (dd,  $J = 7.5, 5.9$  Hz, 1H), 4.22 (q,  $J = 7.3$  Hz, 1H), 4.06 – 3.91 (m, 1H), 3.68 (s, 3H), 2.55 – 2.46 (m, 1H), 2.06 – 2.01 (m, 3H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  159.4, 154.0, 133.1, 132.5, 130.4, 130.1, 123.6, 113.5, 77.6, 69.2, 30.5, 28.8, 25.6.

*1-methyl-3-(1,3,5-trioxan-2-yl)quinoxalin-2(1H)-one (3fa)*<sup>3</sup>



Purification by flash column chromatography (PE:EA, 2:1 v/v) to provide **3fa**. White solid (26 mg, 53% yield);  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  8.09 (dd,  $J = 8.0, 1.1$  Hz, 1H), 7.68 – 7.60 (m, 1H), 7.44 – 7.33 (m, 2H), 6.51 (s, 1H), 5.44 (q,  $J = 14.2, 7.9$  Hz, 4H), 3.73 (s, 3H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  153.6, 150.8, 133.6, 132.3, 131.9, 131.4, 124.1, 113.7, 96.4, 94.0, 29.1.

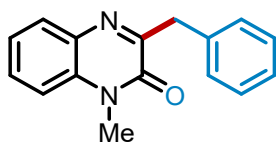
*3-(2,3-dimethylbutan-2-yl)-1-methylquinoxalin-2(1H)-one (3ga)*





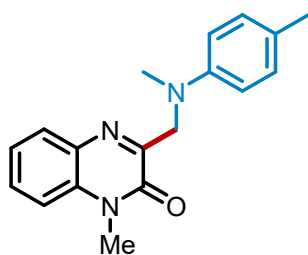
Purification by flash column chromatography (PE:EA, 15:1 v/v) to provide **3ga**. Colorless liquid (22 mg, 46% yield);  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.86 (dd,  $J = 8.0, 1.6$  Hz, 1H), 7.52 (ddd,  $J = 8.3, 7.3, 1.5$  Hz, 1H), 7.36 – 7.27 (m, 2H), 3.69 (s, 3H), 3.05 – 2.94 (m,  $J = 6.9$  Hz, 1H), 1.39 (s, 6H), 0.84 (d,  $J = 6.9$  Hz, 6H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  165.6, 153.8, 133.2, 132.1, 130.2, 129.4, 123.1, 113.2, 46.0, 31.9, 28.8, 22.0, 18.0. HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{15}\text{H}_{21}\text{N}_2\text{O}$  [ $\text{M} + \text{H}$ ] $^+$ , 245.1648; found, 245.1664.

*3-benzyl-1-methylquinoxalin-2(1H)-one (3ha)*<sup>2b</sup>



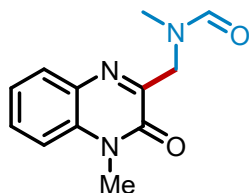
Purification by flash column chromatography (PE:EA, 10:1 v/v) to provide **3ha**. White solid (7 mg, 14% yield);  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.87 (d,  $J = 1.6$  Hz, 1H), 7.56 – 7.48 (m, 3H), 7.38 – 7.22 (m, 5H), 4.30 (s, 2H), 3.68 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  159.3, 154.7, 137.1, 133.3, 132.8, 130.0, 129.9, 129.5, 128.4, 126.6, 123.6, 113.6, 40.8, 29.1.

*1-methyl-3-((methyl(p-tolyl)amino)methyl)quinoxalin-2(1H)-one (3ia)*<sup>8</sup>



Purification by flash column chromatography (PE:EA, 5:1 v/v) to provide **3ia**. Yellow liquid (22 mg, 37% yield);  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.85 (dd,  $J = 8.0, 1.6$  Hz, 1H), 7.57 – 7.53 (m, 1H), 7.37 – 7.29 (m, 2H), 7.04 (d,  $J = 8.3$  Hz, 2H), 6.86 (d,  $J = 8.7$  Hz, 2H), 4.73 (s, 2H), 3.73 (s, 3H), 3.19 (s, 3H), 2.25 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  156.8, 154.8, 147.6, 133.2, 132.6, 130.4, 130.1, 129.5, 125.7, 123.6, 113.5, 112.9, 55.6, 39.7, 28.9, 20.2.

*N-methyl-N-((4-methyl-3-oxo-3,4-dihydroquinoxalin-2-yl)methyl)formamide (3ja)*



Purification by thin-layer chromatography (PE:EA:DCM, 1:2:1 v/v) to provide **3ja**. Yellow solid (15 mg, 33% yield); m.p. 166.1 – 167.3 °C;  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  8.30 (d,  $J = 2.9$  Hz, 1H), 7.92 – 7.83 (m, 1H), 7.65 – 7.54 (m, 1H), 7.43 – 7.31 (m, 2H), 4.74 (d,  $J = 57.9$  Hz, 2H), 3.73 (d,  $J = 6.7$  Hz, 3H), 3.07 (d,  $J = 61.3$  Hz, 3H).  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  164.1, 163.2, 154.3, 154.0, 153.4, 133.3, 133.1, 132.5, 132.4, 130.9, 130.5, 130.3, 124.0, 123.7, 113.8, 113.6, 51.3, 46.4, 35.6, 30.7, 29.0, 28.9. HRMS (ESI)  $m/z$ : calcd for  $\text{C}_{12}\text{H}_{14}\text{N}_3\text{O}_2$  [ $\text{M} + \text{H}$ ] $^+$ , 232.1081; found, 232.1093.

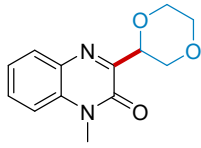
## Reference

1. K.-J. Li, Y.-Y. Jiang, K. Xu, C.-C. Zeng and B.-G. Sun, Electrochemically dehydrogenative C–H/P–H cross-coupling: effective synthesis of phosphonated quinoxalin-2(1H)-ones and xanthenes. *Green Chem.* 2019, **21**, 4412.
2. (a) X.-K. He, J. Lu, A.-J. Zhang, Q.-Q. Zhang, G.-Y. Xu and J. Xuan, BI-OAc-Accelerated C3–H Alkylation of Quinoxalin-2(1H)-ones under Visible-Light Irradiation. *Org. Lett.* 2020, **22**, 5984; (b) X. Zhu, M. Jiang, X. Li, E. Zhu, Q. Deng, X. Song, J. Lv and D. Yang, Alkylsulfonium salts for the photochemical desulphurizative functionalization of heteroarenes. *Org. Chem. Front.* 2022, 10.1039/D1QO01570B.
3. J. Yuan, J. Fu, J. Yin, Z. Dong, Y. Xiao, P. Mao and L. Qu, Transition-metal-free direct C-3 alkylation of quinoxalin-2(1H)-ones with ethers. *Org. Chem. Front.* 2018, **5**, 2820.
4. H. Zhang, J. Xu, M. Zhou, J. Zhao, P. Zhang and W. Li, The visible-light-triggered regioselective alkylation of quinoxalin-2(1H)-ones via decarboxylation coupling. *Org. Biomol. Chem.* 2019, **17**, 10201.
5. L. Liu, N. Pan, W. Sheng, L. Su, L. Liu, J. Dong, Y. Zhou and S.-F. Yin, Visible Light-Induced Regioselective Decarboxylative Alkylation of the C(sp<sup>2</sup>)–H Bonds of Non-Aromatic Heterocycles. *Adv. Synth. Catal.* 2019, **361**, 4126.
6. S. Singh, N. Dagar and S. Raha Roy, Direct functionalization of quinoxalin-2(1H)-one with alkanes: C(sp<sup>2</sup>)–H/C(sp<sup>3</sup>)–H cross coupling in transition metal-free mode. *Org. Biomol. Chem.* 2021, **19**, 5383.
7. W. Wei, L. Wang, H. Yue, P. Bao, W. Liu, C. Hu, D. Yang and H. Wang, Metal-Free Visible-Light-Induced C–H/C–H Cross-Dehydrogenative-Coupling of Quinoxalin-2(H)-ones with Simple Ethers. *ACS Sustainable Chem. Eng.* 2018, **6**, 17252.
8. H.-Y. Zhang, J. Chen, C.-C. Lu, Y.-P. Han, Y. Zhang and J. Zhao, Visible-Light-Induced C(sp<sup>2</sup>)–C(sp<sup>3</sup>) Cross-Dehydrogenative-Coupling Reaction of N-Heterocycles with N-Alkyl-N-methylanilines under Mild Conditions. *J. Org. Chem.* 2021, **86**, 11723.

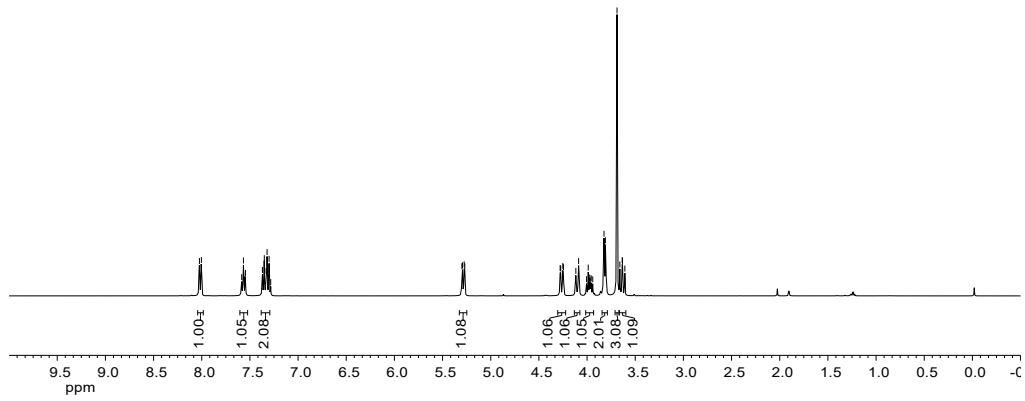
## 3.2 NMR Spectra for Products

8.02  
8.00  
7.59  
7.57  
7.55  
7.37  
7.35  
7.33  
7.32  
7.30  
7.28

5.30  
5.29  
5.28  
5.27  
4.28  
4.27  
4.25  
4.25  
4.12  
4.09  
4.01  
3.99  
3.97  
3.96  
3.94  
3.82  
3.81  
3.69  
3.66  
3.64  
3.63  
3.61



**3aa**,  $^1\text{H}$  NMR, 400 MHz,  $\text{CDCl}_3$



155.08  
153.63

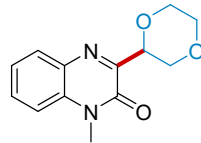
133.06  
132.55  
130.82  
130.67

123.86

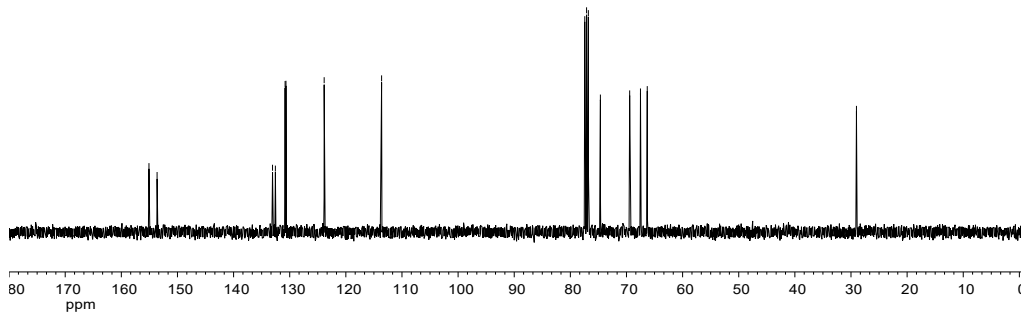
113.63

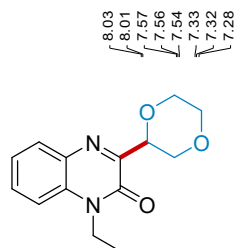
77.43  
77.11  
76.79  
74.65  
69.43  
67.51  
66.30

29.03

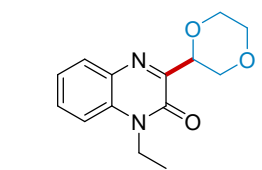
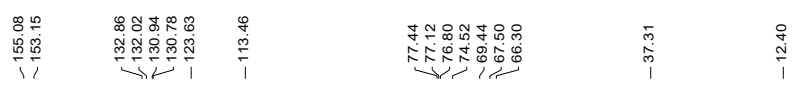
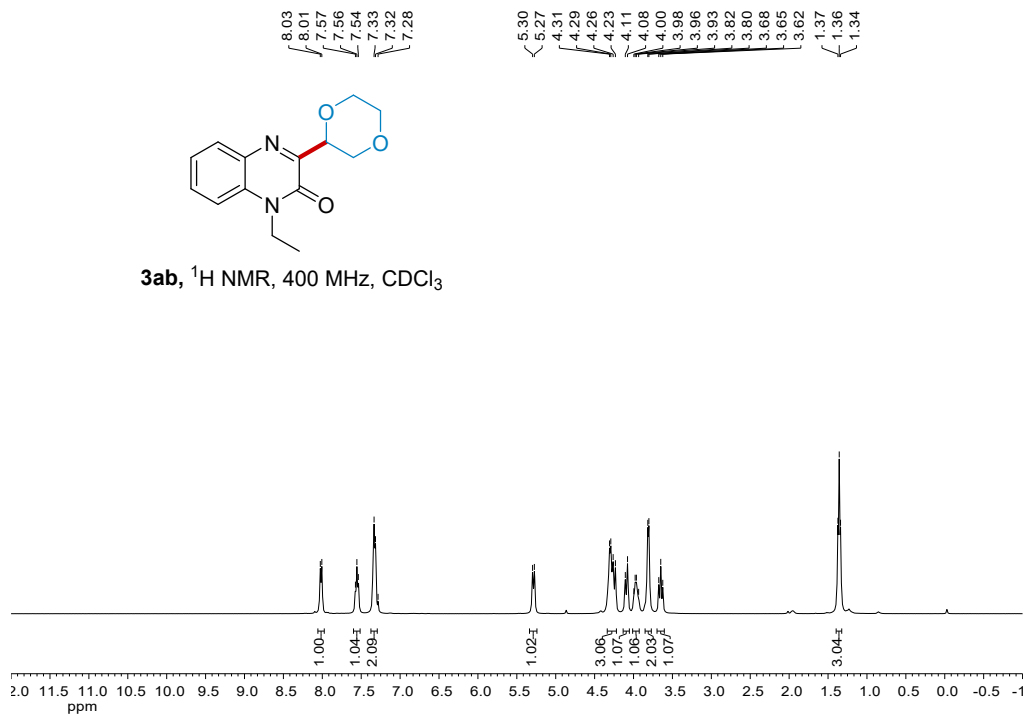


**3aa**,  $^{13}\text{C}$  NMR, 101 MHz,  $\text{CDCl}_3$

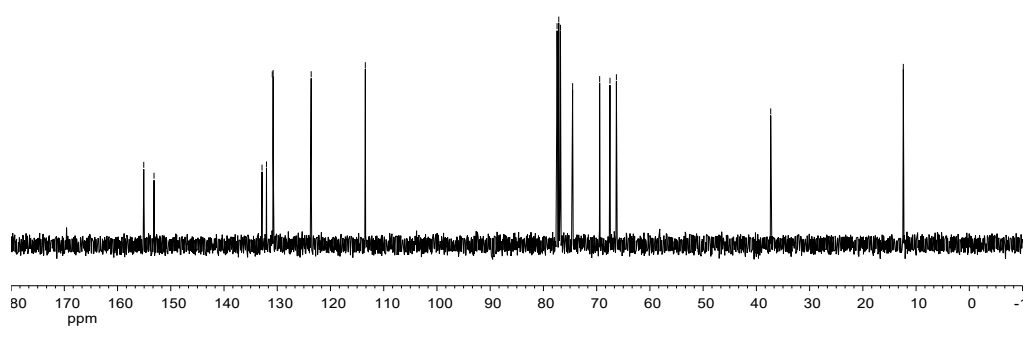




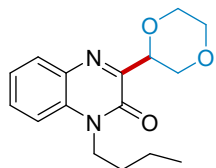
**3ab**,  $^1\text{H NMR}$ , 400 MHz,  $\text{CDCl}_3$



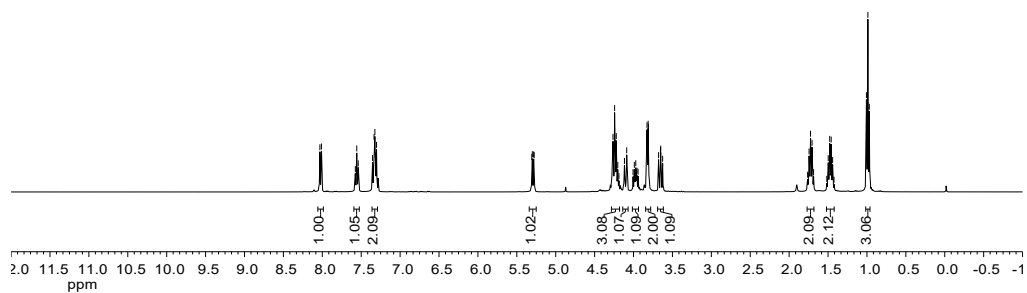
**3ab**,  $^{13}\text{C NMR}$ , 101 MHz,  $\text{CDCl}_3$



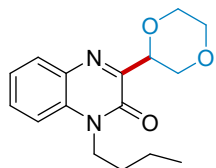
8.03  
8.01  
7.58  
7.56  
7.54  
7.35  
7.33  
7.32  
7.32  
7.30  
5.31  
5.30  
5.28  
5.28  
4.27  
4.26  
4.24  
4.22  
4.20  
4.19  
4.12  
4.09  
4.01  
3.99  
3.97  
3.96  
3.96  
3.94  
3.83  
3.83  
3.81  
3.81  
3.68  
3.65  
3.63  
1.76  
1.74  
1.72  
1.71  
1.69  
1.52  
1.50  
1.48  
1.46  
1.44  
1.42  
1.01  
0.99  
0.97



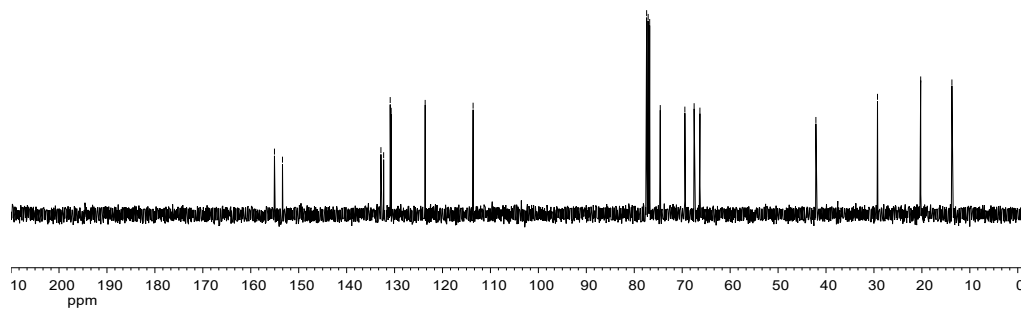
**3ac**,  $^1\text{H NMR}$ , 400 MHz,  $\text{CDCl}_3$

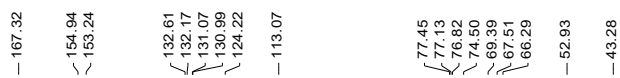
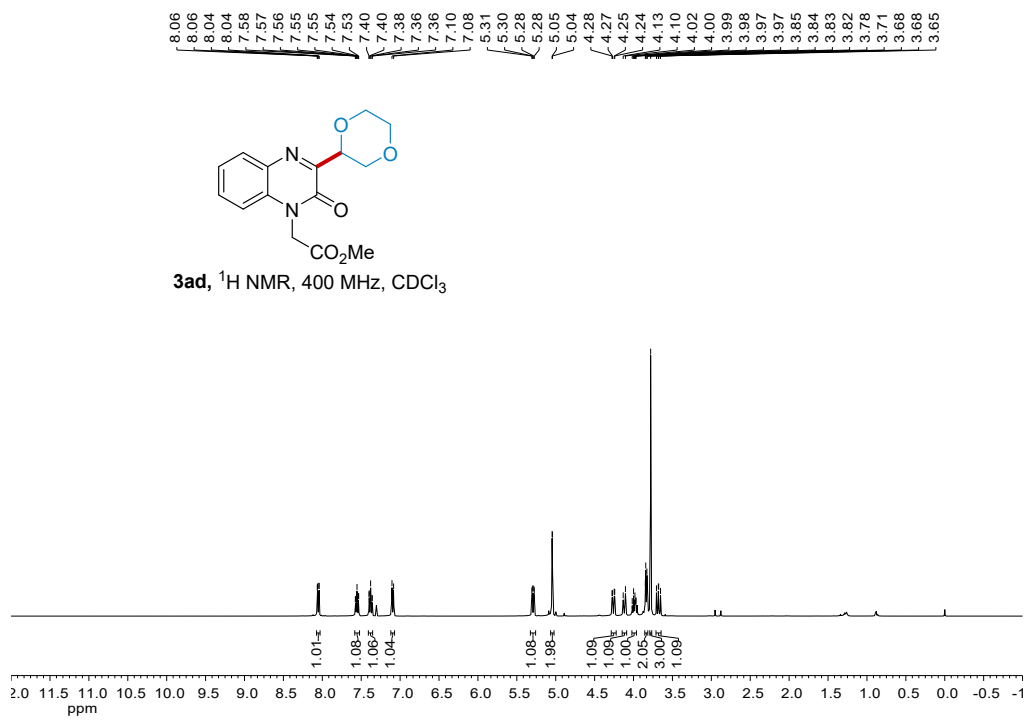


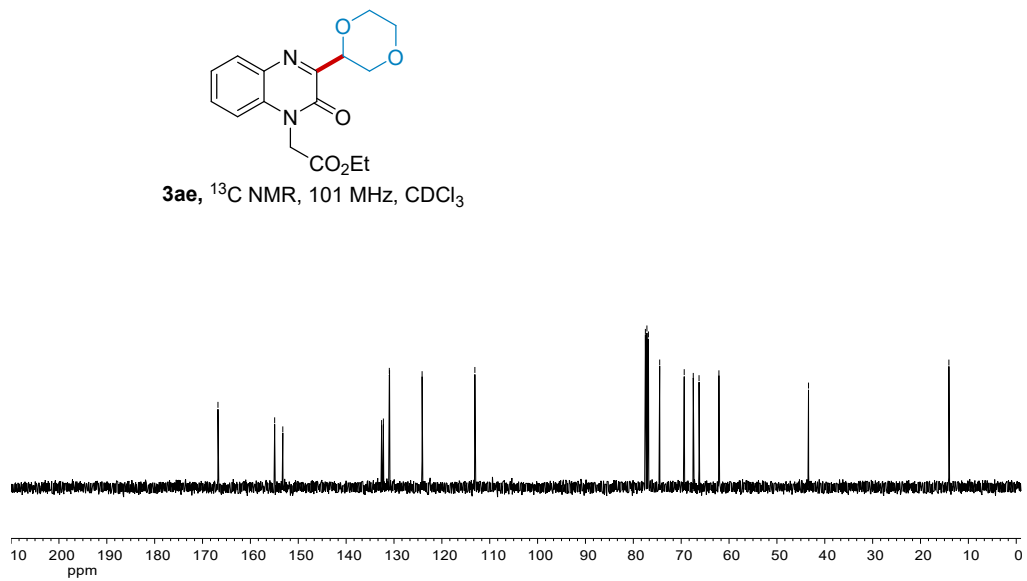
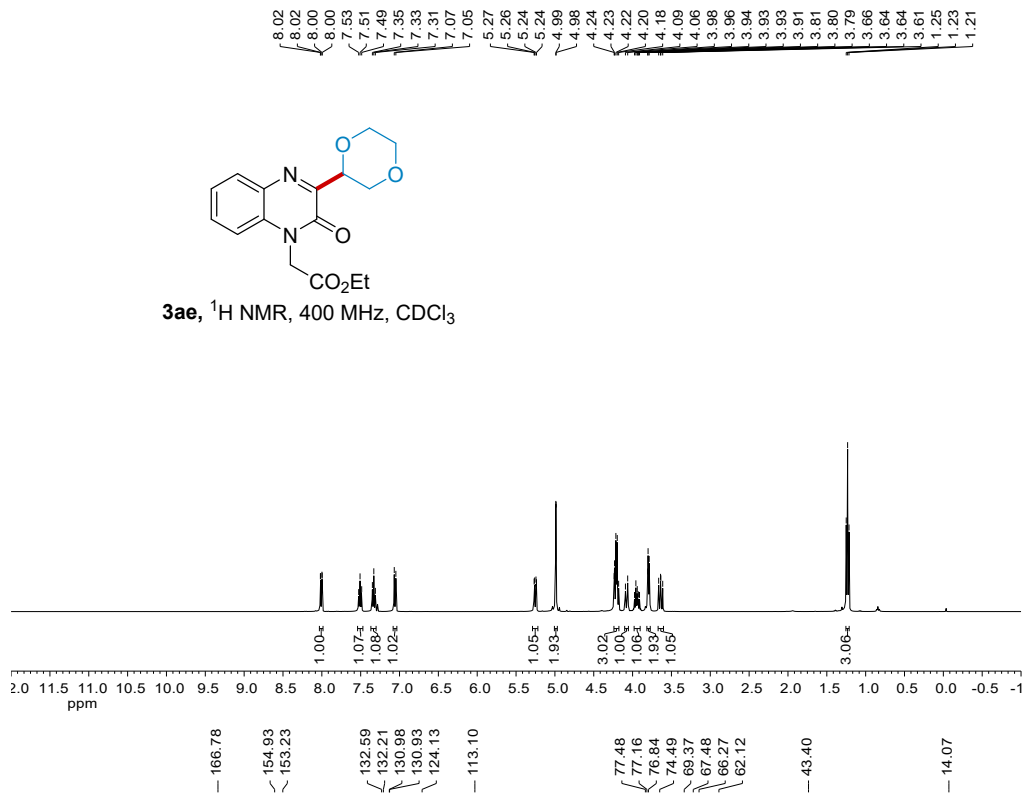
155.05  
153.38  
132.85  
132.28  
130.91  
130.71  
123.62  
113.62  
77.42  
77.10  
76.78  
74.58  
69.44  
67.51  
66.30  
42.11  
29.26  
20.25  
13.74

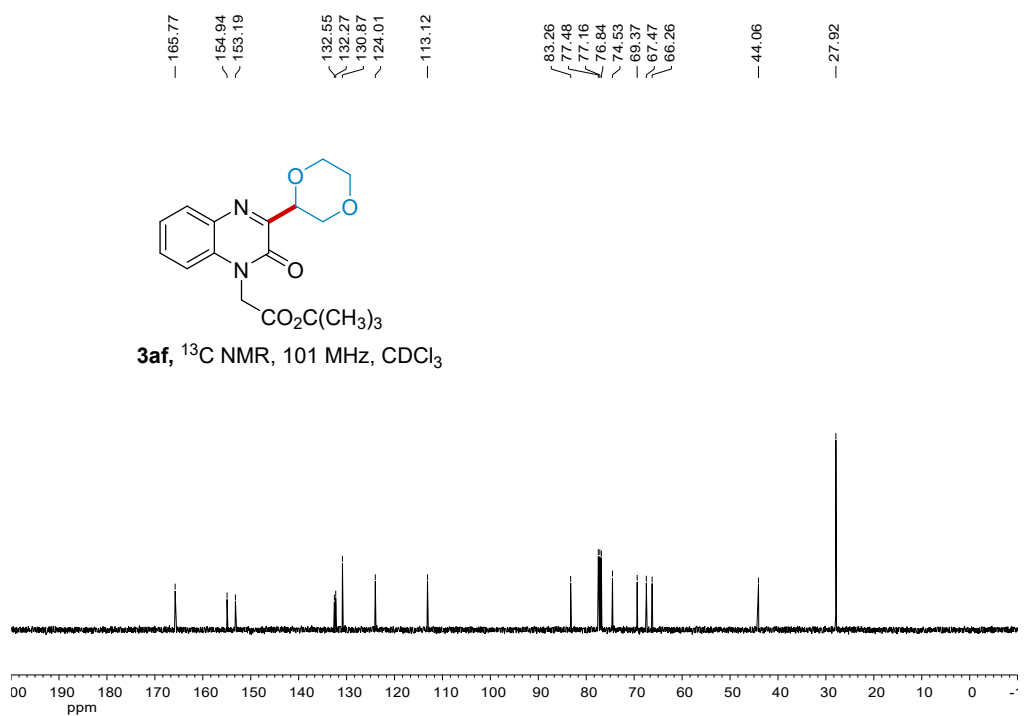
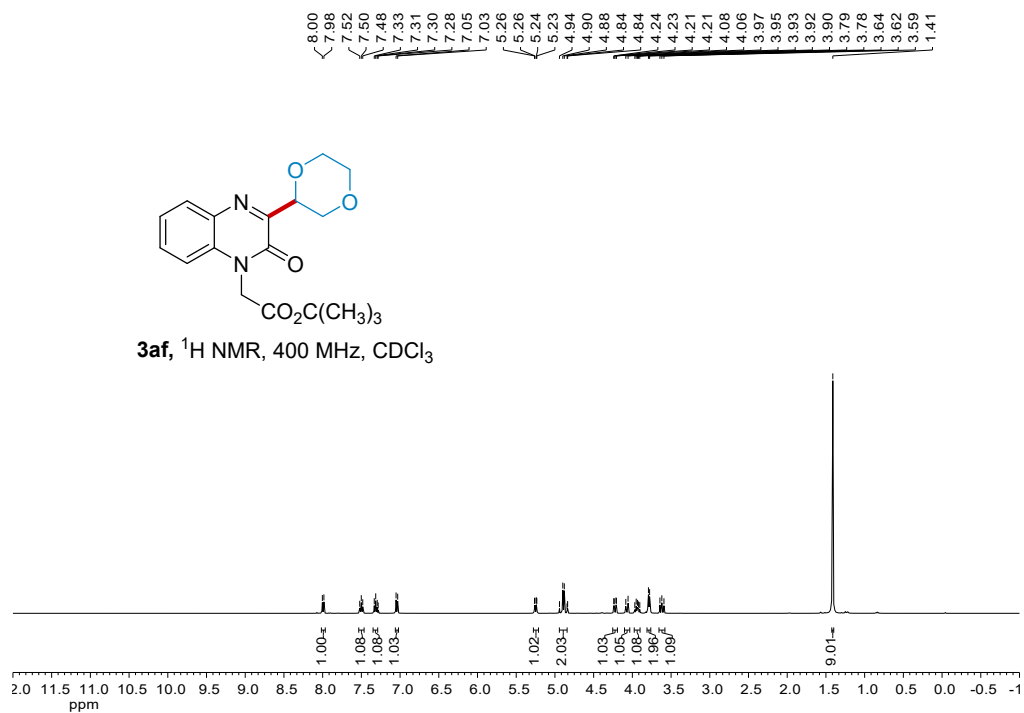


**3ac**,  $^{13}\text{C NMR}$ , 101 MHz,  $\text{CDCl}_3$



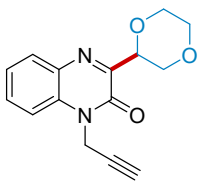




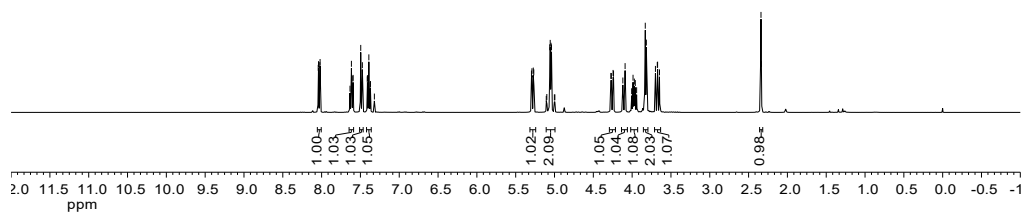




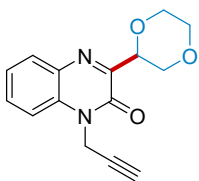
8.04  
8.04  
8.02  
8.02  
7.64  
7.63  
7.62  
7.60  
7.59  
7.50  
7.48  
7.41  
7.41  
7.39  
7.37  
7.37  
7.32  
5.30  
5.29  
5.27  
5.27  
5.11  
5.11  
5.10  
5.06  
5.06  
5.05  
5.04  
5.04  
5.00  
5.00  
4.27  
4.27  
4.25  
4.25  
4.24  
4.24  
4.12  
4.12  
4.09  
4.01  
3.99  
3.98  
3.97  
3.96  
3.94  
3.83  
3.82  
3.70  
3.68  
3.67  
3.65  
2.35  
2.34  
2.33



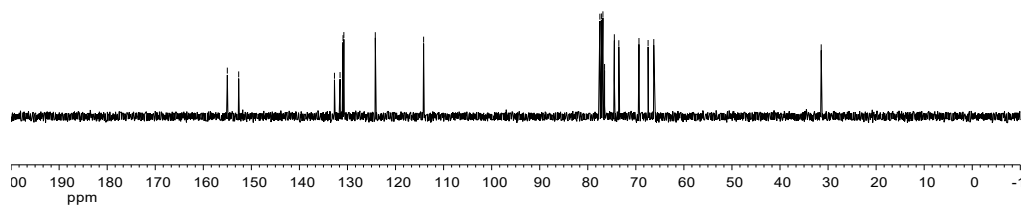
**3ag**, <sup>1</sup>H NMR, 400 MHz, CDCl<sub>3</sub>



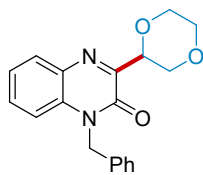
155.00  
152.64  
132.71  
131.55  
130.93  
130.76  
124.21  
114.16  
77.49  
77.17  
76.85  
76.51  
74.47  
73.54  
69.35  
67.45  
66.27  
31.43



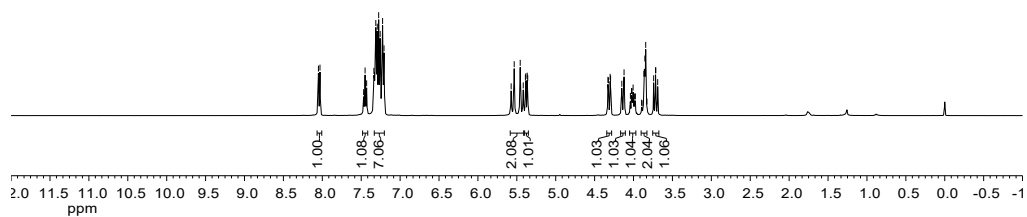
**3ag**, <sup>13</sup>C NMR, 101 MHz, CDCl<sub>3</sub>



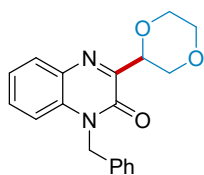
8.05, 8.03, 7.47, 7.45, 7.43, 7.34, 7.33, 7.31, 7.29, 7.28, 7.26, 7.23, 7.21, 5.57, 5.53, 5.46, 5.42, 5.39, 5.38, 5.37, 5.36, 4.33, 4.32, 4.30, 4.30, 4.15, 4.12, 4.04, 4.03, 4.02, 4.01, 4.00, 3.99, 3.98, 3.90, 3.89, 3.87, 3.85, 3.84, 3.83, 3.74, 3.72, 3.71, 3.69



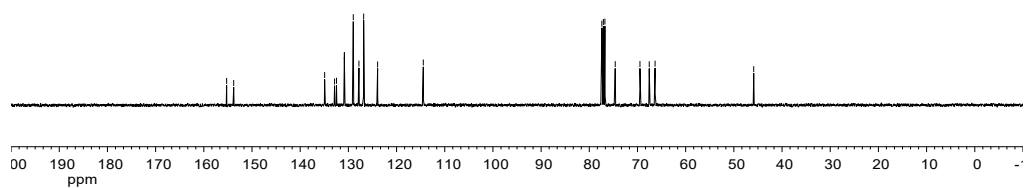
**3ah**,  $^1\text{H}$  NMR, 400 MHz,  $\text{CDCl}_3$

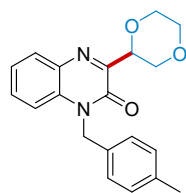


155.27, 153.80, 134.93, 132.87, 132.46, 130.84, 130.82, 128.98, 127.81, 126.83, 123.93, 114.45, 77.40, 77.08, 76.76, 74.62, 69.49, 67.56, 66.35, 45.88

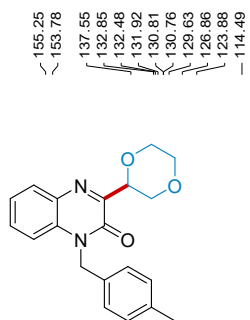
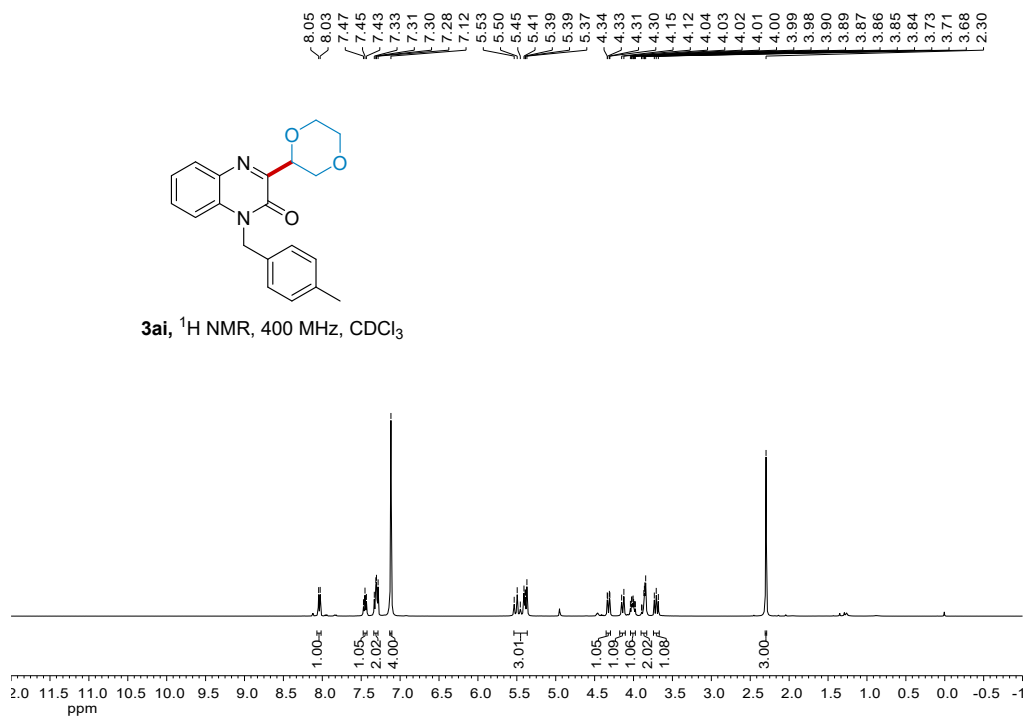


**3ah**,  $^{13}\text{C}$  NMR, 101 MHz,  $\text{CDCl}_3$

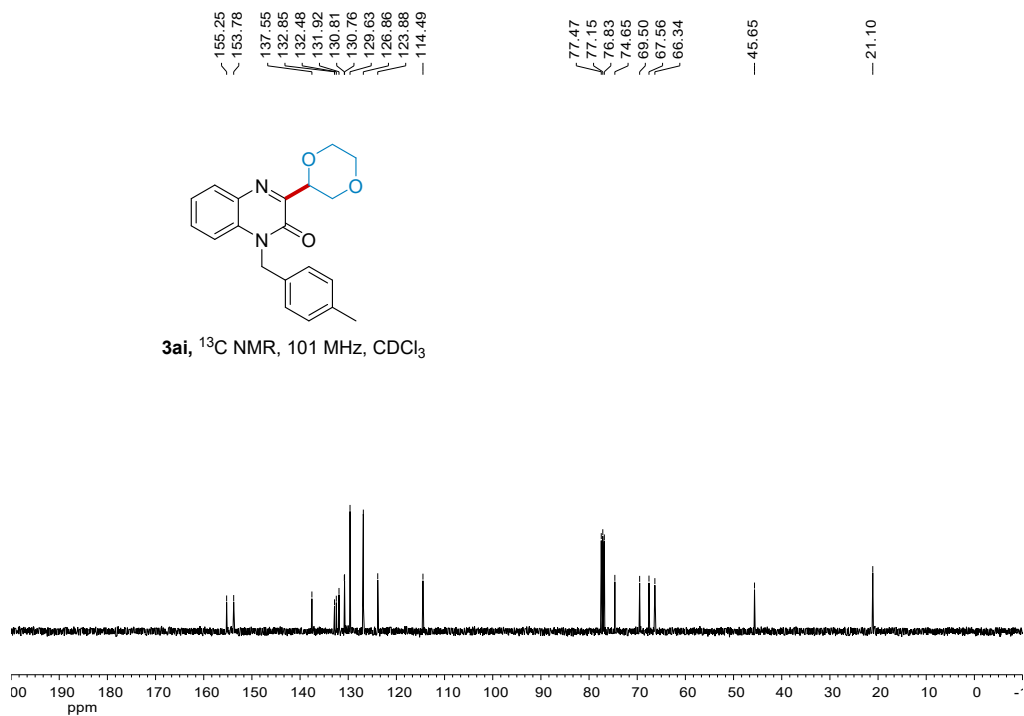




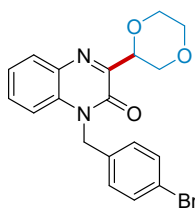
**3ai**,  $^1\text{H NMR}$ , 400 MHz,  $\text{CDCl}_3$



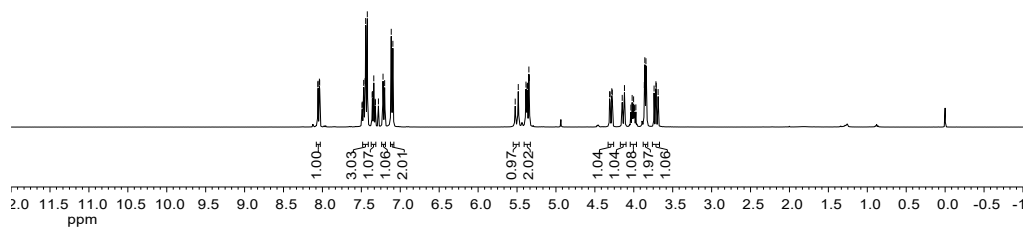
**3ai**,  $^{13}\text{C NMR}$ , 101 MHz,  $\text{CDCl}_3$



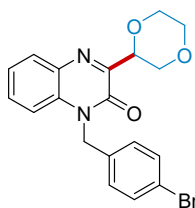
8.06  
8.06  
8.04  
8.04  
7.49  
7.49  
7.47  
7.45  
7.44  
7.42  
7.36  
7.36  
7.34  
7.34  
7.32  
7.32  
7.28  
7.28  
7.22  
7.20  
7.12  
7.09  
5.52  
5.48  
5.38  
5.37  
5.37  
5.35  
5.35  
5.34  
4.31  
4.30  
4.28  
4.28  
4.15  
4.15  
4.12  
4.12  
4.02  
4.02  
4.01  
4.00  
3.99  
3.99  
3.97  
3.86  
3.86  
3.84  
3.84  
3.74  
3.72  
3.71  
3.69



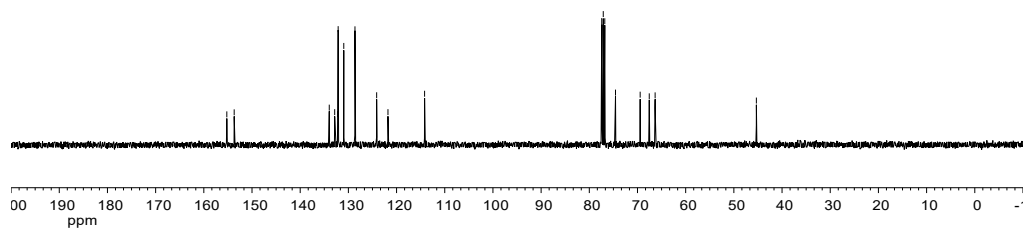
3aj, <sup>1</sup>H NMR, 400 MHz, CDCl<sub>3</sub>



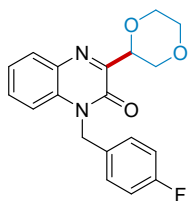
155.23  
153.70  
133.97  
132.84  
132.22  
132.12  
130.96  
128.63  
124.13  
121.78  
114.18  
77.42  
77.10  
76.79  
74.56  
69.45  
67.56  
66.34  
45.33



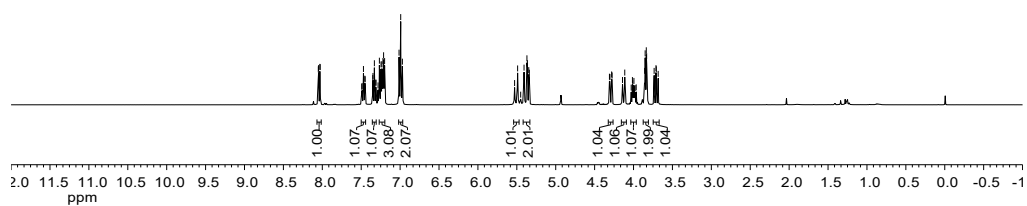
3aj, <sup>13</sup>C NMR, 101 MHz, CDCl<sub>3</sub>



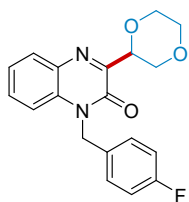
8.05  
8.05  
8.03  
8.03  
7.49  
7.49  
7.47  
7.45  
7.45  
7.35  
7.35  
7.33  
7.33  
7.31  
7.31  
7.28  
7.27  
7.25  
7.23  
7.22  
7.21  
7.20  
7.01  
7.01  
6.99  
6.97  
5.53  
5.49  
5.45  
5.41  
5.37  
5.36  
5.35  
5.34  
4.31  
4.30  
4.28  
4.28  
4.14  
4.11  
4.03  
4.01  
4.01  
4.00  
3.99  
3.99  
3.98  
3.97  
3.86  
3.85  
3.84  
3.83  
3.74  
3.71  
3.68



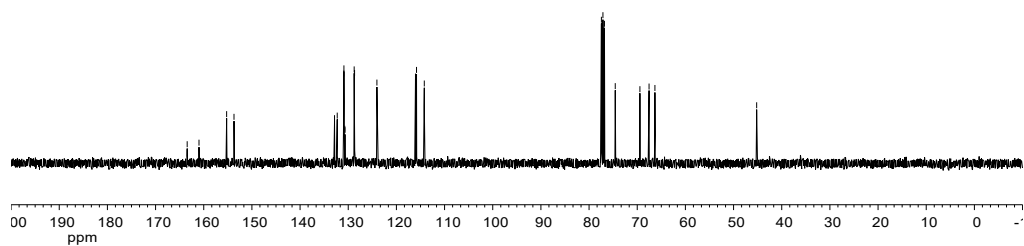
**3ak**,  $^1\text{H}$  NMR, 400 MHz,  $\text{CDCl}_3$



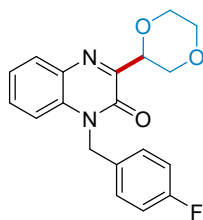
163.44  
160.99  
155.25  
153.72  
132.85  
132.29  
130.91  
130.90  
130.71  
130.68  
128.77  
128.69  
124.05  
116.04  
115.82  
114.21  
77.44  
77.12  
76.80  
74.57  
69.44  
67.55  
66.33  
-45.20



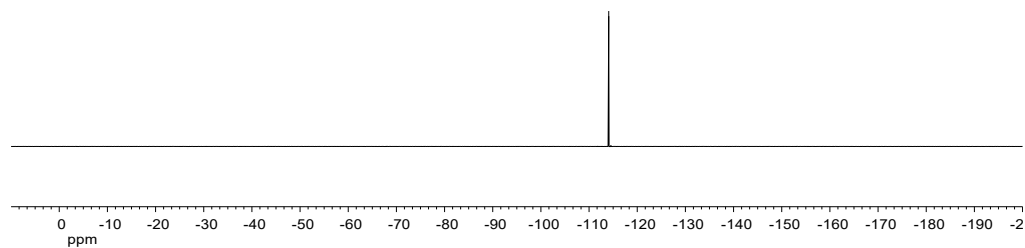
**3ak**,  $^{13}\text{C}$  NMR, 101 MHz,  $\text{CDCl}_3$



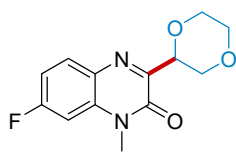
--114.08



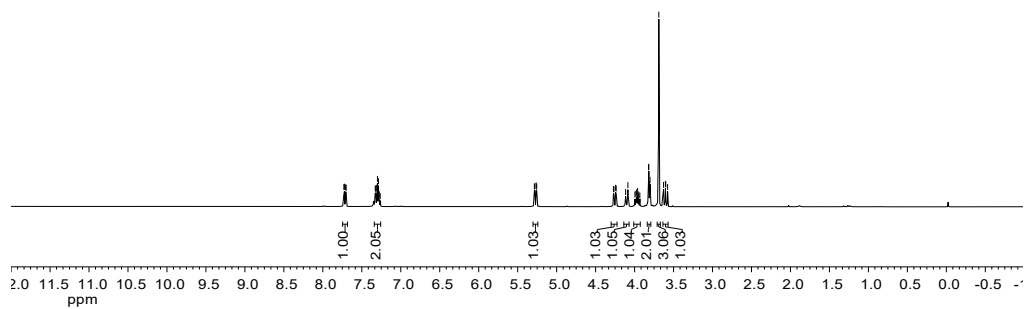
**3ak**,  $^{19}\text{F}$  NMR, 376 MHz,  $\text{CDCl}_3$



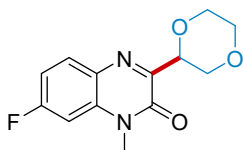
7.73  
7.72  
7.71  
7.70  
7.33  
7.32  
7.31  
7.30  
7.29  
7.28  
7.26  
5.29  
5.28  
5.26  
5.26  
4.27  
4.26  
4.24  
4.24  
4.11  
4.09  
4.00  
3.98  
3.97  
3.97  
3.96  
3.95  
3.95  
3.93  
3.82  
3.82  
3.81  
3.80  
3.80  
3.69  
3.63  
3.60  
3.60  
3.58



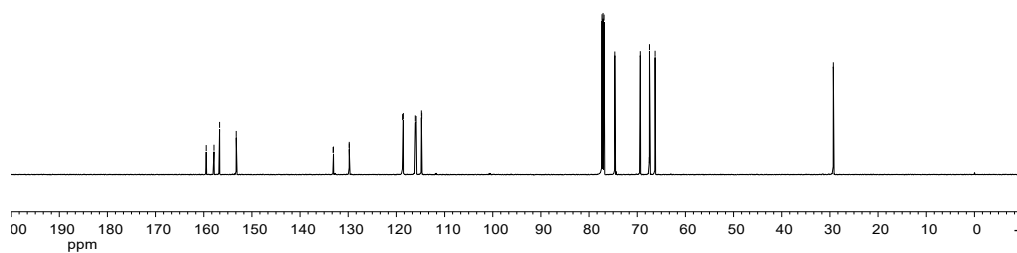
**3al**,  $^1\text{H}$  NMR, 400 MHz,  $\text{CDCl}_3$



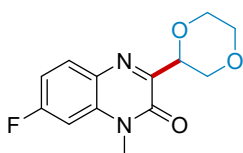
<sup>13</sup>C NMR peaks (ppm):  
 159.49, 157.88, 156.71, 153.26, 133.13, 133.06, 129.79, 129.77, 118.71, 118.55, 116.08, 115.93, 114.83, 114.77, 77.31, 77.10, 76.89, 74.65, 69.36, 67.50, 66.30, -29.29



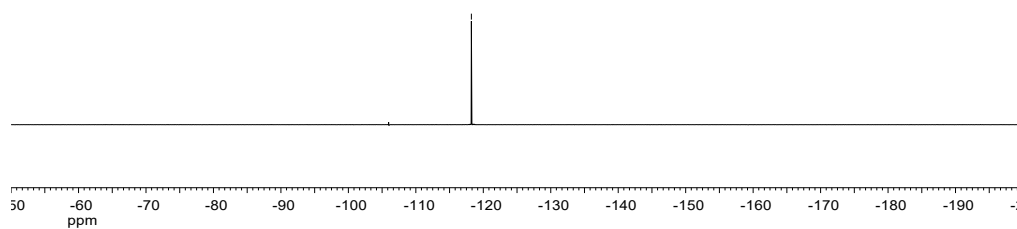
**3al**, <sup>13</sup>C NMR, 151 MHz, CDCl<sub>3</sub>

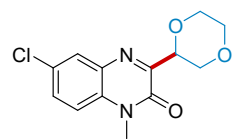


-118.23

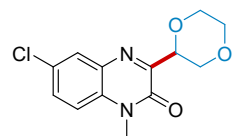
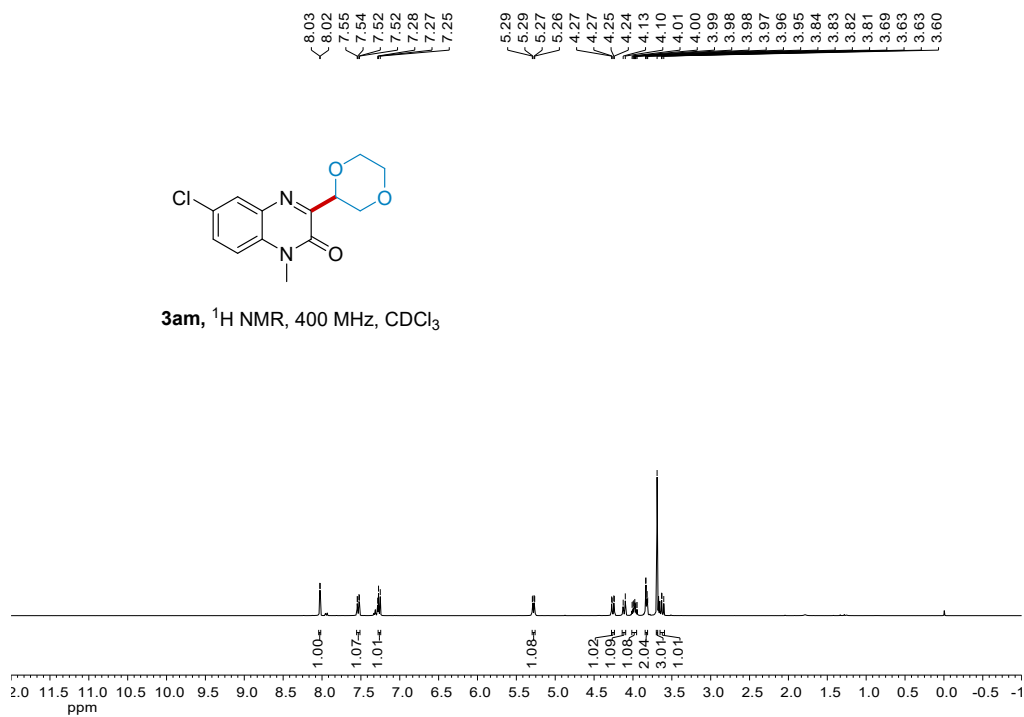


**3al**, <sup>19</sup>F NMR, 376 MHz, CDCl<sub>3</sub>

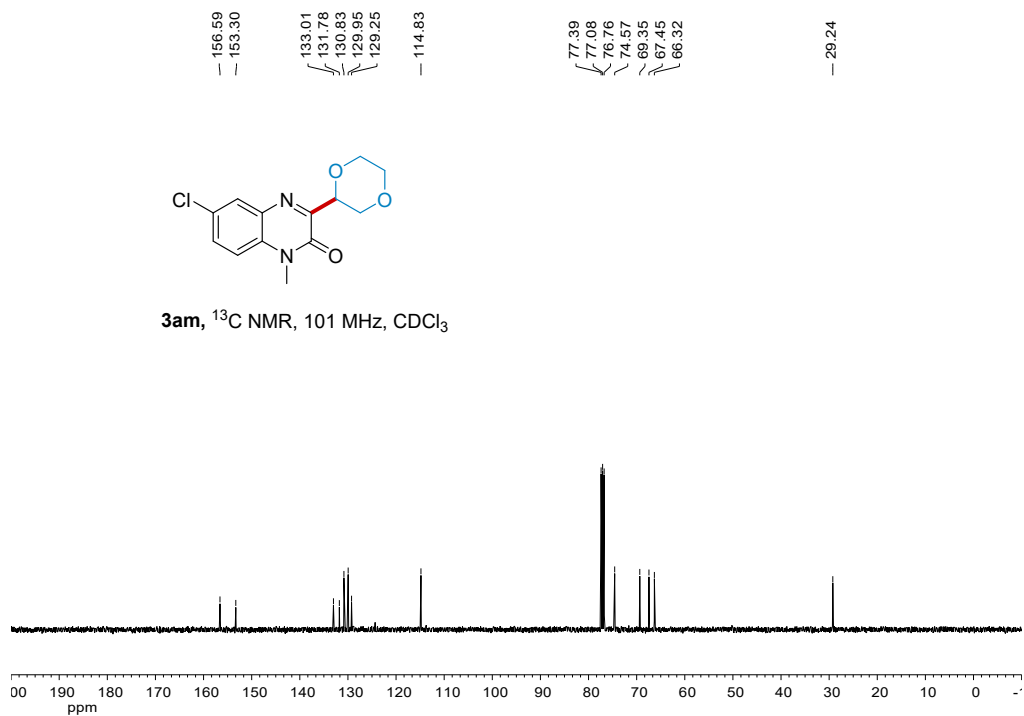




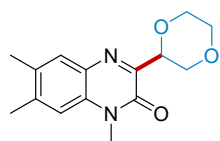
**3am**,  $^1\text{H}$  NMR, 400 MHz,  $\text{CDCl}_3$



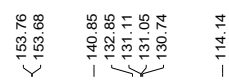
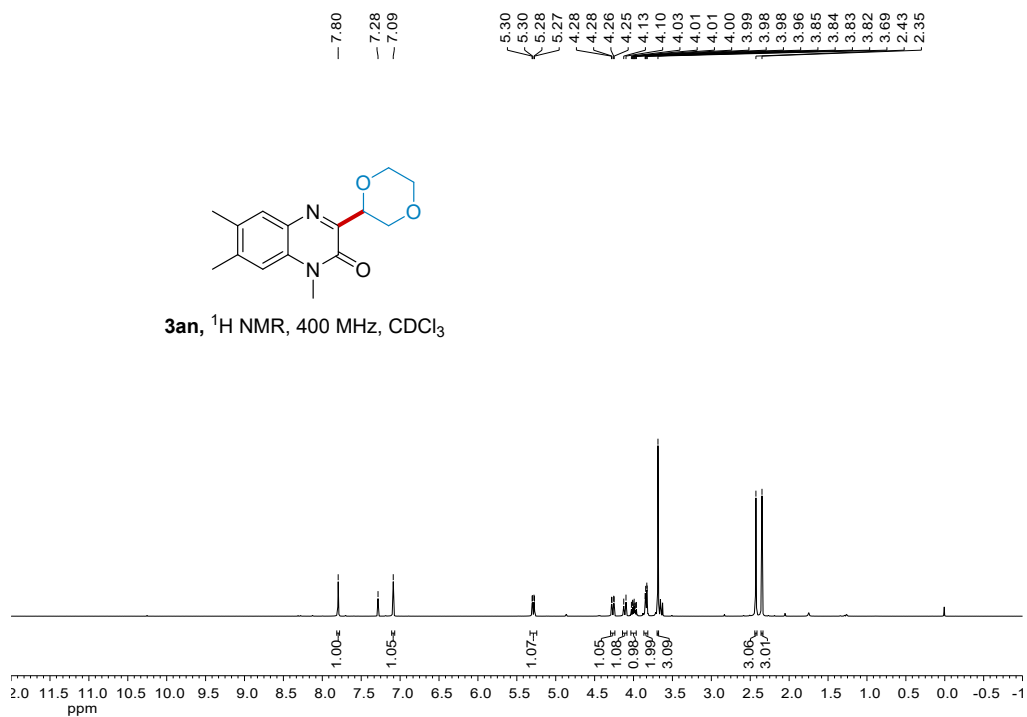
**3am**,  $^{13}\text{C}$  NMR, 101 MHz,  $\text{CDCl}_3$



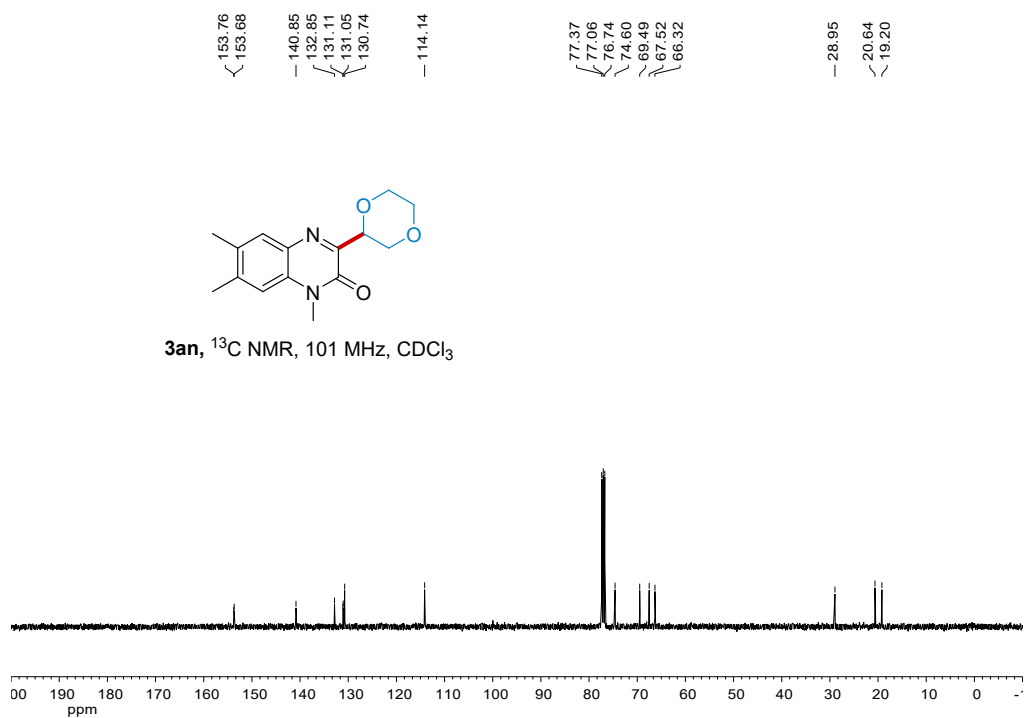


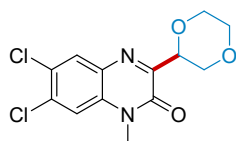


**3an**,  $^1\text{H}$  NMR, 400 MHz,  $\text{CDCl}_3$

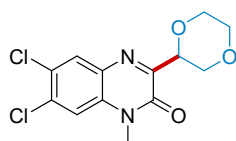
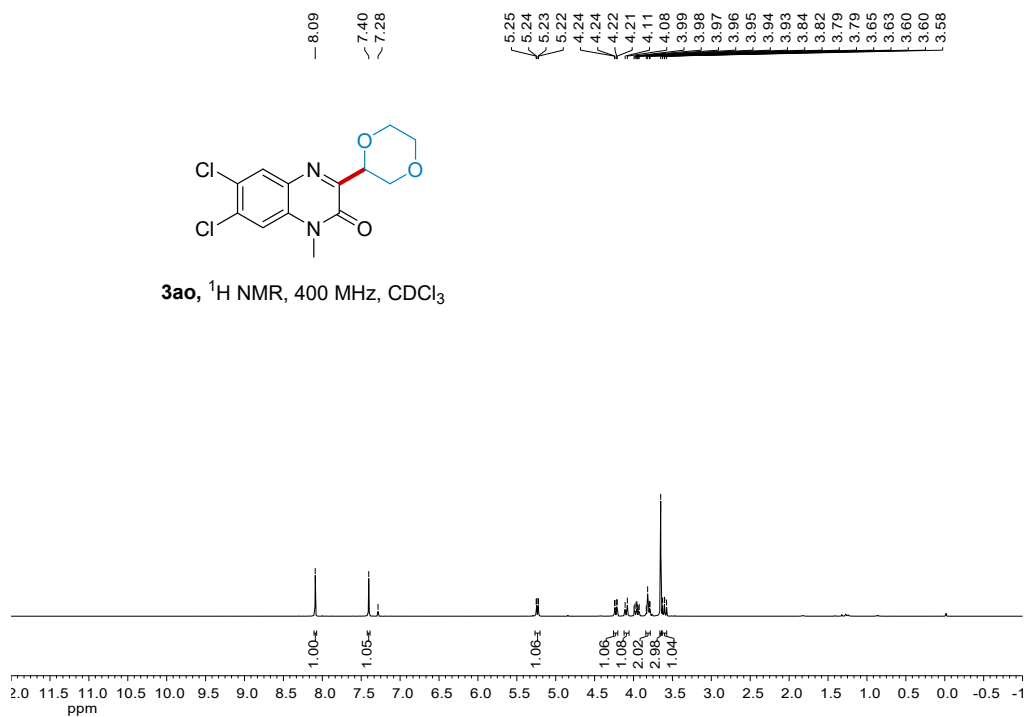


**3an**,  $^{13}\text{C}$  NMR, 101 MHz,  $\text{CDCl}_3$

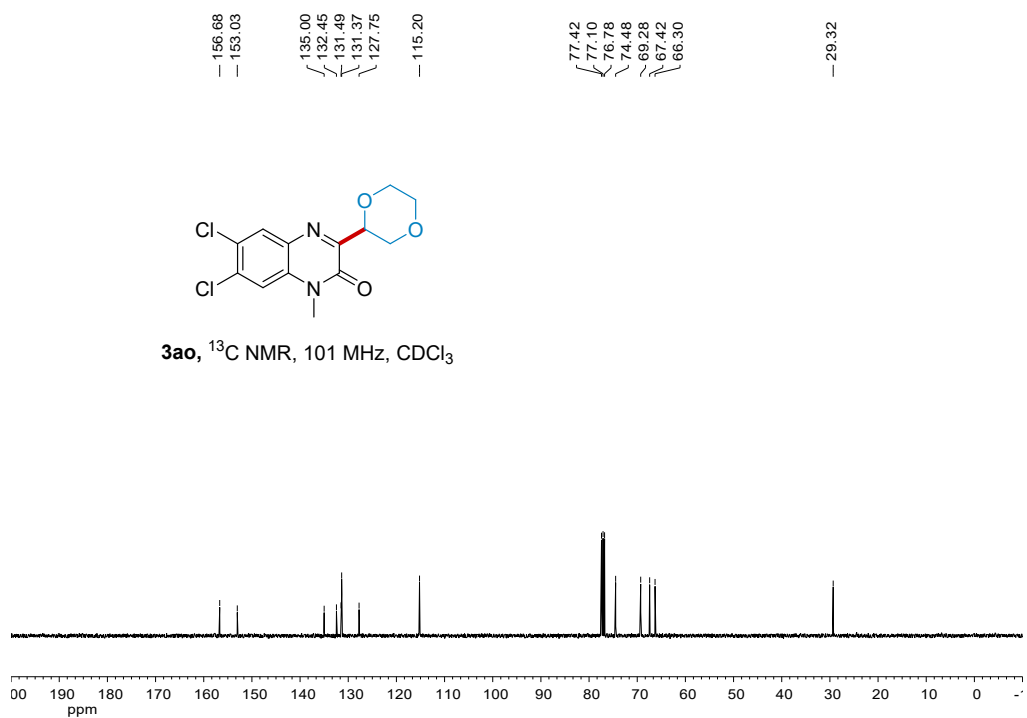




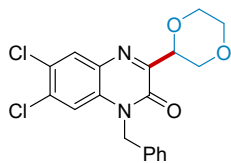
**3ao**,  $^1\text{H}$  NMR, 400 MHz,  $\text{CDCl}_3$



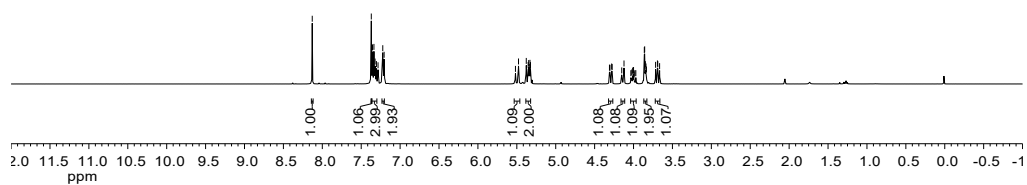
**3ao**,  $^{13}\text{C}$  NMR, 101 MHz,  $\text{CDCl}_3$



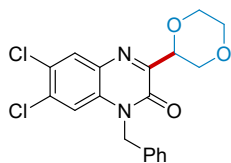
8.13  
7.37  
7.35  
7.35  
7.34  
7.32  
7.31  
7.30  
7.28  
7.21  
7.21  
5.52  
5.48  
5.38  
5.35  
5.35  
5.34  
5.34  
5.33  
5.32  
4.31  
4.30  
4.28  
4.27  
4.15  
4.12  
4.03  
4.02  
4.01  
4.01  
4.00  
3.99  
3.98  
3.97  
3.86  
3.86  
3.85  
3.84  
3.83  
3.72  
3.69  
3.66



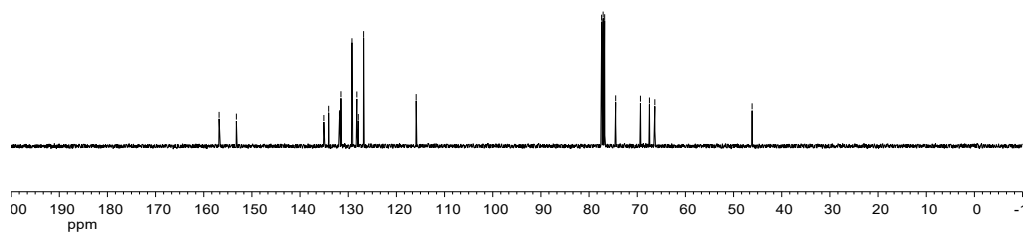
**3ap**,  $^1\text{H}$  NMR, 400 MHz,  $\text{CDCl}_3$

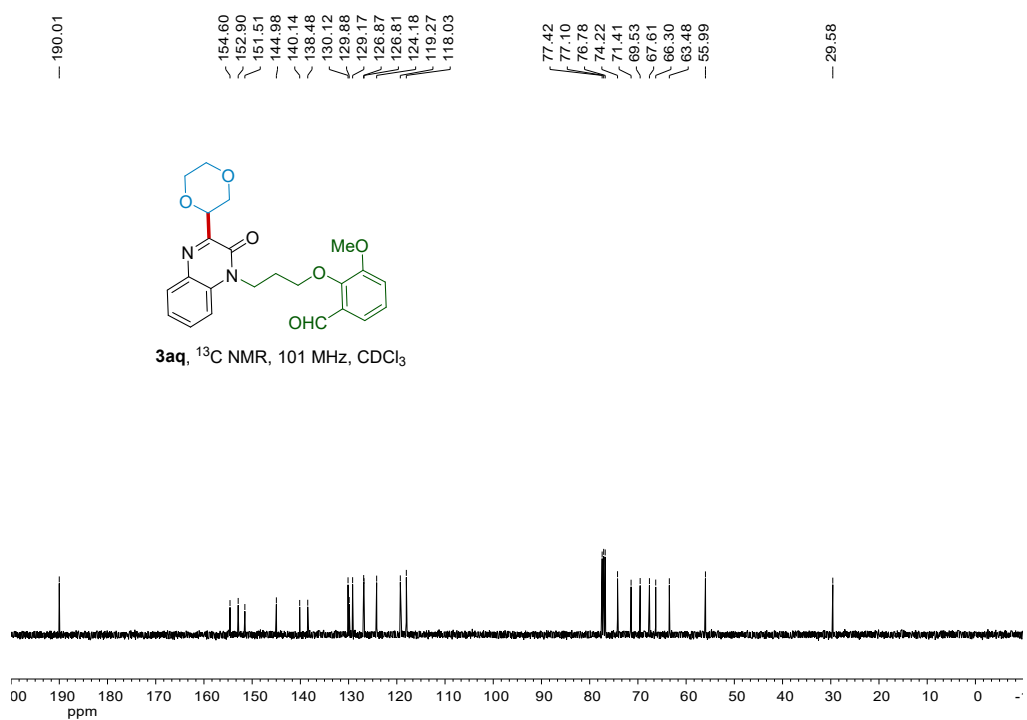
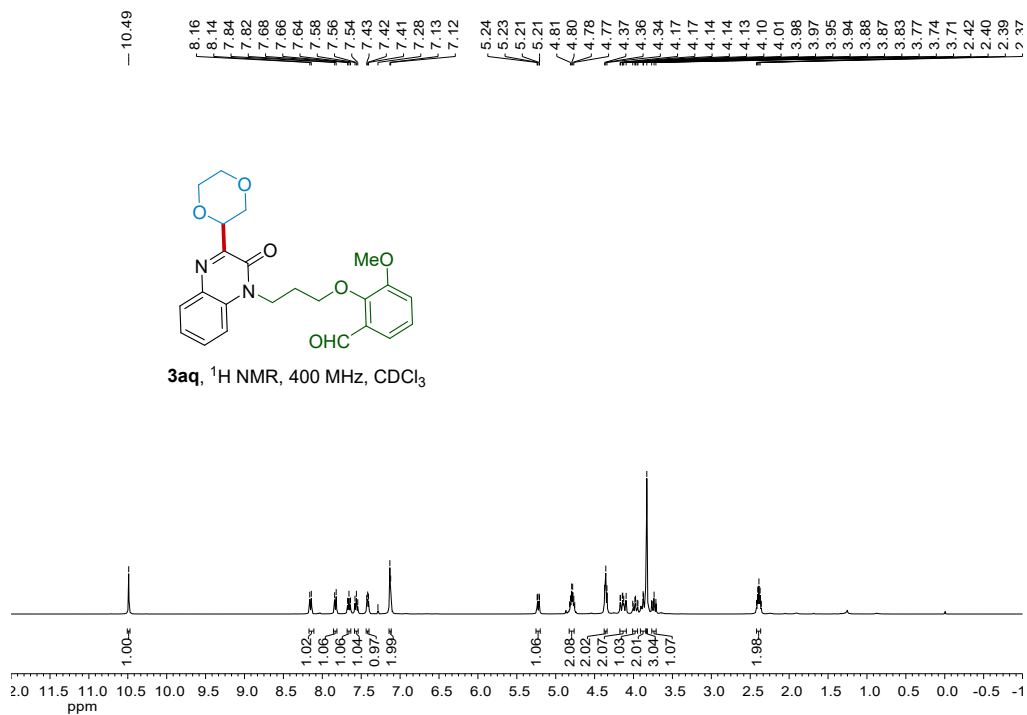


156.82  
153.22  
135.06  
134.06  
131.82  
131.80  
131.51  
129.23  
128.22  
127.92  
126.81  
115.89  
77.40  
77.08  
76.76  
74.47  
69.33  
67.47  
66.35  
46.15

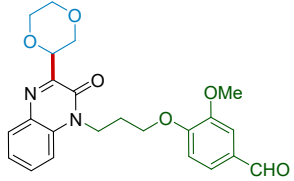


**3ap**,  $^{13}\text{C}$  NMR, 101 MHz,  $\text{CDCl}_3$

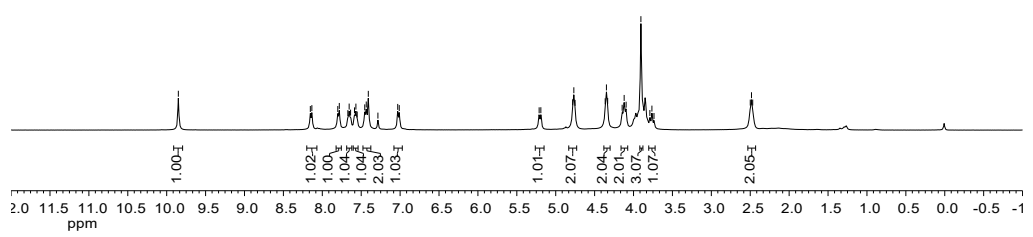




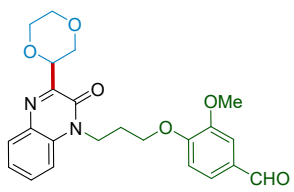
9.85, 8.15, 8.13, 7.80, 7.78, 7.67, 7.65, 7.64, 7.59, 7.57, 7.55, 7.45, 7.44, 7.41, 7.29, 7.03, 7.01, 5.21, 5.19, 4.78, 4.77, 4.75, 4.36, 4.35, 4.33, 4.15, 4.12, 4.10, 3.91, 3.79, 3.76, 3.74, 2.50, 2.48, 2.47



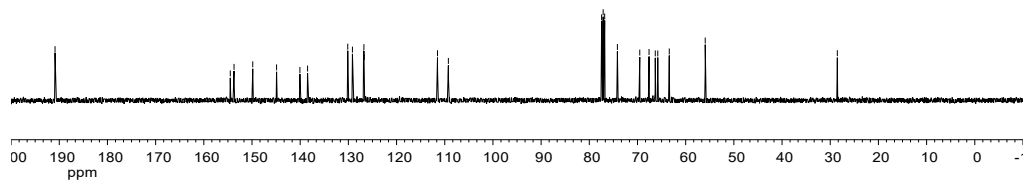
**3ar**,  $^1\text{H}$  NMR, 400 MHz,  $\text{CDCl}_3$



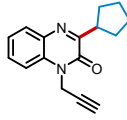
190.88, 154.51, 153.73, 149.85, 144.90, 140.06, 138.47, 130.17, 130.13, 129.16, 126.86, 126.78, 126.70, 111.50, 109.25, 77.44, 77.13, 76.81, 74.17, 69.53, 67.60, 66.31, 65.79, 63.41, 55.94, 28.53



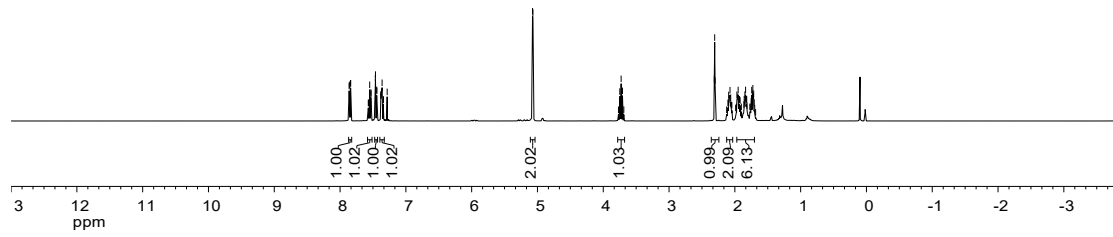
**3ar**,  $^{13}\text{C}$  NMR, 101 MHz,  $\text{CDCl}_3$



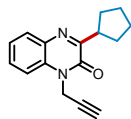
7.86  
7.86  
7.84  
7.84  
7.57  
7.57  
7.55  
7.55  
7.55  
7.53  
7.53  
7.46  
7.46  
7.44  
7.44  
7.44  
7.38  
7.38  
7.36  
7.36  
7.34  
7.34  
7.28  
5.07  
5.07  
3.75  
3.75  
3.73  
3.73  
3.71  
3.71  
2.31  
2.31  
2.30  
2.30  
2.11  
2.11  
2.09  
2.09  
2.07  
2.07  
2.06  
2.06  
2.05  
2.05  
2.05  
1.97  
1.96  
1.96  
1.95  
1.94  
1.94  
1.93  
1.93  
1.92  
1.92  
1.90  
1.90  
1.86  
1.86  
1.85  
1.85  
1.84  
1.84  
1.82  
1.82  
1.81  
1.81  
1.77  
1.77  
1.75  
1.75  
1.74  
1.74  
1.73  
1.73  
1.71  
1.71



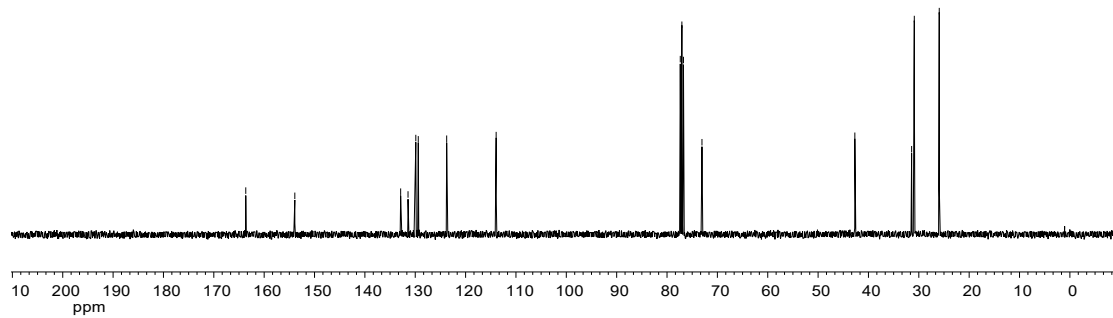
**3bb**,  $^1\text{H NMR}$ , 400 MHz,  $\text{CDCl}_3$

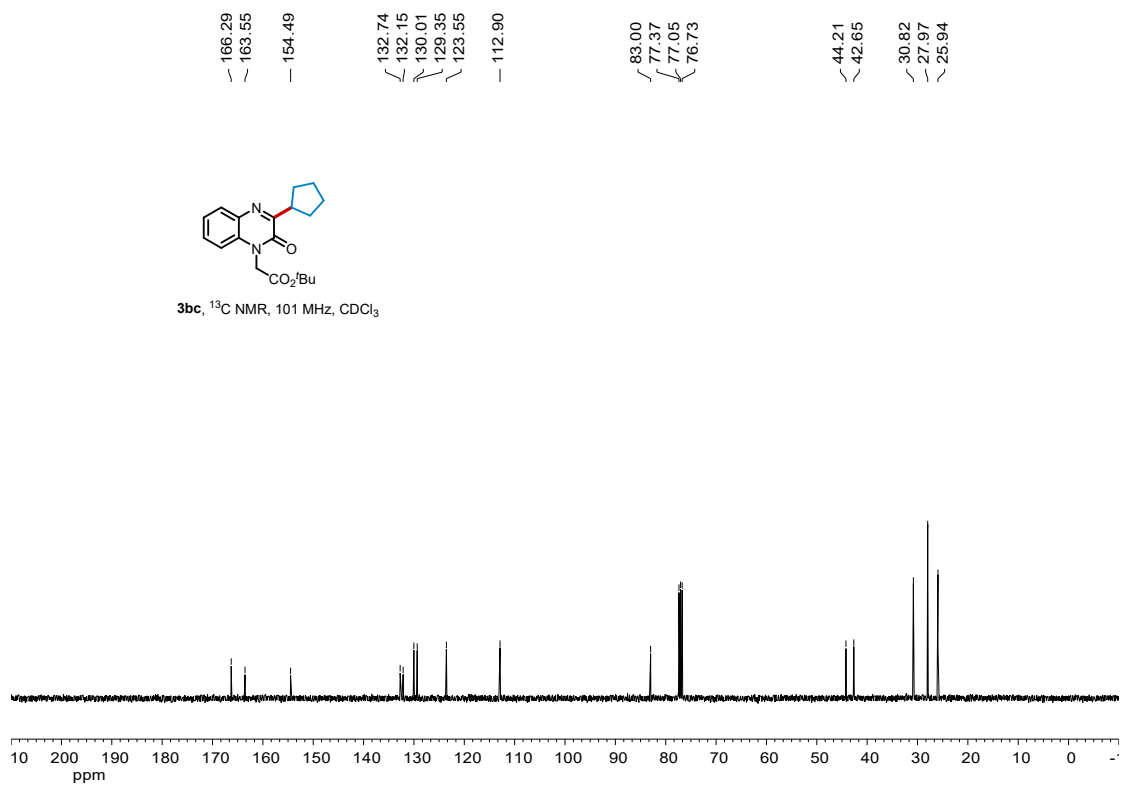
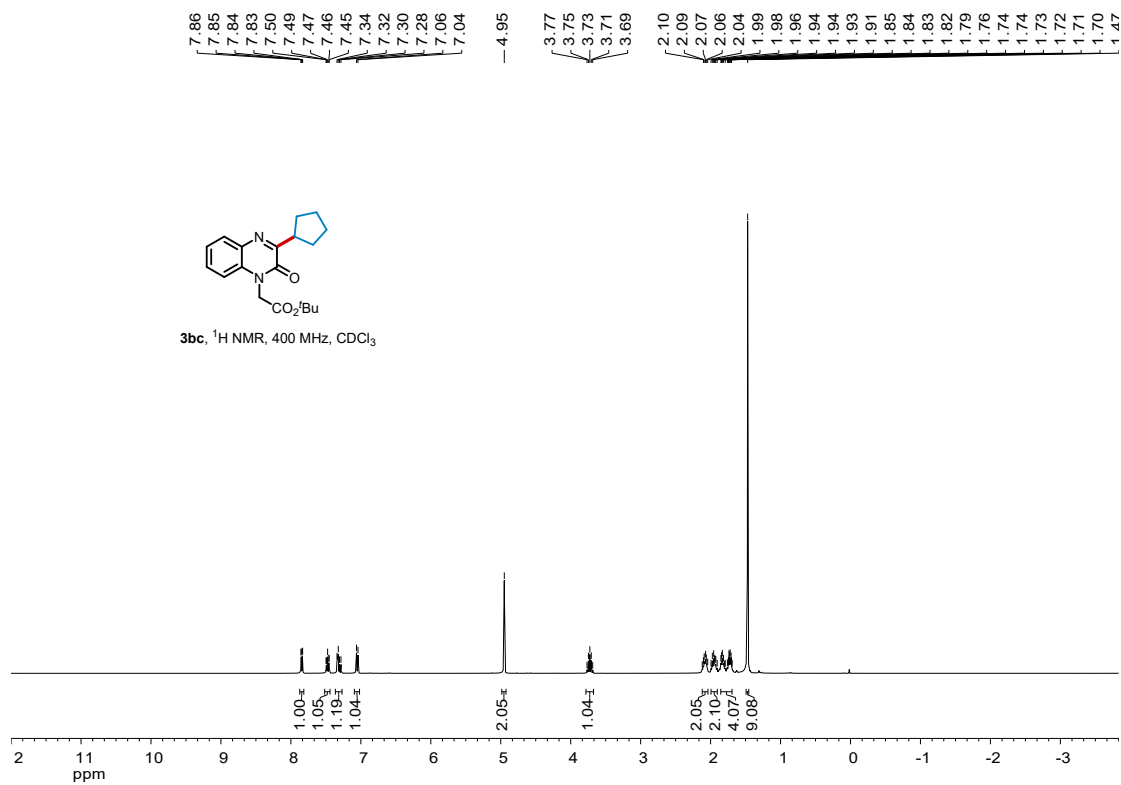


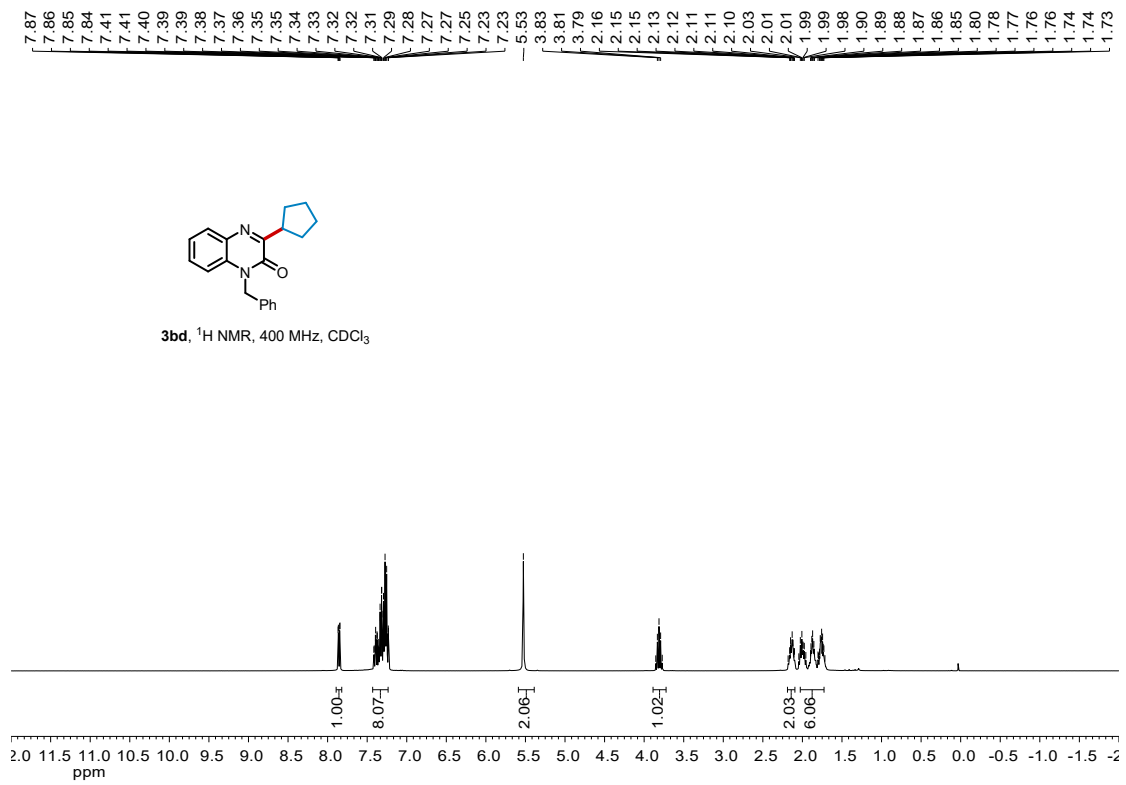
163.65  
153.91  
132.90  
131.43  
129.89  
129.40  
123.76  
113.93  
77.37  
77.05  
76.73  
73.06  
42.70  
31.43  
30.90  
25.94



**3bb**,  $^{13}\text{C NMR}$ , 101 MHz,  $\text{CDCl}_3$

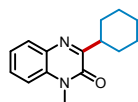




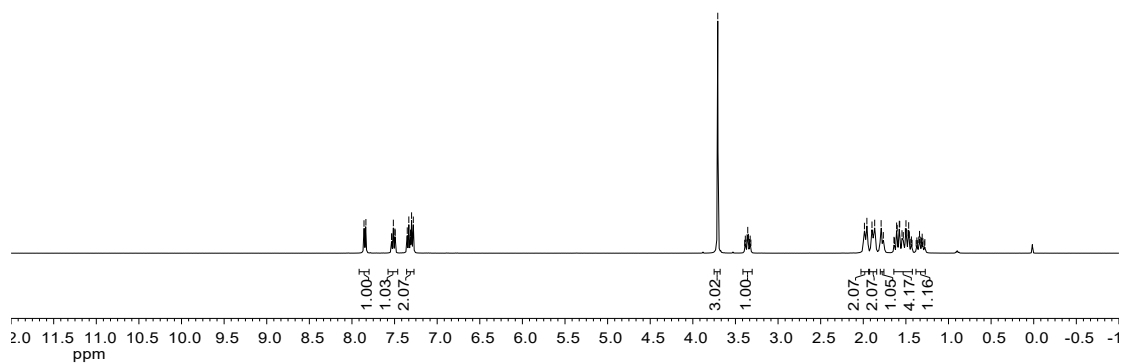




7.86  
7.84  
7.54  
7.53  
7.51  
7.50  
7.49  
7.35  
7.33  
7.32  
7.31  
7.30  
7.28  
3.71  
3.39  
3.38  
3.37  
3.36  
3.36  
3.35  
3.34  
3.33  
3.32  
1.99  
1.96  
1.90  
1.87  
1.79  
1.76  
1.64  
1.63  
1.61  
1.60  
1.58  
1.57  
1.55  
1.53  
1.51  
1.50  
1.47  
1.47  
1.46  
1.44  
1.44  
1.43  
1.38  
1.37  
1.36  
1.35  
1.34  
1.33  
1.32  
1.31  
1.30  
1.29  
1.28



3ca, <sup>1</sup>H NMR, 400 MHz, CDCl<sub>3</sub>



164.26

154.53

132.89

129.76

129.36

123.37

113.44

77.36

77.05

76.73

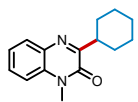
40.78

30.53

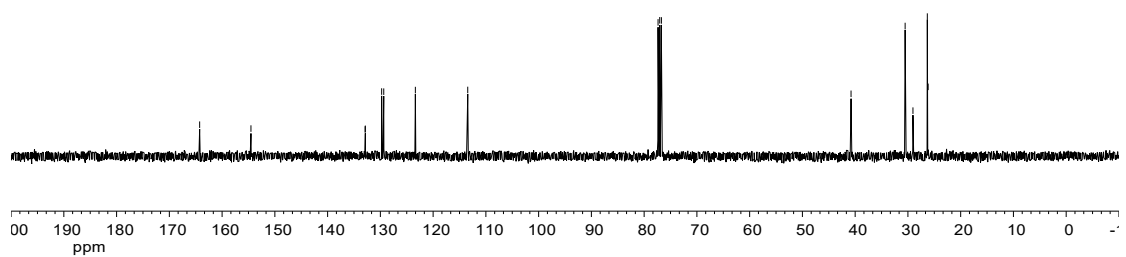
29.05

26.32

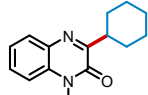
26.17



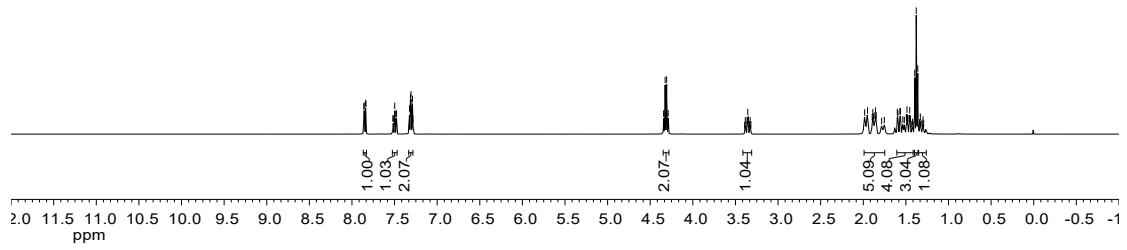
3ca, <sup>13</sup>C NMR, 101 MHz, CDCl<sub>3</sub>



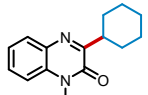
7.86  
7.86  
7.84  
7.84  
7.84  
7.52  
7.52  
7.50  
7.50  
7.49  
7.48  
7.48  
7.32  
7.32  
7.31  
7.31  
7.29  
7.29  
7.28  
7.28  
4.34  
4.33  
4.31  
4.31  
4.29  
3.38  
3.36  
3.36  
3.35  
3.33  
3.33  
1.98  
1.95  
1.90  
1.89  
1.88  
1.86  
1.86  
1.85  
1.85  
1.78  
1.75  
1.75  
1.60  
1.59  
1.57  
1.56  
1.54  
1.54  
1.52  
1.49  
1.49  
1.48  
1.46  
1.46  
1.46  
1.45  
1.42  
1.40  
1.38  
1.36  
1.34  
1.33  
1.32  
1.31  
1.30  
1.29



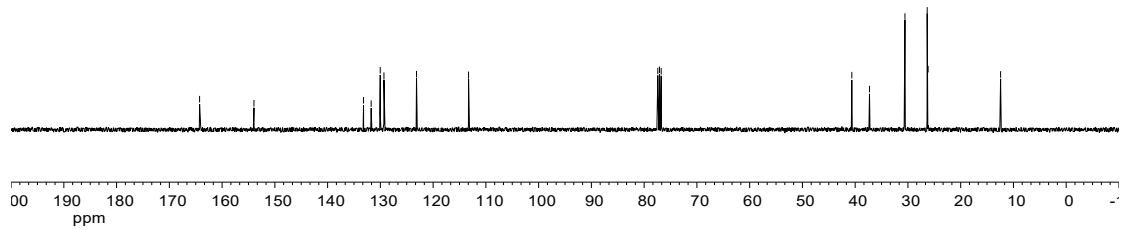
**3cb**, <sup>1</sup>H NMR, 400 MHz, CDCl<sub>3</sub>



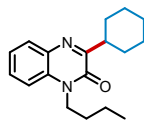
164.27  
153.96  
133.19  
131.75  
130.02  
129.32  
123.15  
113.28  
77.41  
77.09  
76.77  
40.64  
37.28  
30.56  
26.35  
26.17  
12.43



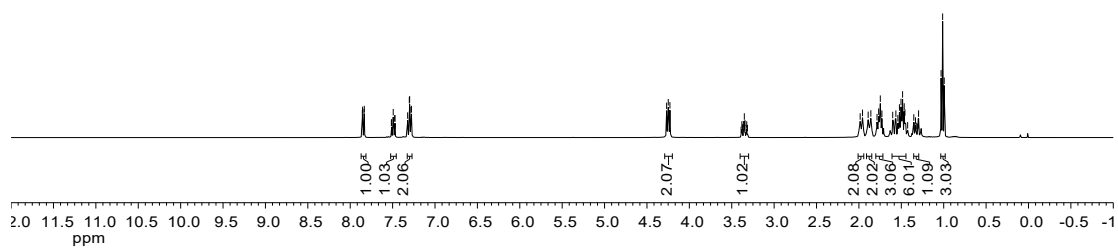
**3cb**, <sup>13</sup>C NMR, 101 MHz, CDCl<sub>3</sub>



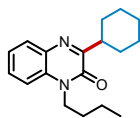
7.86  
7.85  
7.84  
7.83  
7.51  
7.51  
7.49  
7.48  
7.47  
7.32  
7.30  
7.28  
7.28  
4.27  
4.25  
4.23  
3.39  
3.38  
3.37  
3.36  
3.35  
3.34  
3.33  
3.32  
3.31  
1.99  
1.96  
1.89  
1.86  
1.79  
1.77  
1.75  
1.73  
1.60  
1.56  
1.54  
1.52  
1.50  
1.48  
1.47  
1.46  
1.43  
1.35  
1.34  
1.33  
1.32  
1.31  
1.30  
1.30  
1.03  
1.01  
0.99



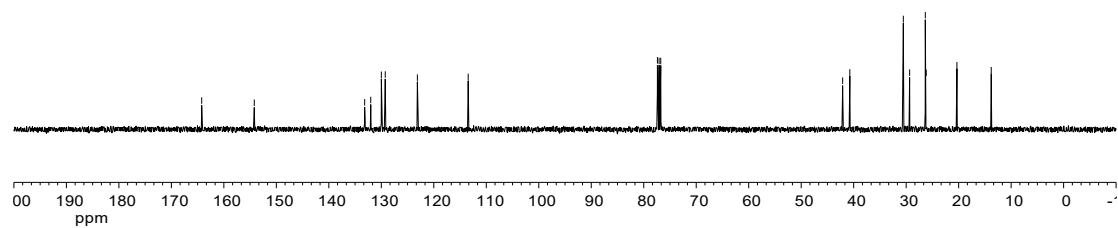
3cc,  $^1\text{H NMR}$ , 400 MHz,  $\text{CDCl}_3$



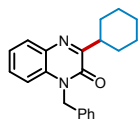
164.22  
154.21  
133.17  
132.01  
129.99  
129.25  
123.13  
113.44  
77.39  
77.07  
76.76  
42.10  
40.73  
30.54  
29.34  
26.35  
26.18  
20.33  
13.79



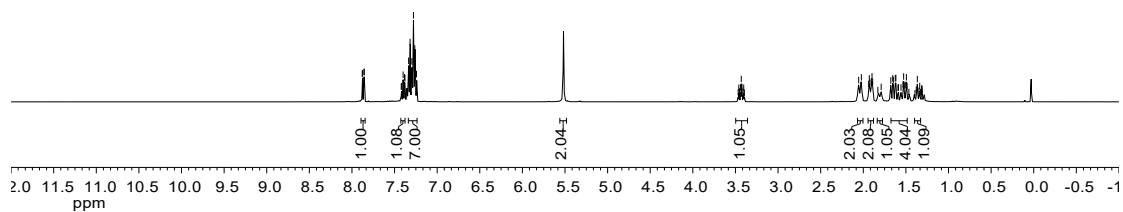
3cc,  $^{13}\text{C NMR}$ , 101 MHz,  $\text{CDCl}_3$



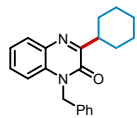
7.88  
7.86  
7.86  
7.86  
7.42  
7.42  
7.40  
7.38  
7.38  
7.34  
7.32  
7.30  
7.28  
7.26  
7.24  
3.47  
3.46  
3.45  
3.44  
3.43  
3.42  
3.41  
3.40  
3.40  
2.05  
2.02  
1.94  
1.93  
1.92  
1.90  
1.90  
1.89  
1.83  
1.79  
1.68  
1.65  
1.65  
1.62  
1.62  
1.59  
1.59  
1.56  
1.53  
1.53  
1.52  
1.50  
1.49  
1.49  
1.46  
1.39  
1.38  
1.37  
1.35  
1.34  
1.33



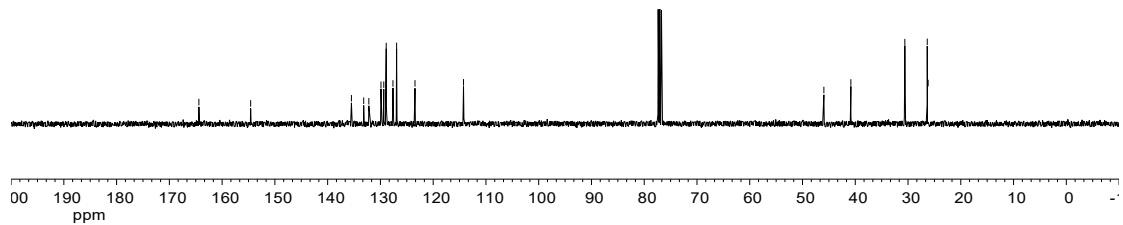
**3cd**,  $^1\text{H NMR}$ , 400 MHz,  $\text{CDCl}_3$



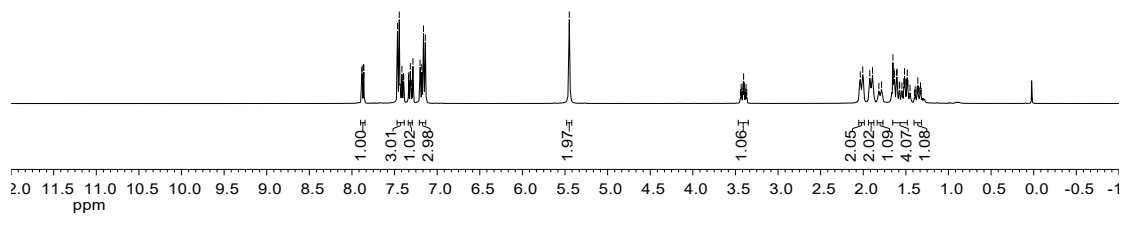
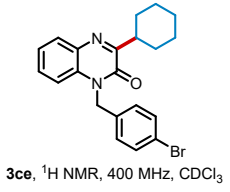
164.41  
154.60  
135.49  
133.17  
132.19  
129.89  
129.36  
128.90  
127.62  
126.93  
123.45  
114.25  
45.95  
40.83  
30.61  
26.36  
26.19



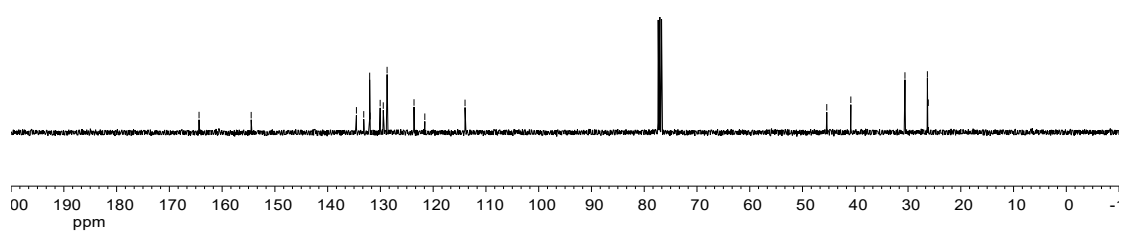
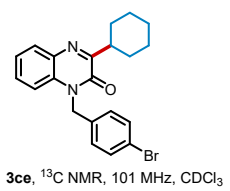
**3cd**,  $^{13}\text{C NMR}$ , 101 MHz,  $\text{CDCl}_3$

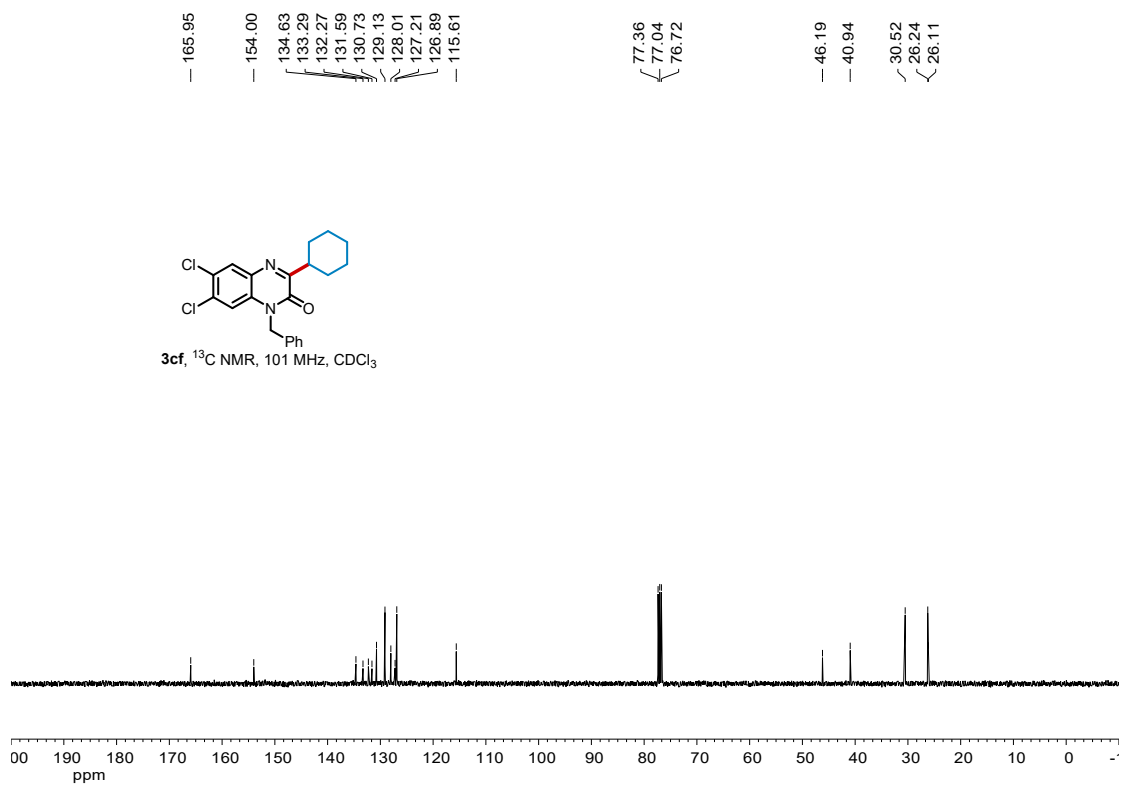
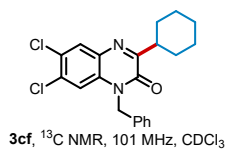
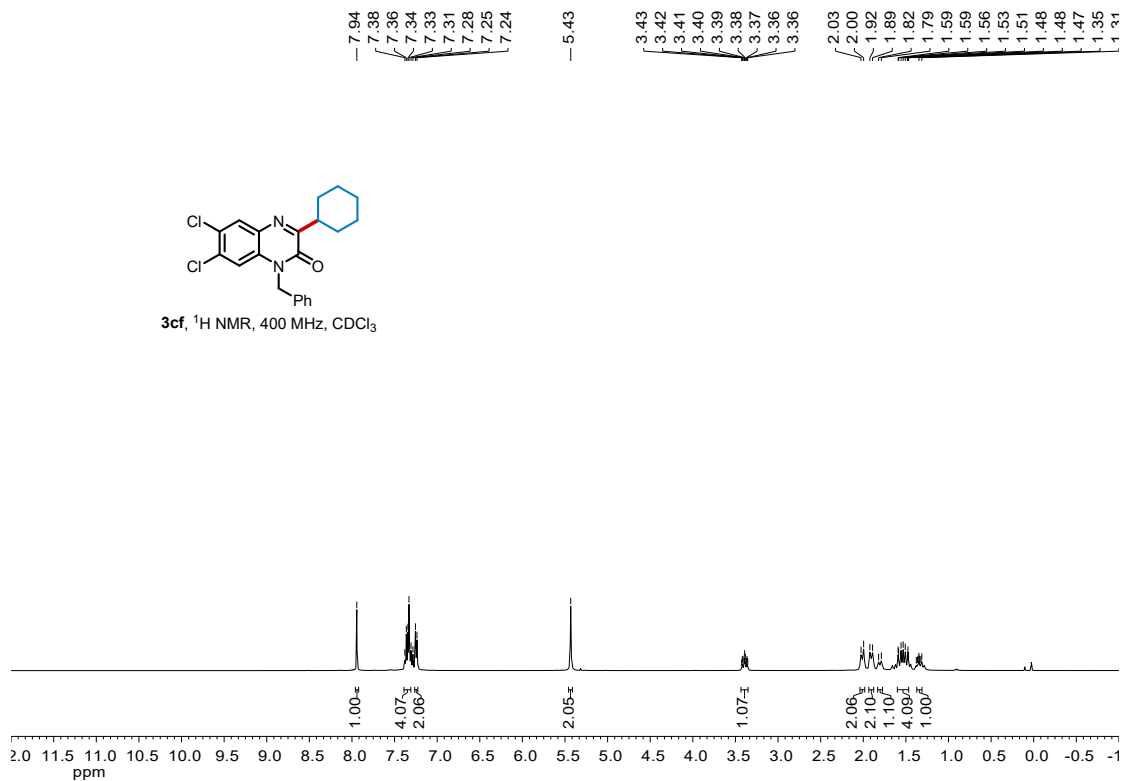
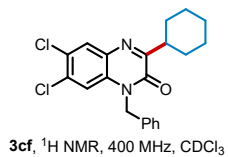


7.88  
7.88  
7.86  
7.86  
7.86  
7.47  
7.44  
7.41  
7.40  
7.39  
7.34  
7.33  
7.31  
7.30  
7.29  
7.28  
7.20  
7.18  
7.16  
7.14  
5.45  
3.44  
3.43  
3.42  
3.41  
3.40  
3.40  
3.38  
3.38  
3.37  
3.37  
2.04  
2.01  
1.92  
1.89  
1.82  
1.79  
1.65  
1.64  
1.63  
1.61  
1.60  
1.58  
1.57  
1.55  
1.52  
1.52  
1.51  
1.49  
1.48  
1.48  
1.45  
1.39  
1.38  
1.37  
1.36  
1.35  
1.34  
1.33  
1.32



164.38  
154.49  
134.55  
133.16  
132.04  
131.97  
130.05  
129.46  
128.74  
123.63  
121.58  
113.96  
45.40  
40.84  
30.58  
26.33  
26.16

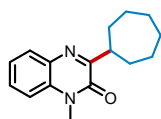




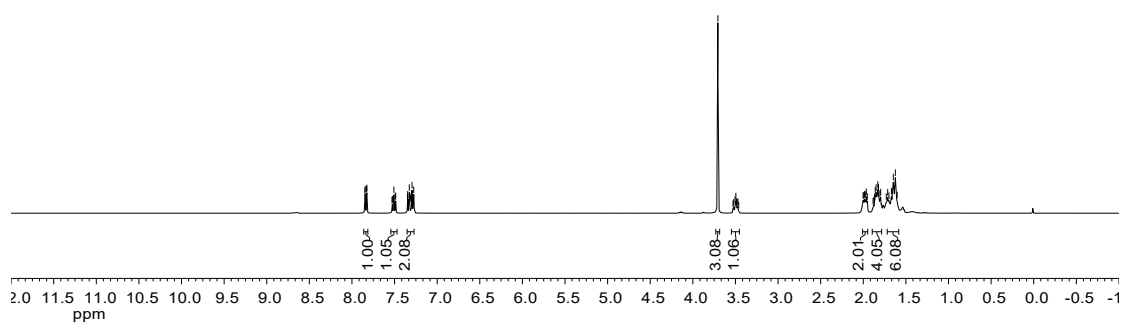
7.85  
7.84  
7.83  
7.82  
7.53  
7.53  
7.51  
7.49  
7.49  
7.35  
7.35  
7.33  
7.31  
7.31  
7.30  
7.28  
7.28

3.71  
3.53  
3.52  
3.50  
3.50  
3.49  
3.48  
3.47  
3.46

2.00  
1.99  
1.98  
1.96  
1.86  
1.85  
1.83  
1.82  
1.80  
1.80  
1.71  
1.70  
1.67  
1.65  
1.62  
1.60



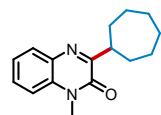
**3da**,  $^1\text{H}$  NMR, 400 MHz,  $\text{CDCl}_3$



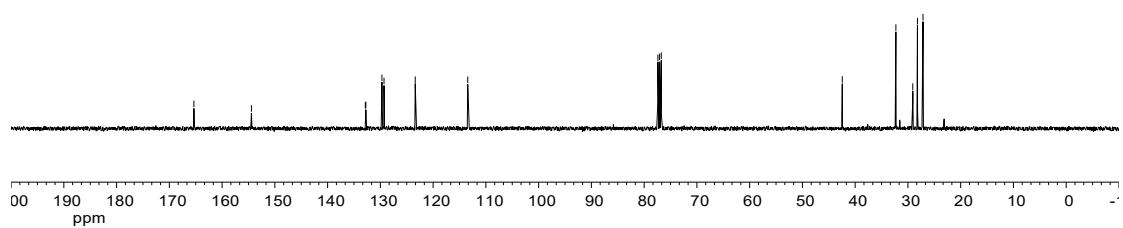
165.34  
154.44  
132.85  
132.75  
129.70  
129.31  
123.39  
113.44

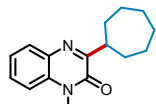
77.39  
77.07  
76.75

42.44  
32.28  
29.08  
28.19  
27.14

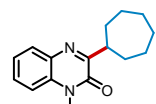
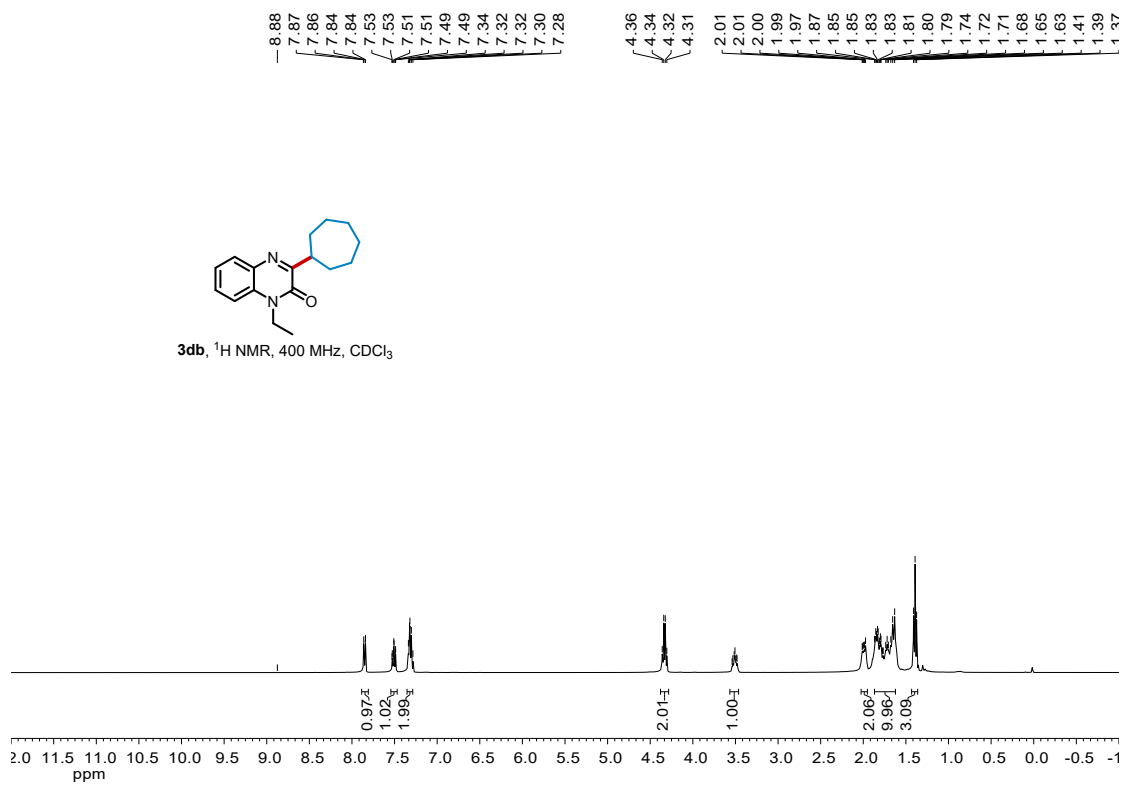


**3da**,  $^{13}\text{C}$  NMR, 101 MHz,  $\text{CDCl}_3$

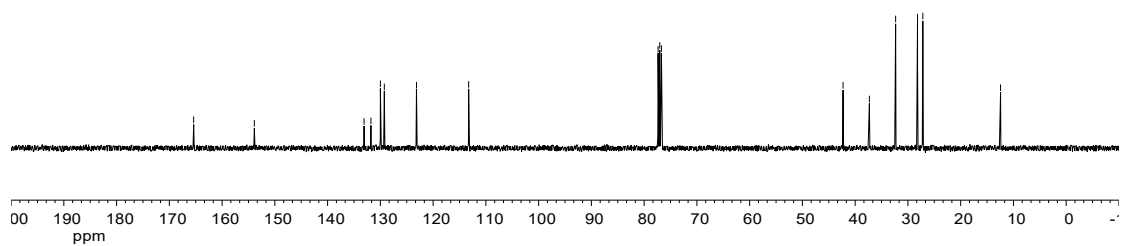




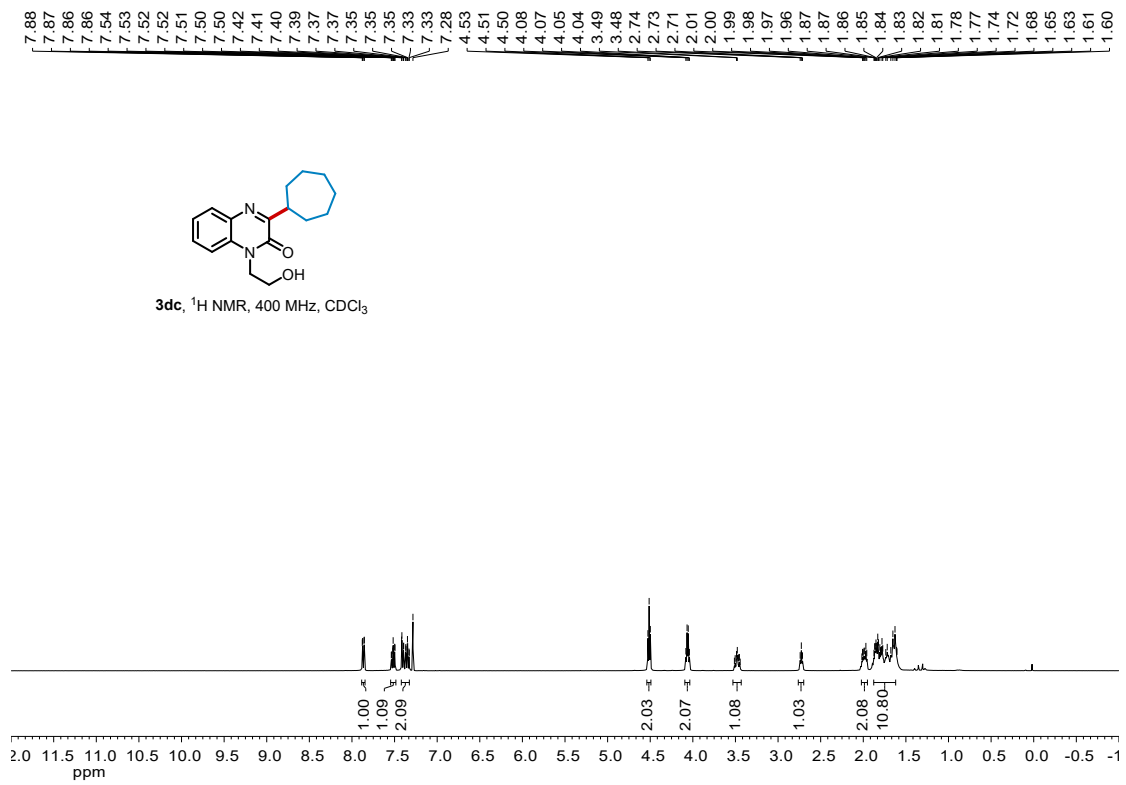
3db, <sup>1</sup>H NMR, 400 MHz, CDCl<sub>3</sub>



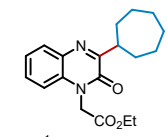
3db, <sup>13</sup>C NMR, 101 MHz, CDCl<sub>3</sub>



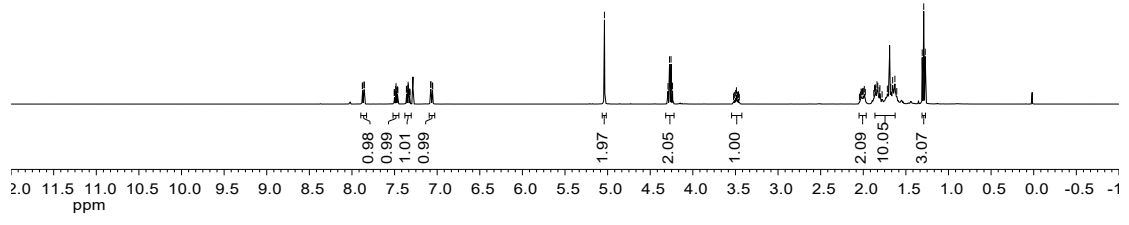




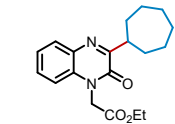
7.88  
7.87  
7.86  
7.85  
7.50  
7.49  
7.48  
7.47  
7.46  
7.36  
7.36  
7.34  
7.34  
7.34  
7.32  
7.32  
7.32  
7.08  
7.07  
7.06  
7.05  
5.04  
4.29  
4.27  
4.26  
4.24  
4.24  
3.51  
3.50  
3.49  
3.48  
3.46  
2.03  
2.03  
2.02  
2.01  
2.00  
2.00  
1.99  
1.98  
1.88  
1.87  
1.86  
1.85  
1.84  
1.83  
1.81  
1.80  
1.78  
1.72  
1.68  
1.66  
1.63  
1.61  
1.31  
1.29  
1.27



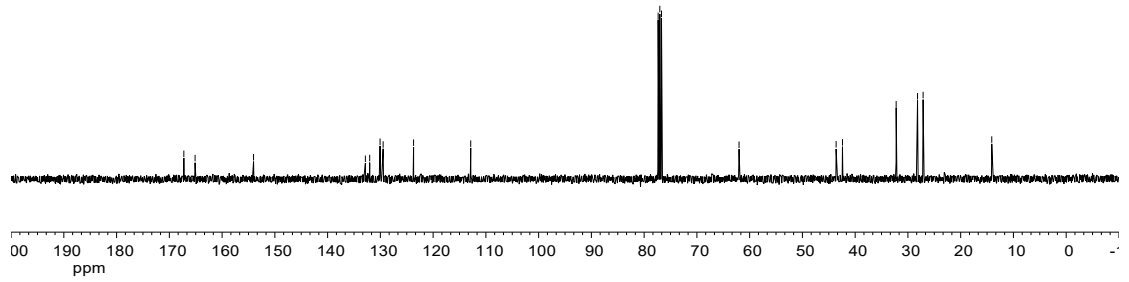
3dd, <sup>1</sup>H NMR, 400 MHz, CDCl<sub>3</sub>

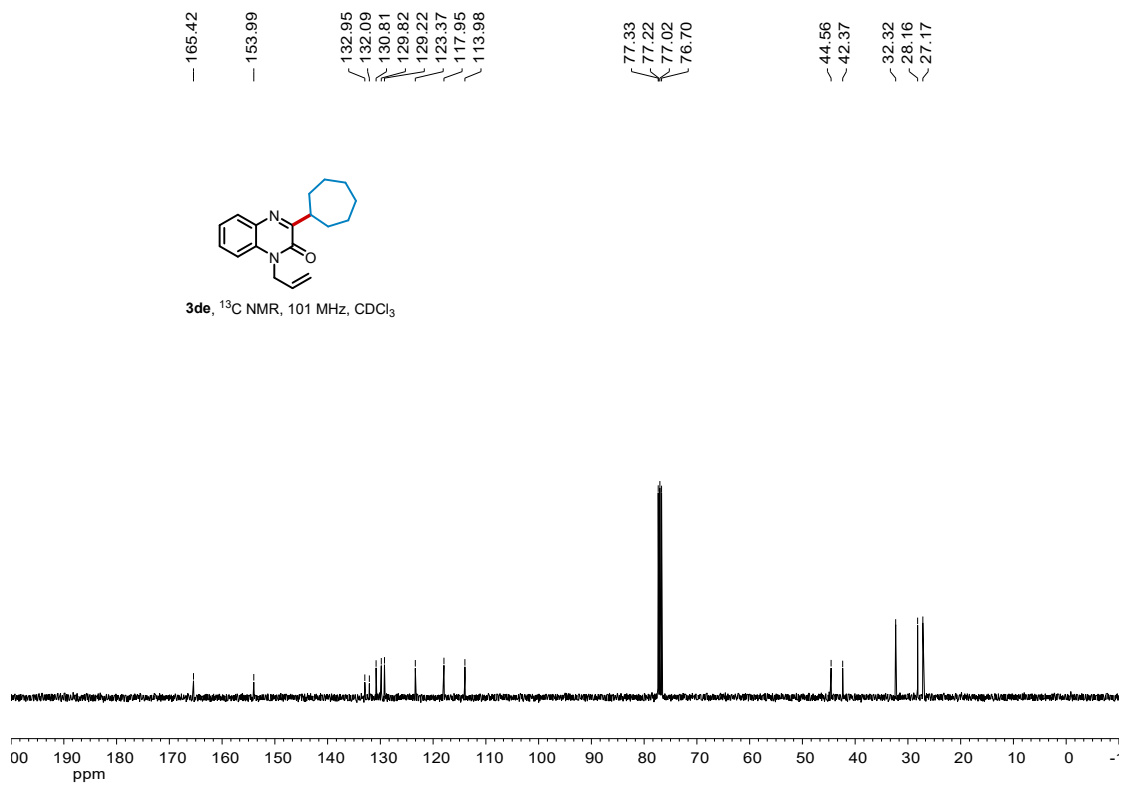
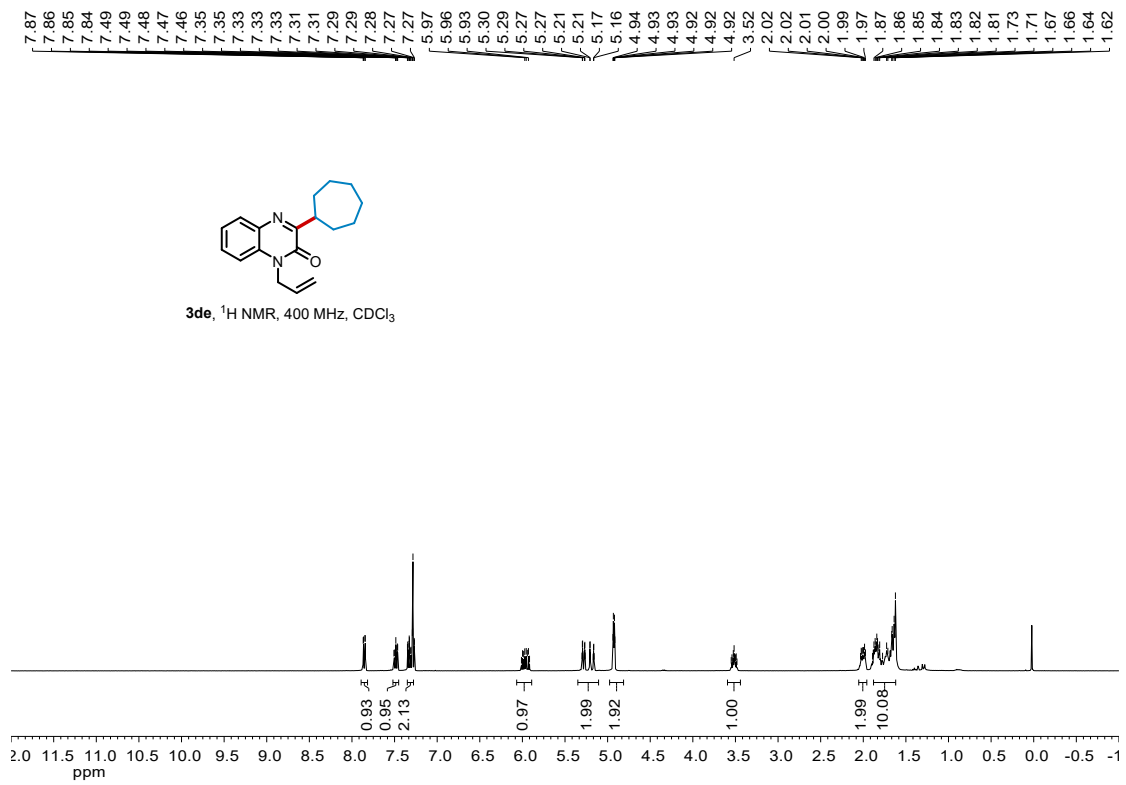


167.25  
165.12  
154.04  
132.84  
132.02  
130.06  
129.47  
123.70  
112.87  
77.34  
77.03  
76.71  
62.01  
43.61  
42.40  
32.23  
28.18  
27.12  
14.11

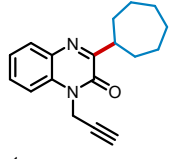


3dd, <sup>13</sup>C NMR, 101 MHz, CDCl<sub>3</sub>

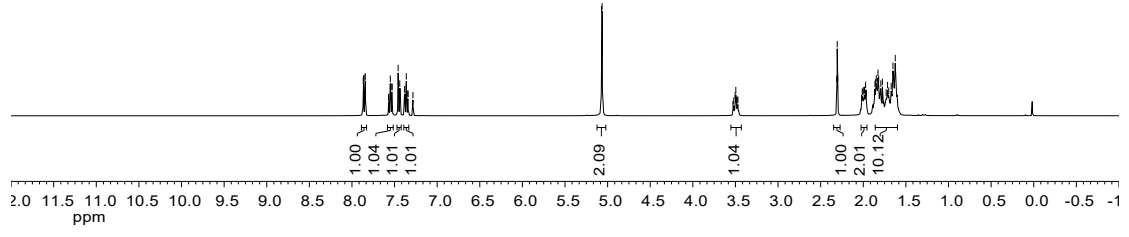




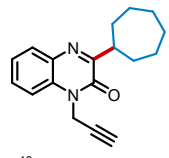
7.87  
7.86  
7.85  
7.84  
7.57  
7.55  
7.55  
7.53  
7.46  
7.44  
7.38  
7.38  
7.36  
7.34  
7.34  
7.28  
5.07  
5.06  
3.53  
3.52  
3.50  
3.50  
3.49  
3.48  
3.47  
2.31  
2.31  
2.30  
1.99  
1.99  
1.86  
1.85  
1.85  
1.83  
1.82  
1.79  
1.78  
1.72  
1.67  
1.65  
1.62



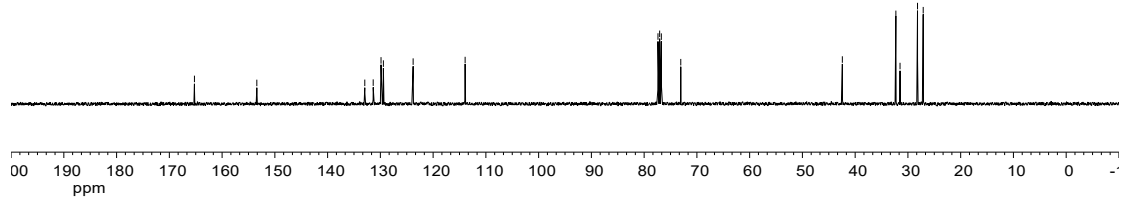
3df, <sup>1</sup>H NMR, 400 MHz, CDCl<sub>3</sub>



165.24  
153.42  
132.95  
131.36  
129.88  
129.43  
123.79  
113.93  
77.38  
77.06  
77.02  
76.74  
73.06  
42.43  
32.28  
31.49  
28.18  
27.10



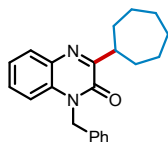
3df, <sup>13</sup>C NMR, 101 MHz, CDCl<sub>3</sub>



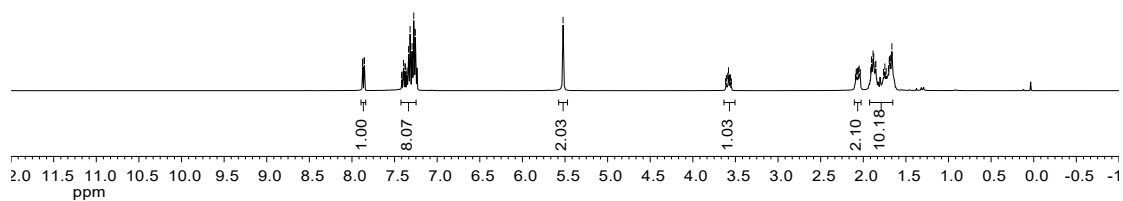
7.88  
7.87  
7.86  
7.85  
7.82  
7.41  
7.39  
7.38  
7.37  
7.36  
7.35  
7.34  
7.32  
7.29  
7.28  
7.27  
7.26  
-5.52

3.62  
3.61  
3.59  
3.58  
3.57  
3.57  
3.56  
3.55

2.08  
2.07  
2.06  
2.05  
1.91  
1.90  
1.89  
1.88  
1.86  
1.85  
1.75  
1.73  
1.70  
1.70  
1.69  
1.68  
1.66



3dg,  $^1\text{H}$  NMR, 400 MHz,  $\text{CDCl}_3$

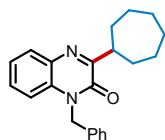


165.50  
154.51  
135.48  
133.04  
132.23  
129.85  
129.32  
128.91  
128.88  
127.62  
126.88  
123.46  
114.25

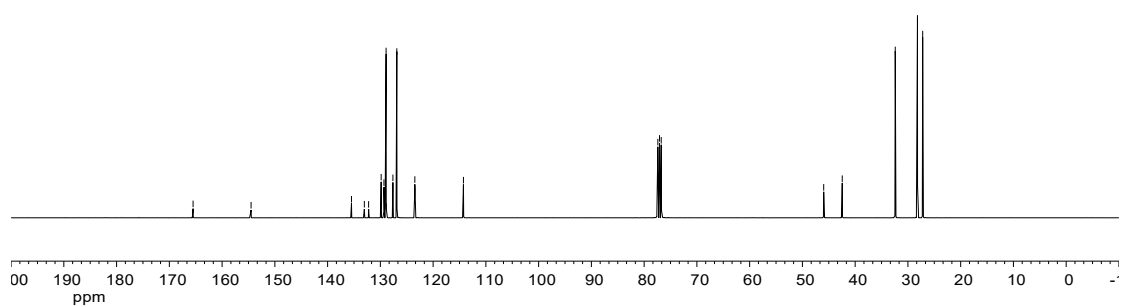
77.40  
77.08  
76.77

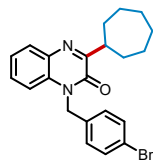
45.96  
42.45

32.40  
28.19  
27.20

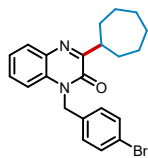
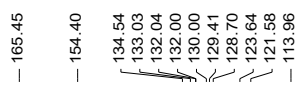
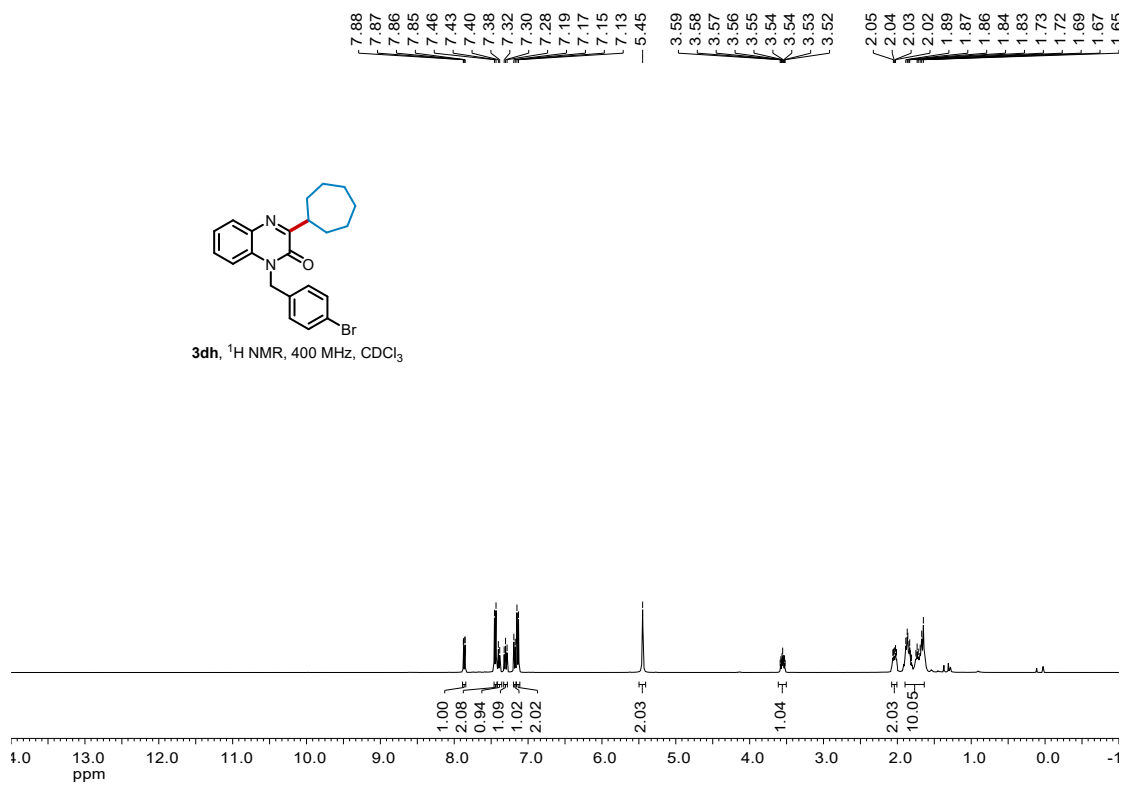


3dg,  $^{13}\text{C}$  NMR, 101 MHz,  $\text{CDCl}_3$

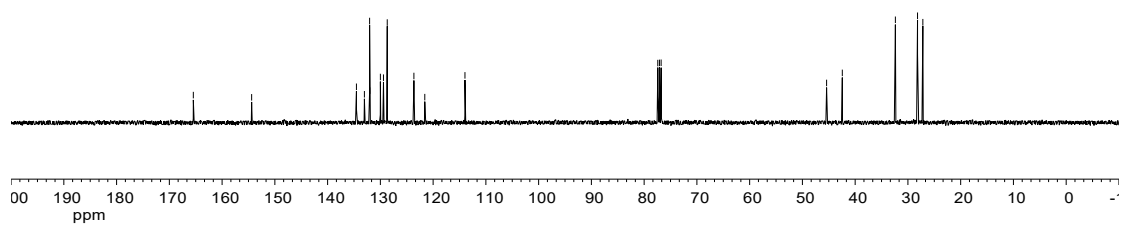


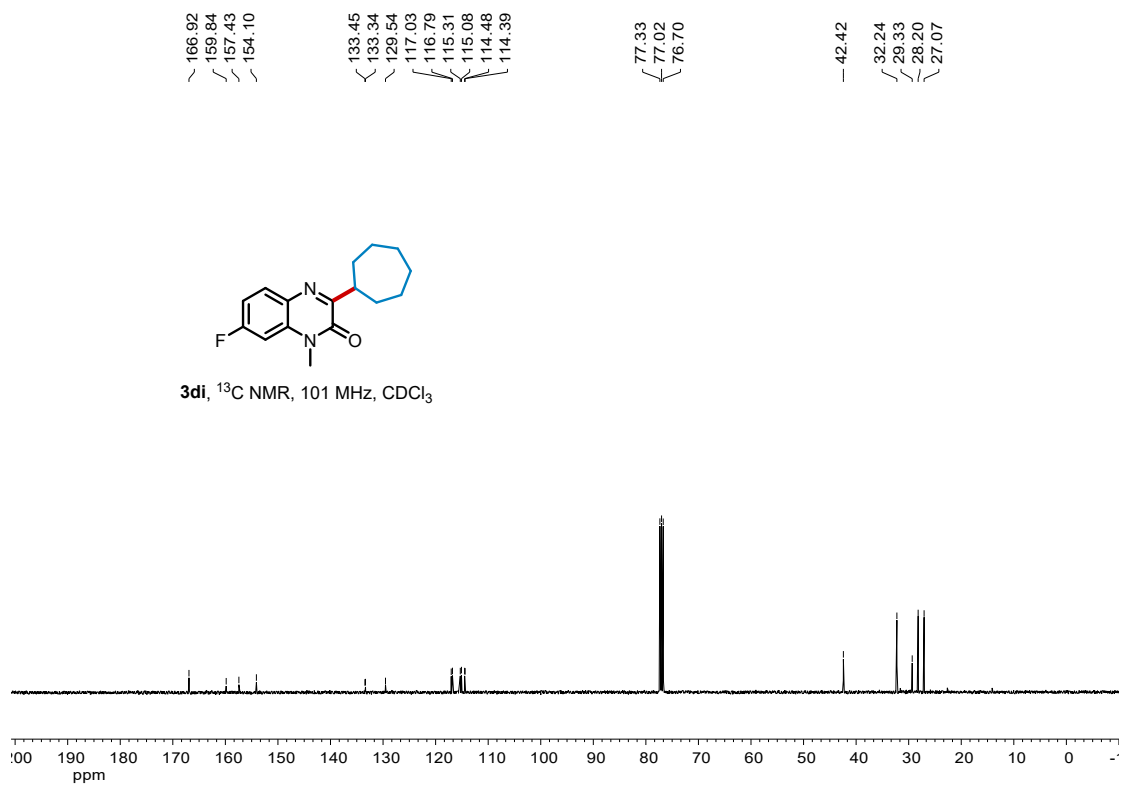
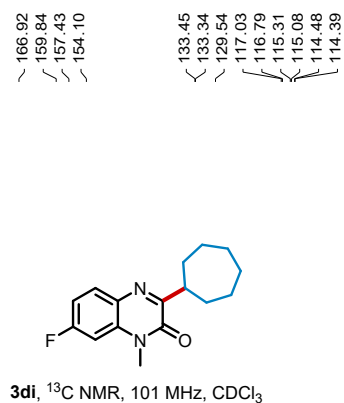
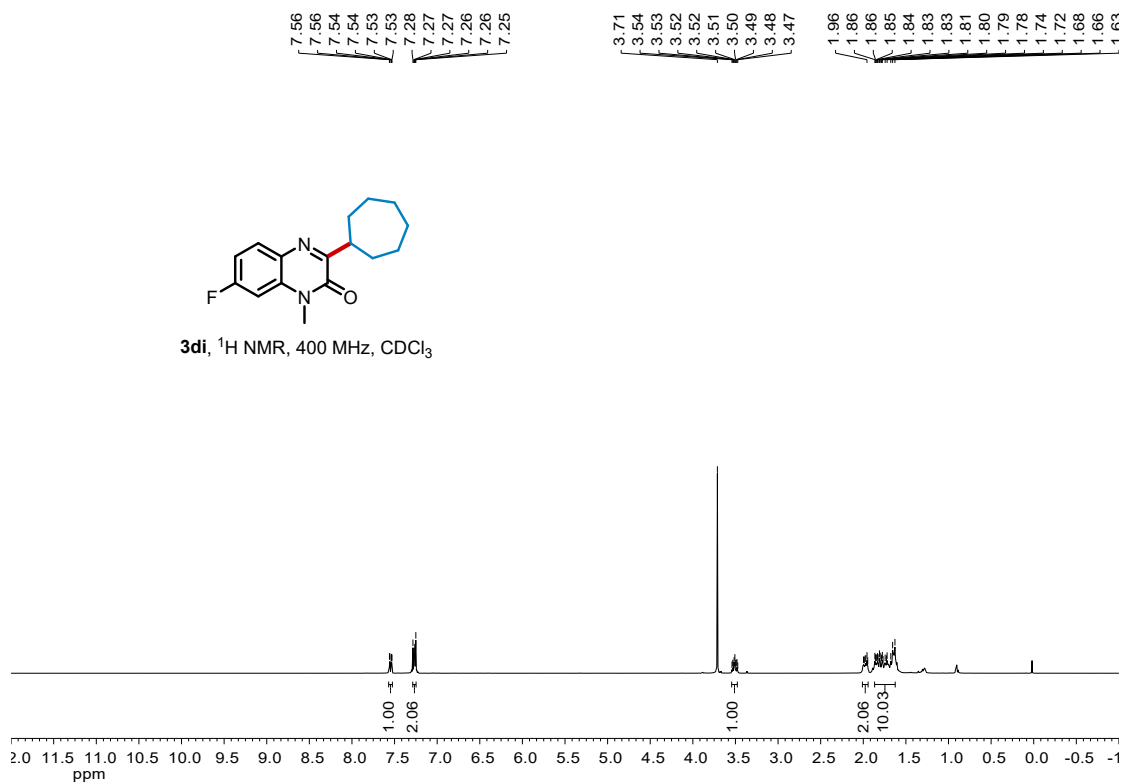
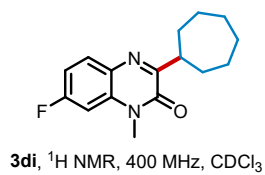


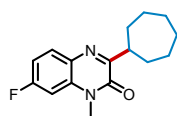
**3dh**,  $^1\text{H}$  NMR, 400 MHz,  $\text{CDCl}_3$



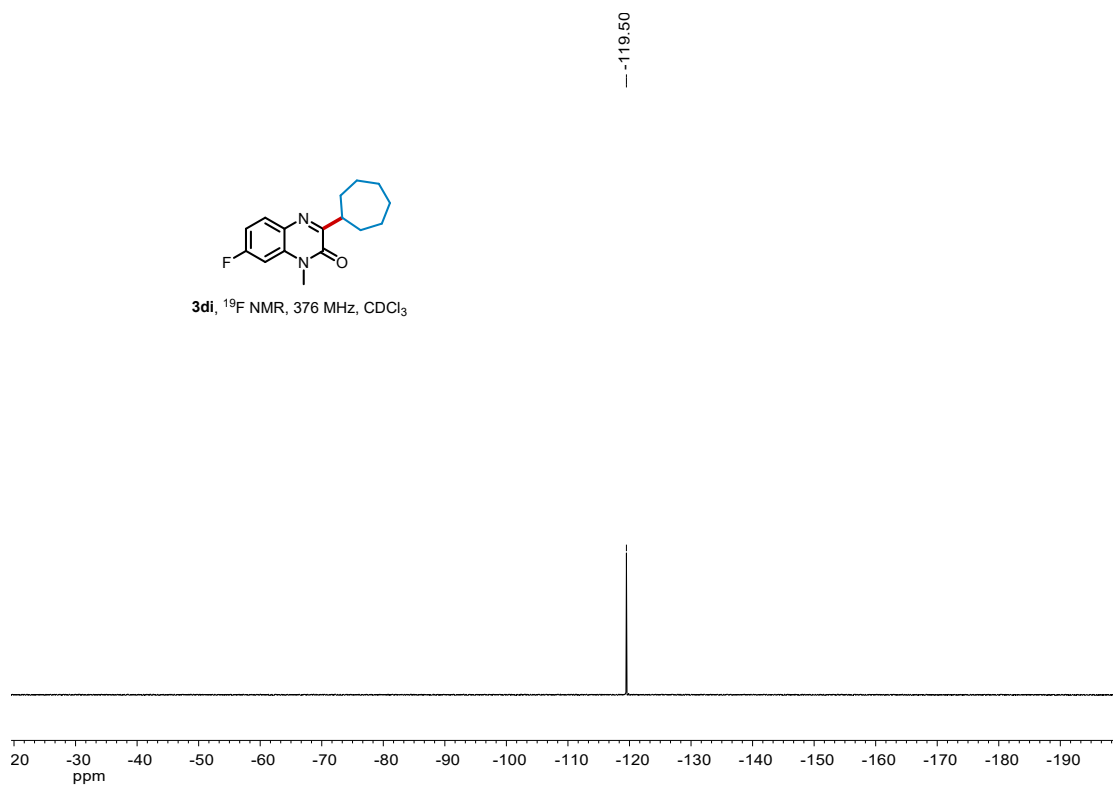
**3dh**,  $^{13}\text{C}$  NMR, 101 MHz,  $\text{CDCl}_3$







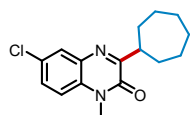
**3di**,  $^{19}\text{F}$  NMR, 376 MHz,  $\text{CDCl}_3$



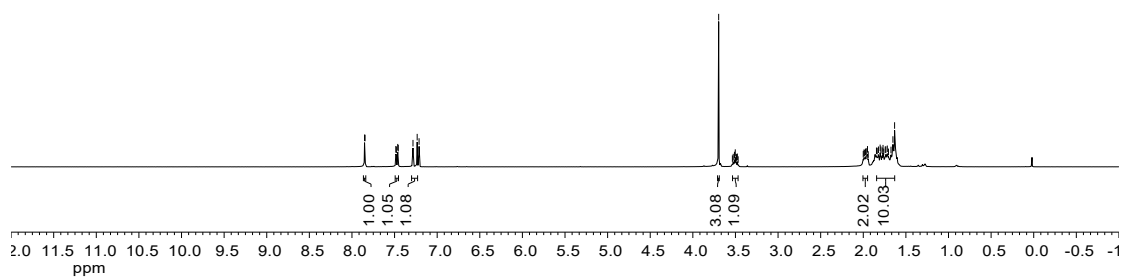
7.85  
7.85  
7.49  
7.48  
7.46  
7.46  
7.28  
7.23  
7.21

3.70  
3.54  
3.53  
3.51  
3.50  
3.49  
3.49  
3.48  
3.47

1.96  
1.95  
1.84  
1.82  
1.80  
1.79  
1.78  
1.77  
1.74  
1.74  
1.72  
1.70  
1.67  
1.65  
1.63

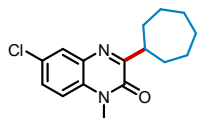


**3dj**,  $^1\text{H}$  NMR, 400 MHz,  $\text{CDCl}_3$

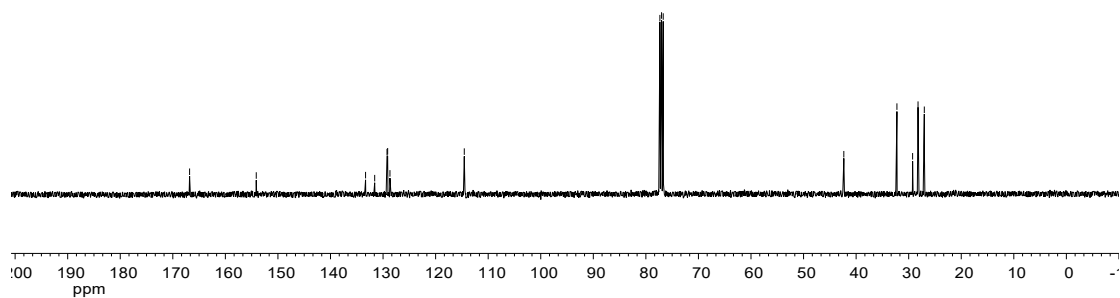




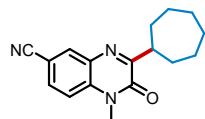
- 166.81  
 - 154.12  
 133.33  
 131.59  
 129.25  
 129.12  
 128.69  
 - 114.55  
 77.33  
 77.02  
 76.70  
 - 42.34  
 32.24  
 29.25  
 28.22  
 27.03



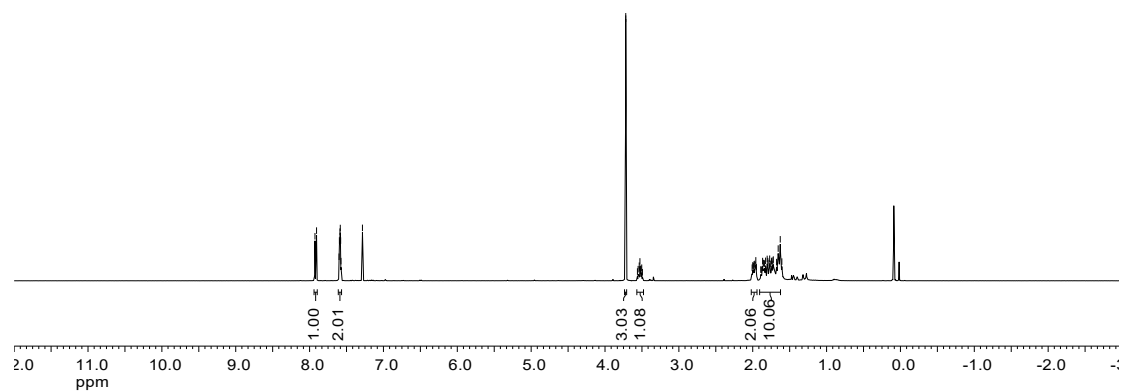
3dj,  $^{13}\text{C}$  NMR, 101 MHz,  $\text{CDCl}_3$



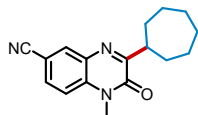
7.93  
 7.91  
 7.60  
 7.59  
 7.58  
 7.58  
 7.28  
 3.72  
 3.56  
 3.55  
 3.54  
 3.53  
 3.52  
 3.51  
 3.50  
 3.49  
 1.98  
 1.97  
 1.96  
 1.87  
 1.86  
 1.85  
 1.84  
 1.83  
 1.80  
 1.80  
 1.78  
 1.77  
 1.76  
 1.75  
 1.74  
 1.73  
 1.72  
 1.68  
 1.66  
 1.63  
 1.54



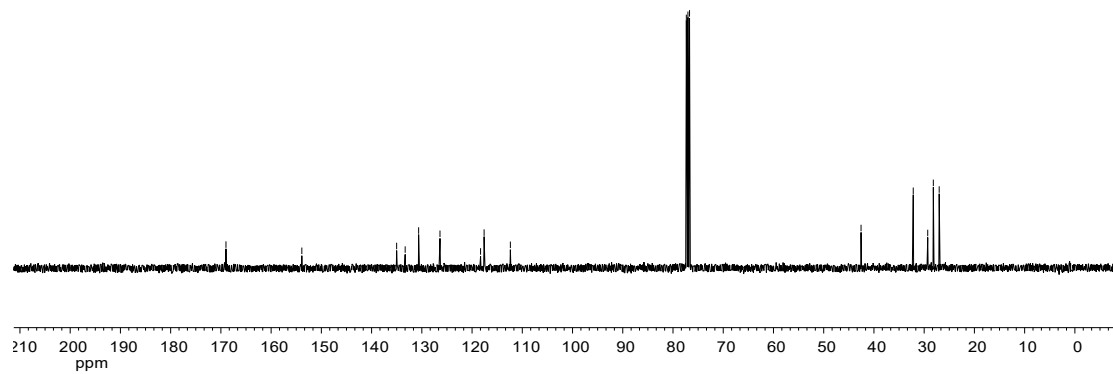
3dk,  $^1\text{H}$  NMR, 400 MHz,  $\text{CDCl}_3$



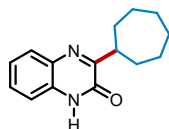
— 168.99  
 — 153.89  
 ~ 135.01  
 ~ 133.32  
 ~ 130.64  
 ~ 126.39  
 ~ 118.31  
 ~ 117.60  
 ~ 112.38  
 ~ 77.35  
 ~ 77.03  
 ~ 76.71  
 — 42.57  
 ~ 32.18  
 ~ 29.29  
 ~ 28.18  
 ~ 27.01



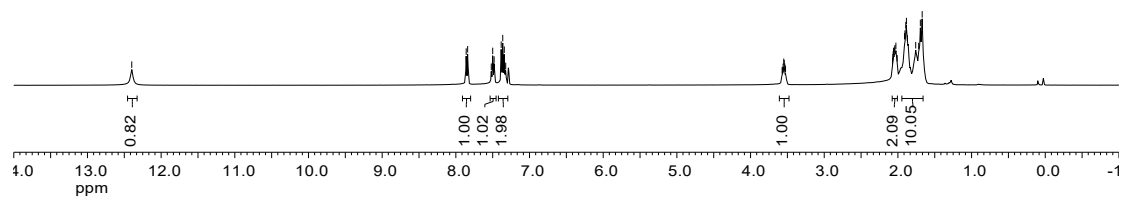
3dk,  $^{13}\text{C}$  NMR, 101 MHz,  $\text{CDCl}_3$



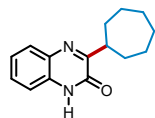
— 12.40  
 ~ 7.86  
 ~ 7.84  
 ~ 7.52  
 ~ 7.50  
 ~ 7.48  
 ~ 7.38  
 ~ 7.36  
 ~ 7.34  
 ~ 7.32  
 ~ 3.57  
 ~ 3.56  
 ~ 3.56  
 ~ 3.55  
 ~ 3.54  
 ~ 3.54  
 ~ 3.53  
 ~ 2.06  
 ~ 2.05  
 ~ 2.05  
 ~ 2.04  
 ~ 2.03  
 ~ 1.91  
 ~ 1.90  
 ~ 1.89  
 ~ 1.88  
 ~ 1.87  
 ~ 1.76  
 ~ 1.72  
 ~ 1.70  
 ~ 1.67



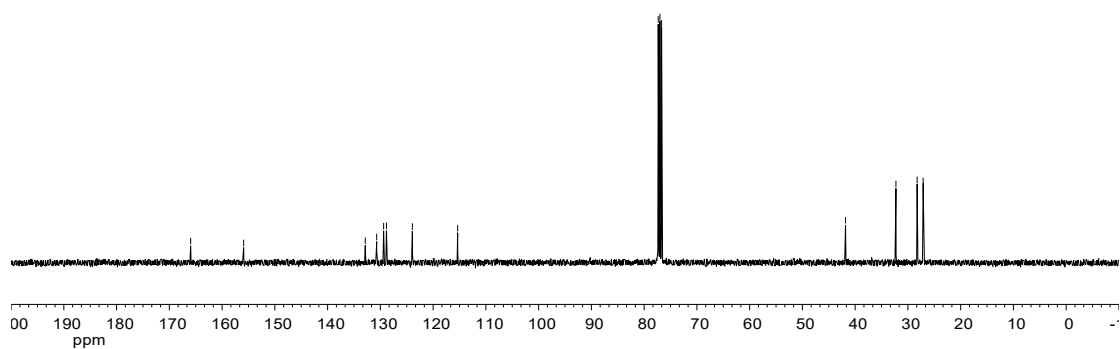
3dl,  $^1\text{H}$  NMR, 400 MHz,  $\text{CDCl}_3$



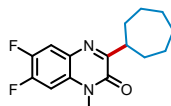
- 165.96  
 - 155.92  
 132.86  
 130.70  
 129.38  
 128.84  
 123.93  
 - 115.35  
 77.33  
 77.21  
 77.01  
 76.69  
 - 41.83  
 32.27  
 28.25  
 27.11



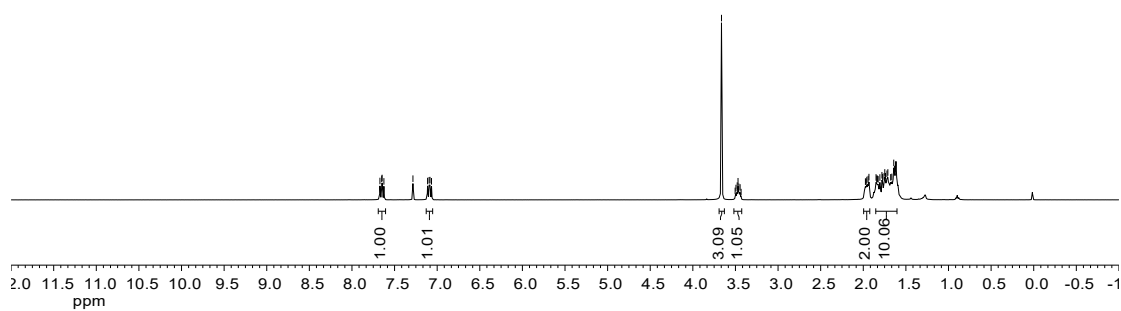
3dl,  $^{13}\text{C}$  NMR, 101 MHz,  $\text{CDCl}_3$



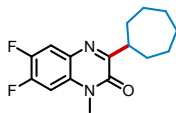
7.67  
 7.65  
 7.63  
 7.28  
 7.11  
 7.09  
 7.08  
 7.07  
 3.66  
 3.50  
 3.49  
 3.48  
 3.47  
 3.46  
 3.45  
 3.43  
 1.93  
 1.85  
 1.84  
 1.83  
 1.81  
 1.78  
 1.77  
 1.76  
 1.75  
 1.74  
 1.72  
 1.71  
 1.68  
 1.67  
 1.64  
 1.63



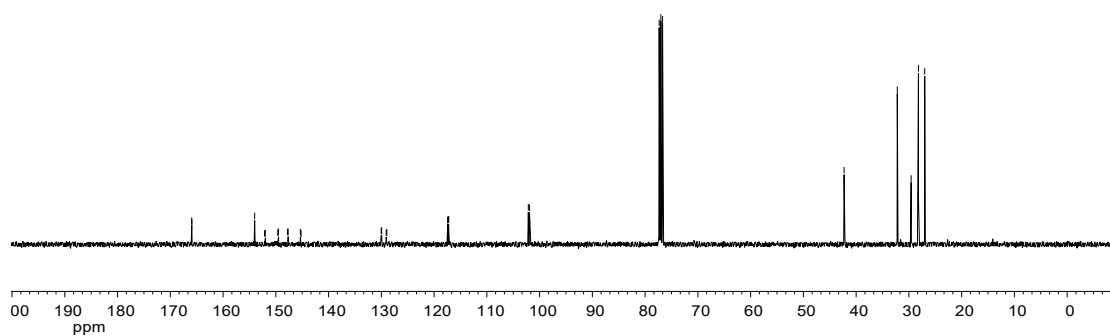
3dm,  $^1\text{H}$  NMR, 400 MHz,  $\text{CDCl}_3$



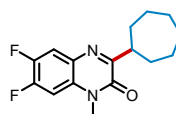
165.94  
165.91  
154.02  
152.14  
151.99  
149.63  
149.49  
147.80  
147.66  
145.35  
145.21  
130.03  
129.94  
129.08  
128.96  
117.43  
117.41  
117.26  
117.23  
102.14  
101.91  
77.33  
77.01  
76.70  
-42.28  
32.20  
29.57  
28.19  
27.00



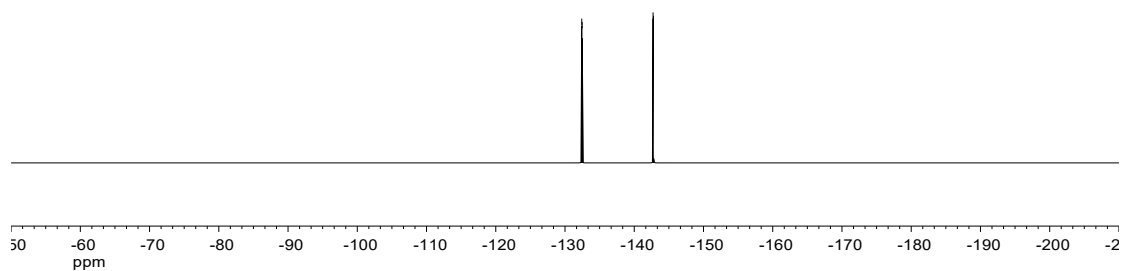
**3dm**,  $^{13}\text{C}$  NMR, 101 MHz,  $\text{CDCl}_3$

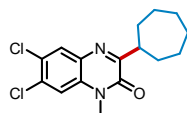


-132.42  
-132.48  
-142.72  
-142.78

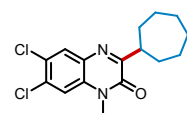
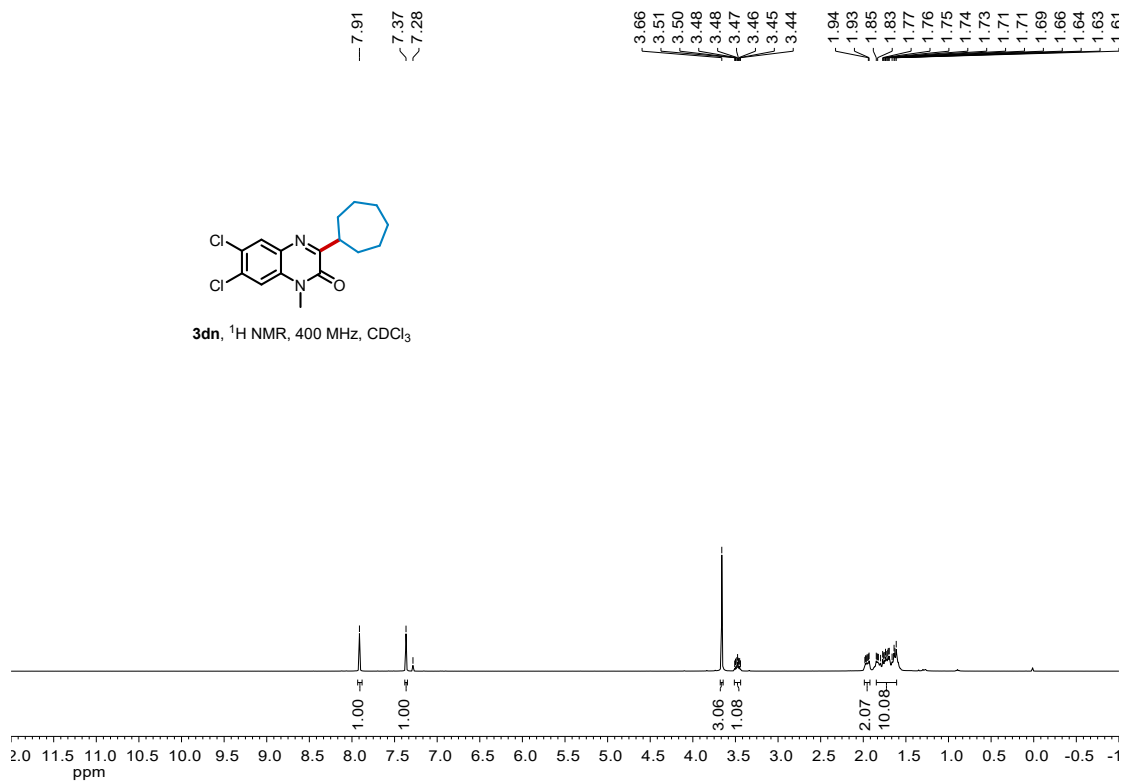


**3dm**,  $^{19}\text{F}$  NMR, 376 MHz,  $\text{CDCl}_3$

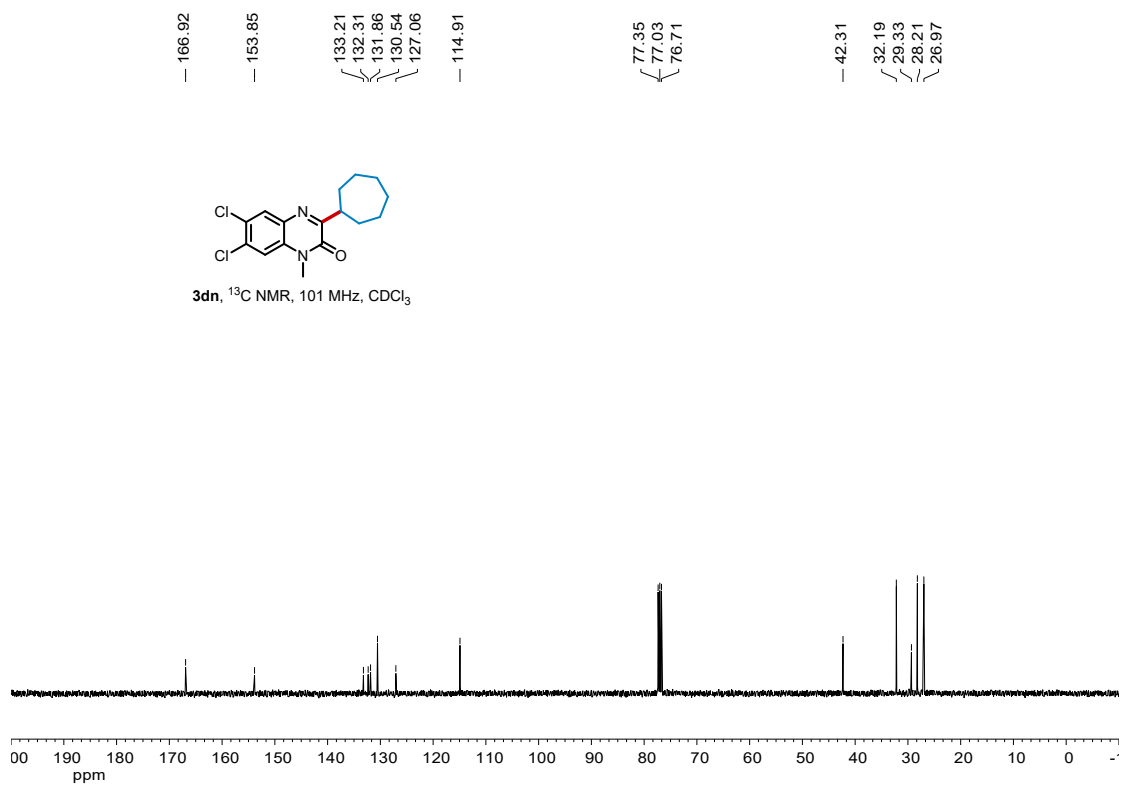




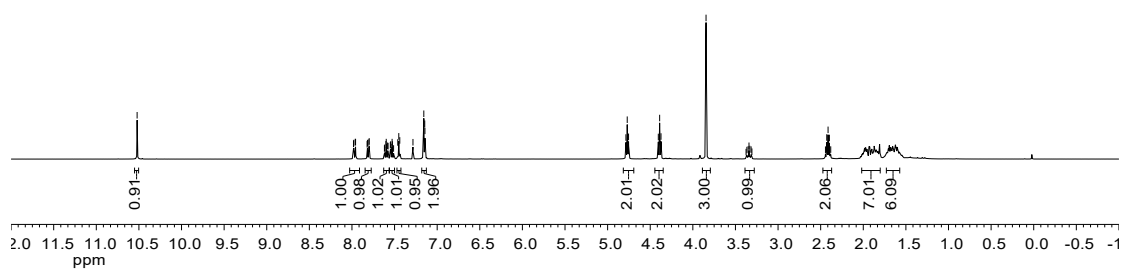
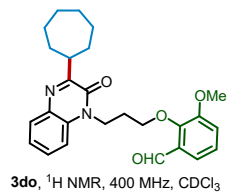
**3dn**,  $^1\text{H NMR}$ , 400 MHz,  $\text{CDCl}_3$



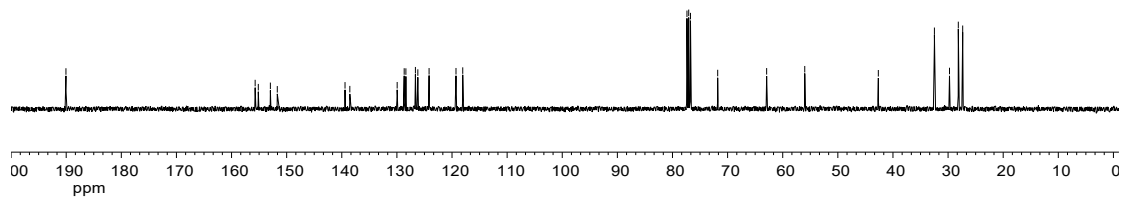
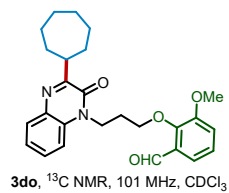
**3dn**,  $^{13}\text{C NMR}$ , 101 MHz,  $\text{CDCl}_3$



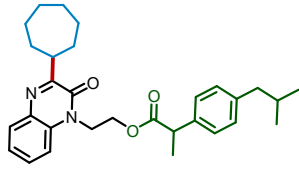
10.52  
7.98  
7.98  
7.96  
7.82  
7.82  
7.80  
7.80  
7.62  
7.62  
7.60  
7.60  
7.59  
7.58  
7.58  
7.55  
7.55  
7.53  
7.53  
7.51  
7.46  
7.45  
7.44  
7.28  
7.16  
7.15  
7.14  
4.79  
4.77  
4.76  
4.75  
4.41  
4.39  
4.37  
3.85  
3.38  
3.37  
3.35  
3.34  
3.33  
3.32  
3.31  
2.44  
2.43  
2.41  
2.40  
2.38



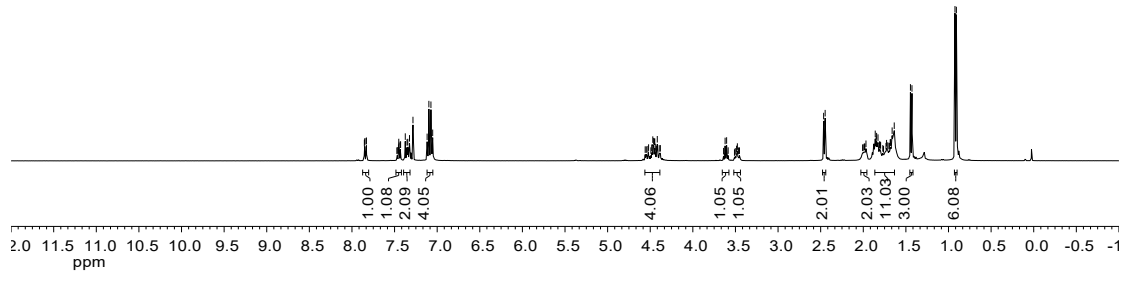
190.05  
155.72  
155.15  
152.96  
151.70  
139.41  
138.54  
129.95  
128.68  
128.37  
126.64  
126.20  
124.16  
119.28  
118.05  
77.37  
77.06  
76.74  
71.80  
62.92  
56.00  
42.65  
32.48  
29.74  
28.14  
27.35



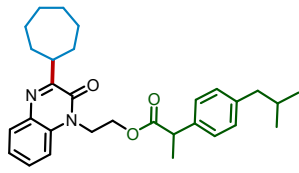
7.85  
7.85  
7.83  
7.83  
7.83  
7.45  
7.45  
7.45  
7.43  
7.43  
7.37  
7.35  
7.34  
7.32  
7.28  
7.12  
7.10  
7.07  
7.05  
7.05  
4.52  
4.48  
4.47  
4.46  
4.45  
4.45  
4.44  
4.44  
4.42  
4.42  
3.62  
3.60  
3.48  
3.48  
2.47  
2.45  
2.00  
2.00  
1.99  
1.99  
1.98  
1.97  
1.86  
1.85  
1.84  
1.84  
1.83  
1.81  
1.81  
1.80  
1.80  
1.73  
1.73  
1.71  
1.71  
1.70  
1.69  
1.68  
1.68  
1.68  
1.66  
1.64  
1.45  
1.43  
0.92  
0.91



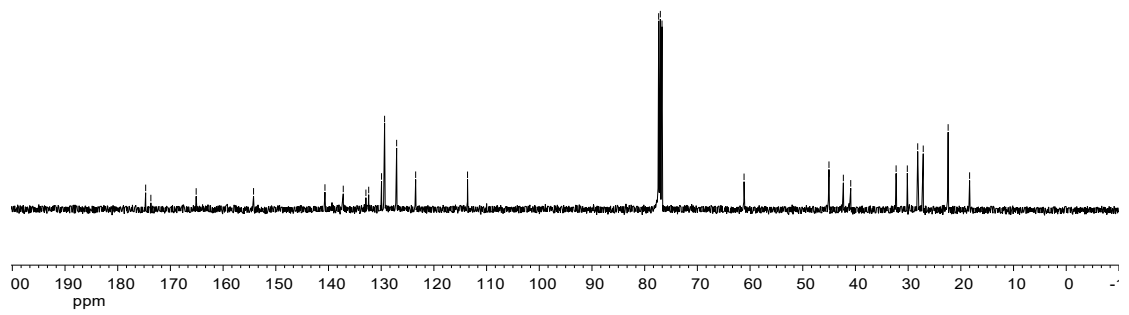
**3dp**,  $^1\text{H}$  NMR, 400 MHz,  $\text{CDCl}_3$



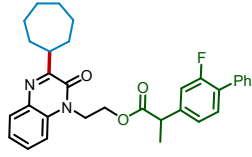
174.70  
173.71  
165.11  
154.22  
140.67  
137.20  
132.90  
132.37  
129.94  
128.36  
127.07  
123.46  
113.62  
77.35  
77.03  
76.71  
61.14  
45.03  
45.00  
42.30  
40.90  
32.29  
32.24  
30.16  
28.19  
27.14  
22.41  
18.34



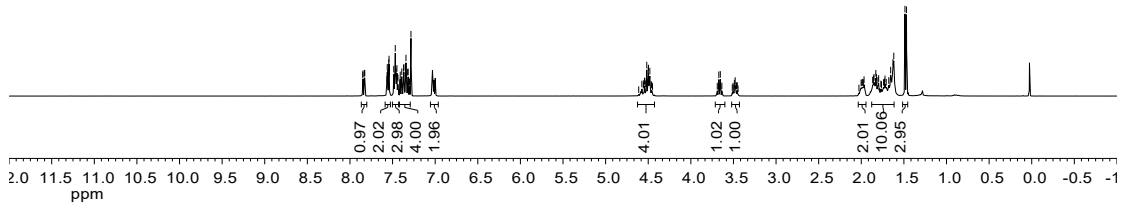
**3dp**,  $^{13}\text{C}$  NMR, 101 MHz,  $\text{CDCl}_3$



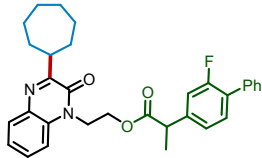
7.85  
7.85  
7.83  
7.83  
7.57  
7.56  
7.55  
7.54  
7.54  
7.49  
7.48  
7.48  
7.47  
7.46  
7.46  
7.46  
7.45  
7.44  
7.44  
7.41  
7.41  
7.39  
7.37  
7.37  
7.34  
7.34  
7.32  
7.32  
7.30  
7.28  
4.54  
4.54  
4.52  
4.50  
4.50  
4.48  
4.48  
3.67  
3.65  
3.48  
1.97  
1.87  
1.86  
1.85  
1.83  
1.82  
1.81  
1.80  
1.73  
1.72  
1.68  
1.66  
1.63  
1.62  
1.49  
1.47



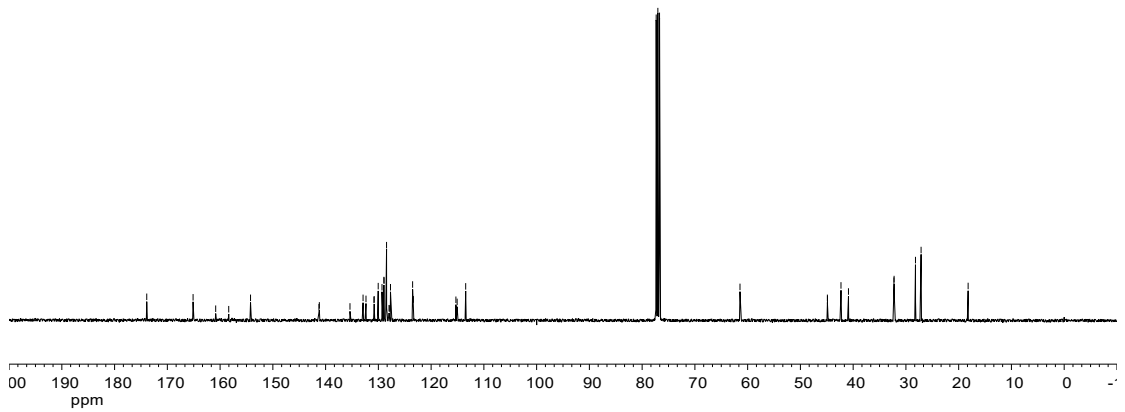
**3dq**,  $^1\text{H}$  NMR, 400 MHz,  $\text{CDCl}_3$



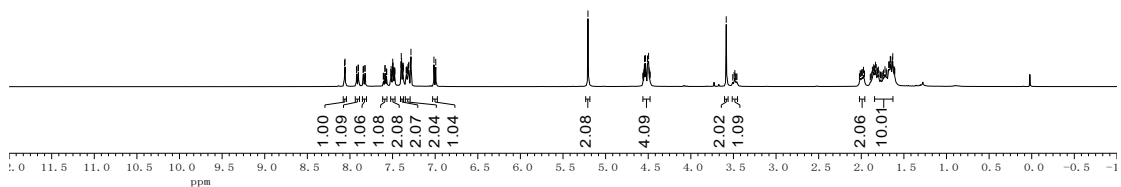
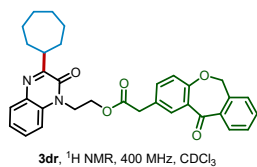
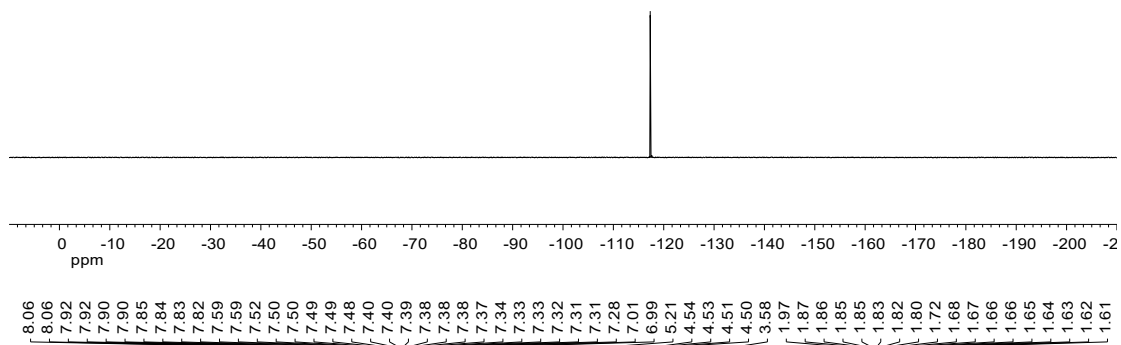
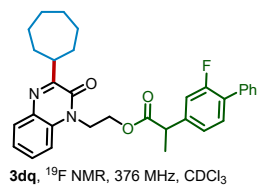
173.90  
165.12  
160.84  
158.37  
154.23  
141.26  
141.19  
135.38  
132.90  
132.35  
130.83  
130.79  
130.03  
129.31  
128.95  
128.92  
128.45  
127.98  
127.85  
127.70  
123.51  
123.42  
123.38  
115.30  
115.07  
113.45  
77.34  
77.02  
76.70  
61.45  
44.88  
44.87  
42.31  
40.89  
32.27  
32.24  
28.19  
27.13  
18.21

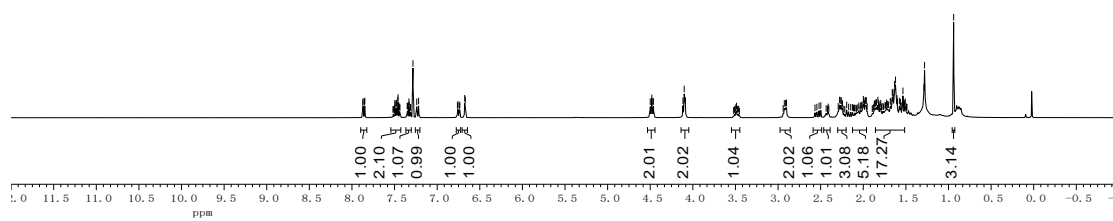
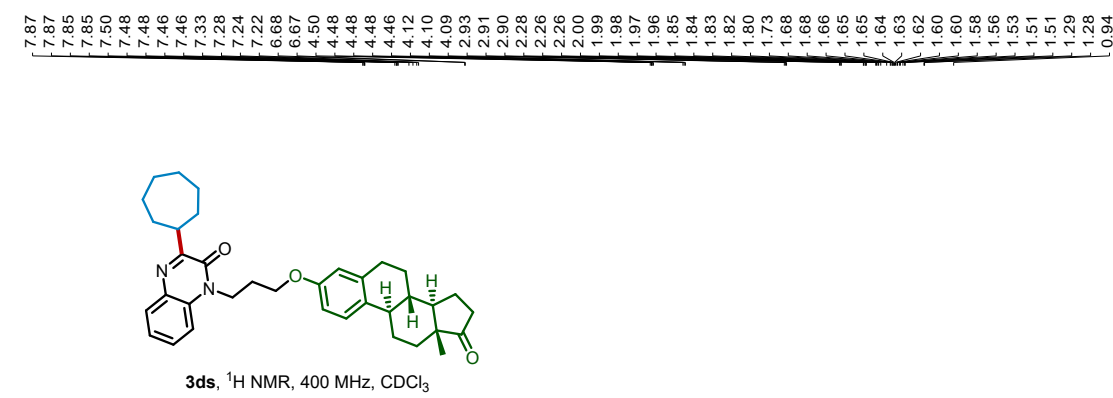
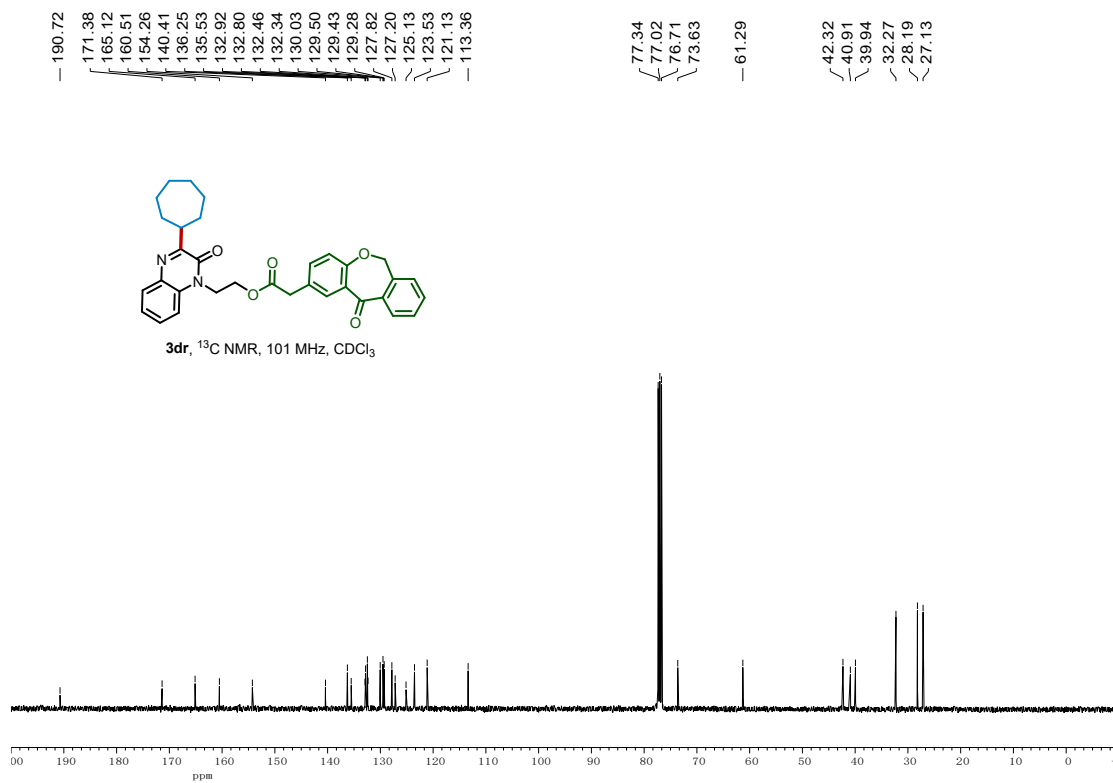


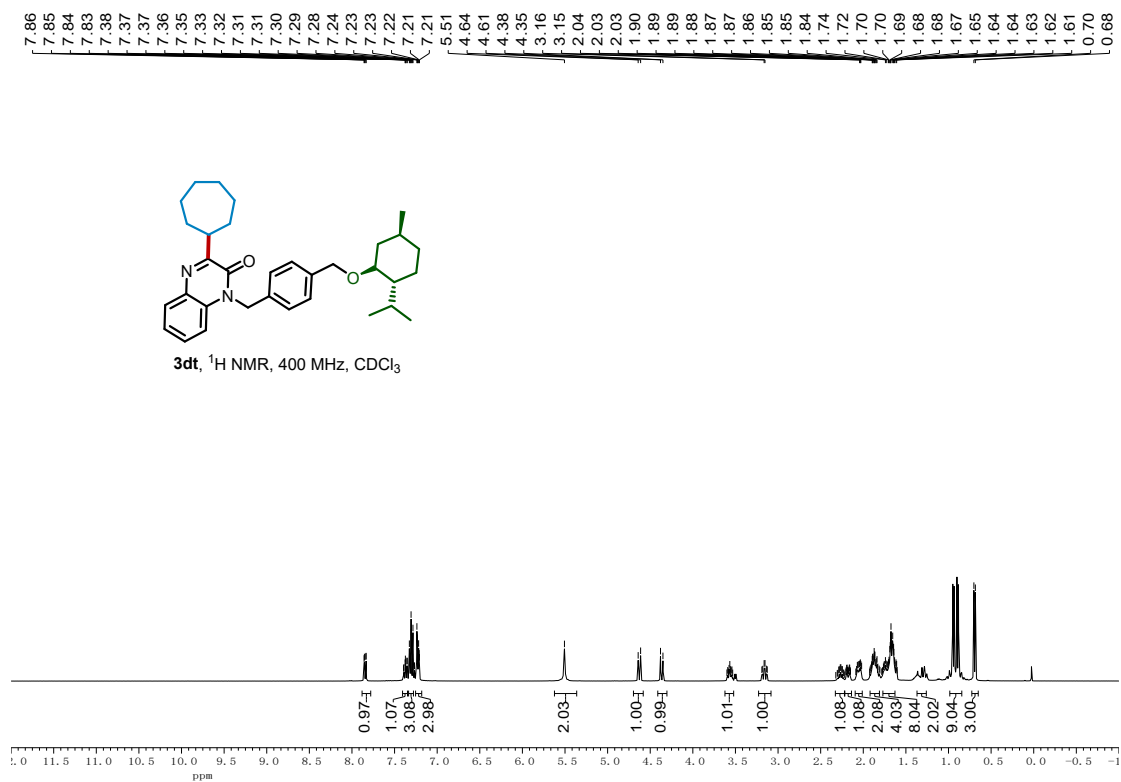
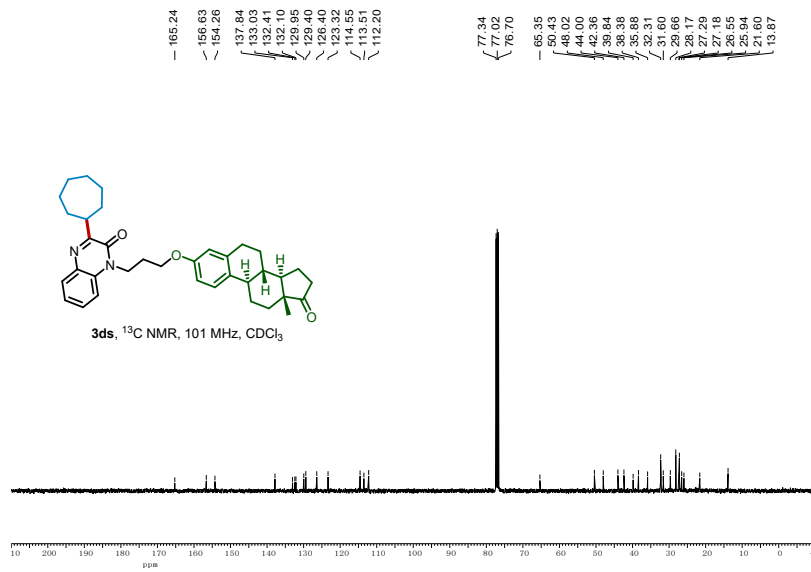
**3dq**,  $^{13}\text{C}$  NMR, 101 MHz,  $\text{CDCl}_3$

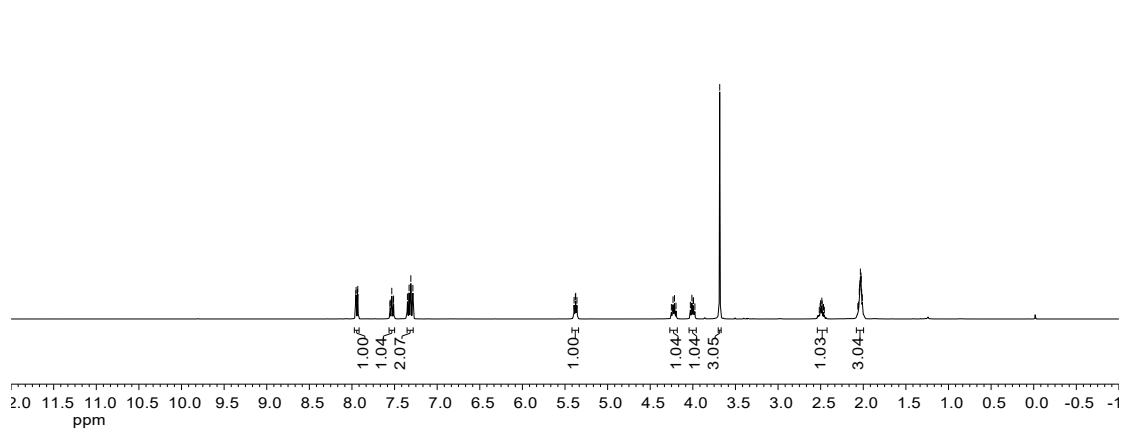
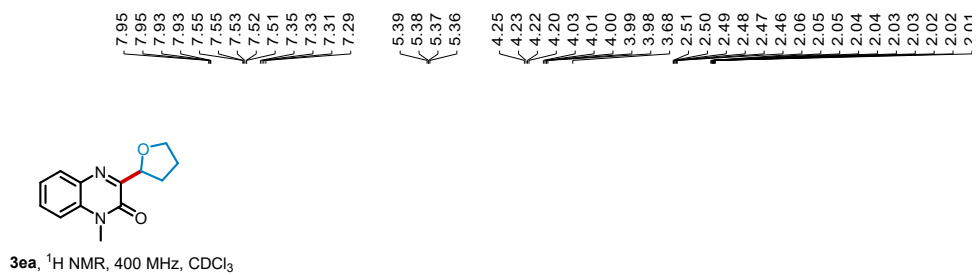
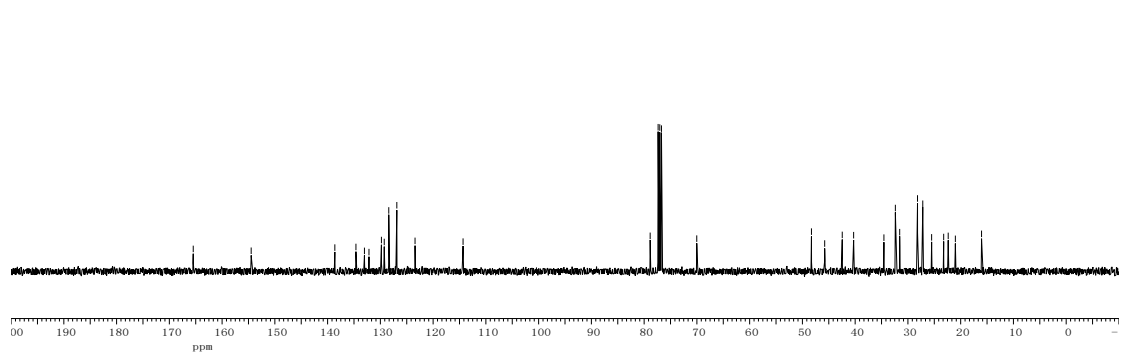
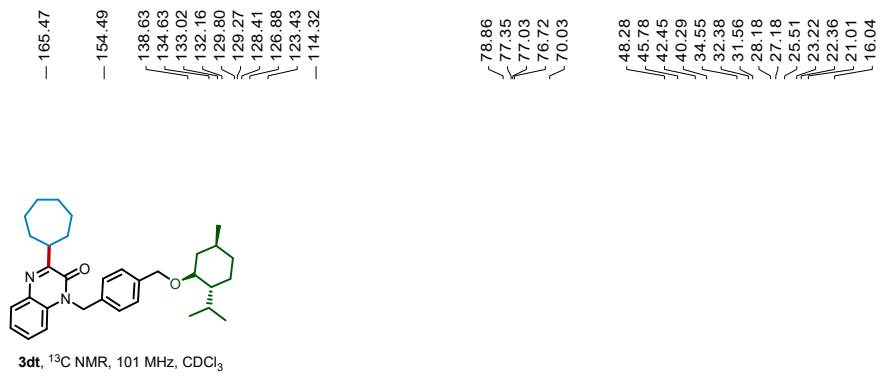




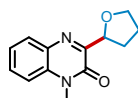




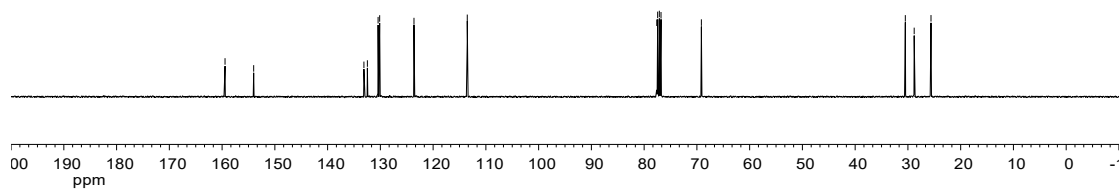




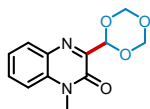
- 159.44  
 - 154.01  
 133.12  
 132.46  
 130.43  
 130.13  
 123.63  
 - 113.53  
 77.61  
 77.44  
 77.12  
 76.80  
 - 69.15  
 30.49  
 28.81  
 25.62



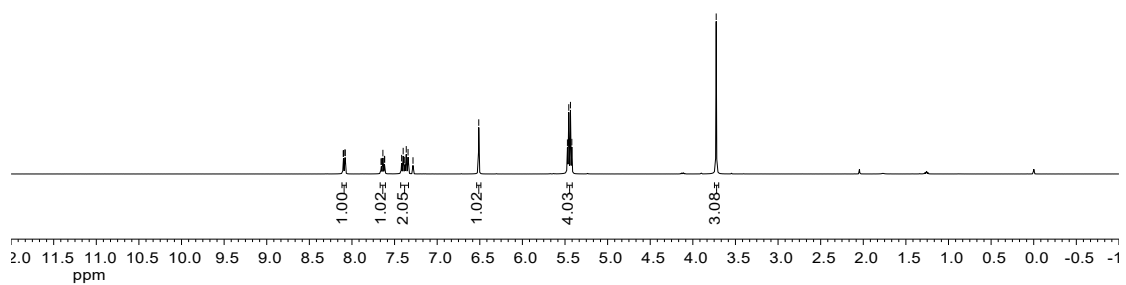
3ea,  $^{13}\text{C}$  NMR, 101 MHz,  $\text{CDCl}_3$

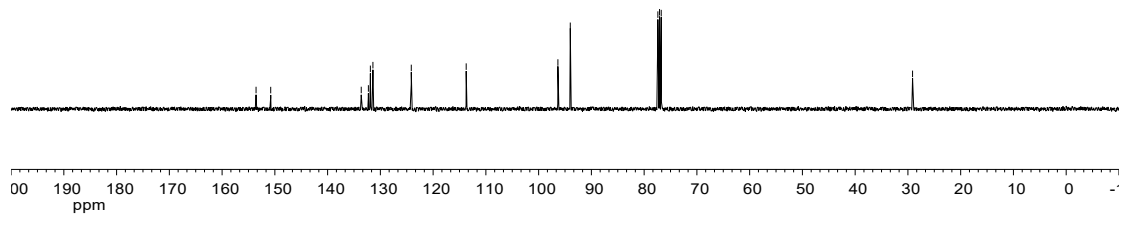
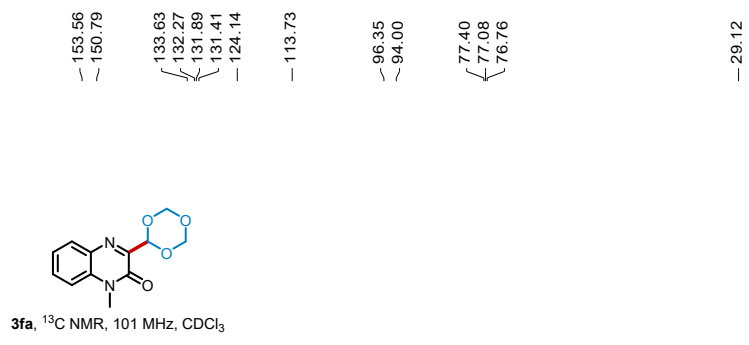


8.10  
 8.10  
 8.08  
 7.66  
 7.66  
 7.64  
 7.62  
 7.42  
 7.40  
 7.38  
 7.36  
 7.34  
 7.28  
 - 6.51  
 5.47  
 5.46  
 5.44  
 5.42  
 - 3.73

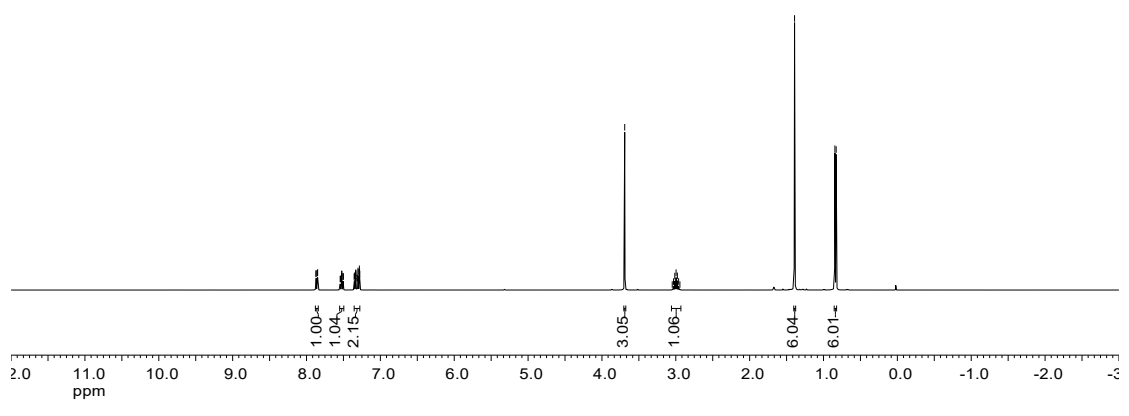
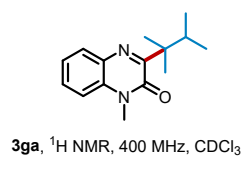


3fa,  $^1\text{H}$  NMR, 400 MHz,  $\text{CDCl}_3$

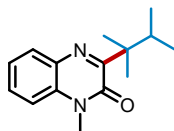




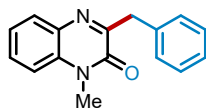
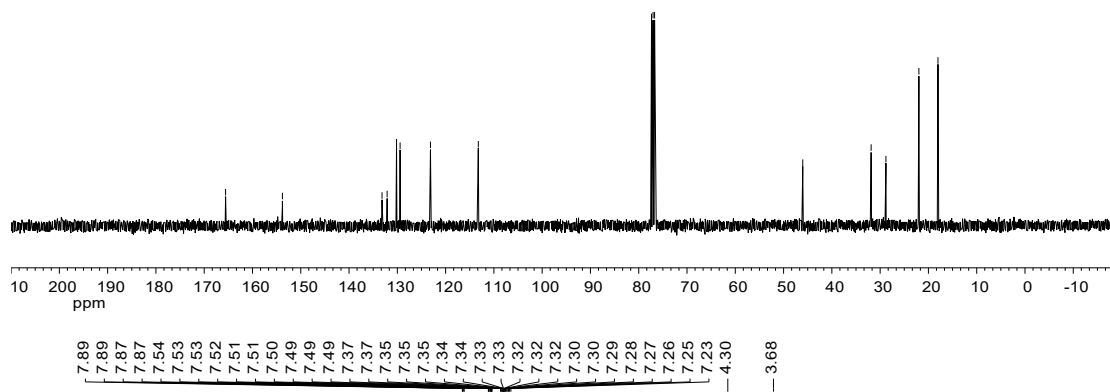
7.87  
 7.87  
 7.85  
 7.85  
 7.54  
 7.54  
 7.53  
 7.52  
 7.52  
 7.52  
 7.51  
 7.50  
 7.35  
 7.35  
 7.34  
 7.34  
 7.33  
 7.33  
 7.33  
 7.32  
 7.31  
 7.30  
 7.30  
 7.28  
 7.28  
 3.69  
 3.05  
 3.03  
 3.01  
 3.00  
 2.99  
 2.98  
 2.96  
 2.94  
 1.39  
 0.85  
 0.83



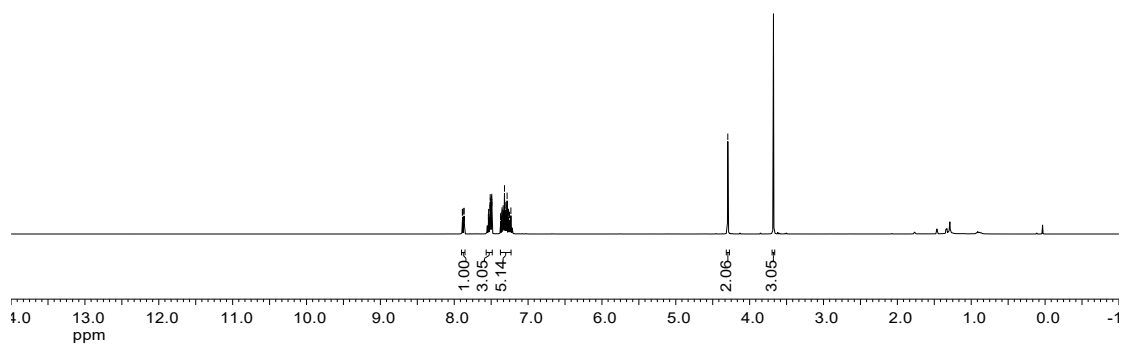
— 165.57  
 — 153.80  
 — 133.17  
 — 132.11  
 — 130.17  
 — 129.43  
 — 123.14  
 — 113.23  
 — 77.35  
 — 77.03  
 — 76.72  
 — 46.03  
 — 31.87  
 — 28.80  
 — 21.97  
 — 18.02



**3ga**,  $^{13}\text{C}$  NMR, 101 MHz,  $\text{CDCl}_3$



**3ha**,  $^1\text{H}$  NMR, 400 MHz,  $\text{CDCl}_3$

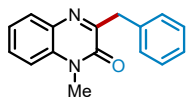


159.31  
154.71  
137.06  
133.34  
132.75  
129.95  
129.87  
129.54  
128.41  
126.60  
123.57  
113.56

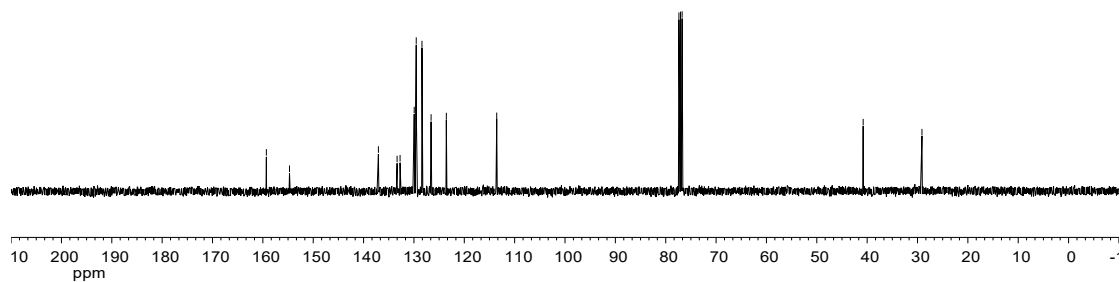
77.38  
77.06  
76.74

40.79

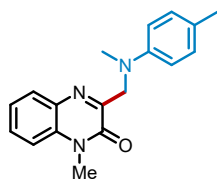
29.12



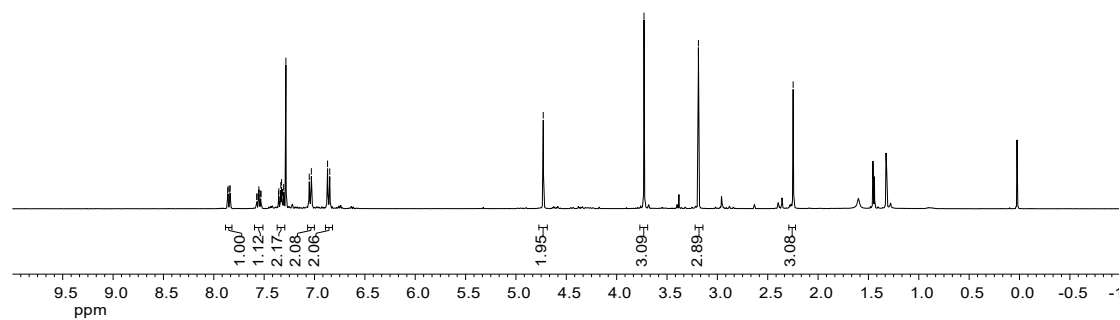
**3ha**,  $^{13}\text{C}$  NMR, 101 MHz,  $\text{CDCl}_3$



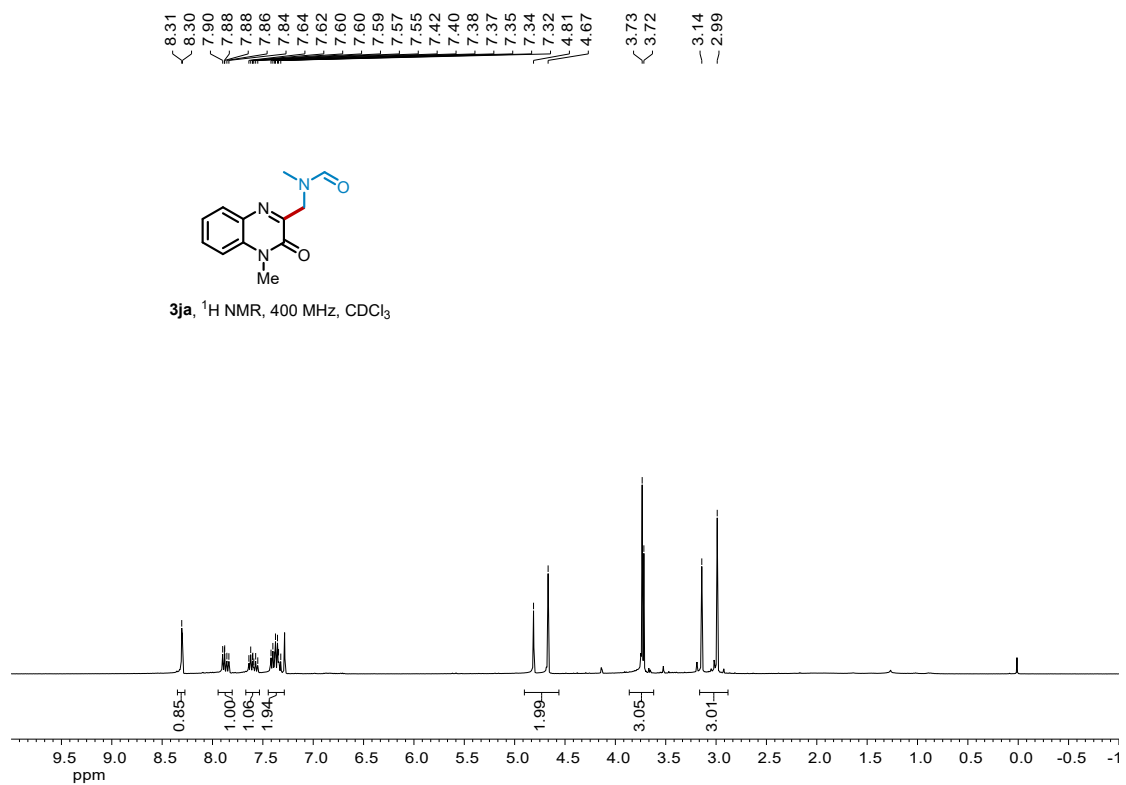
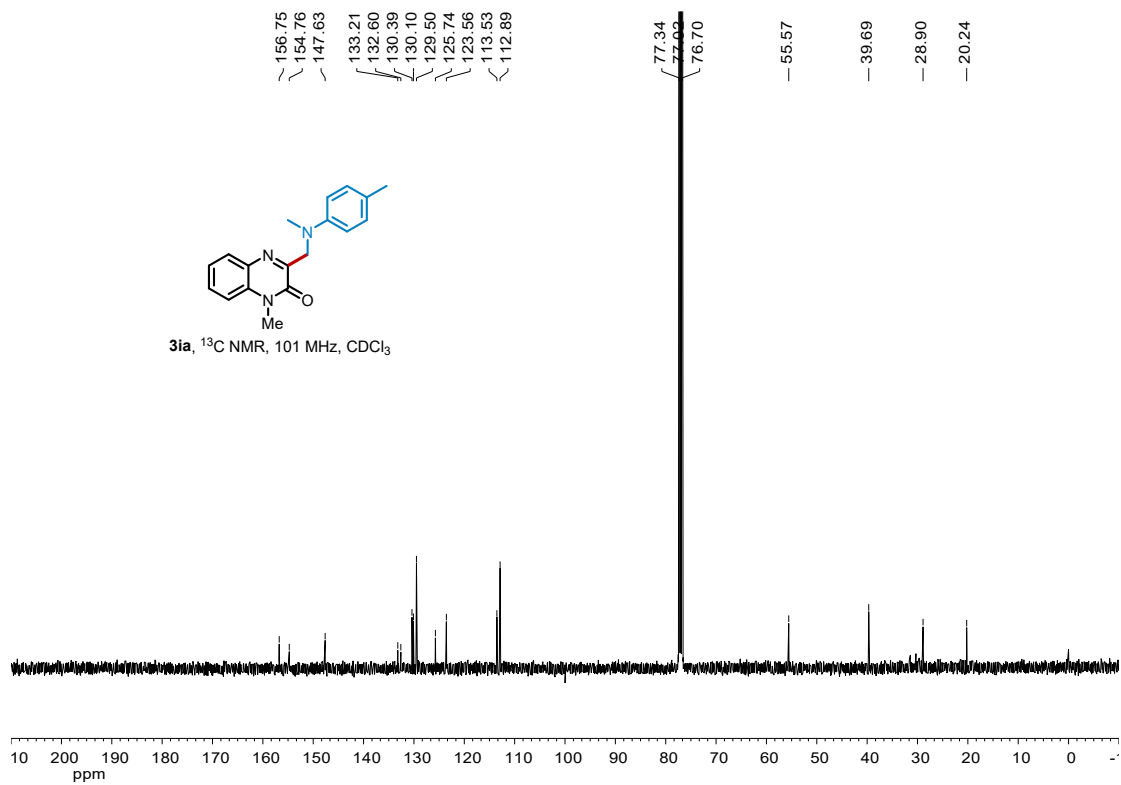
7.86  
7.86  
7.84  
7.84  
7.57  
7.57  
7.56  
7.55  
7.55  
7.54  
7.53  
7.36  
7.35  
7.33  
7.33  
7.32  
7.32  
7.31  
7.28  
7.05  
7.03  
6.87  
6.85  
4.73  
3.73  
3.19  
2.25



**3ia**,  $^1\text{H}$  NMR, 400 MHz,  $\text{CDCl}_3$





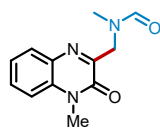


164.07  
163.18  
154.25  
153.98  
153.38  
133.29  
133.07  
132.53  
132.42  
130.91  
130.48  
130.28  
124.04  
123.71  
113.77  
113.62

77.36  
77.04  
76.72

51.29  
46.44

35.55  
30.71  
29.02  
28.87



3ja,  $^{13}\text{C}$  NMR, 101 MHz,  $\text{CDCl}_3$

