

## Supporting Information

### Catalyst-free visible-light-induced decarbonylative C-H alkylation of quinoxalin-2(1*H*)-one

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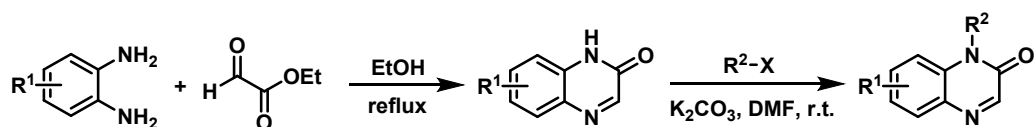
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## 1. General Information

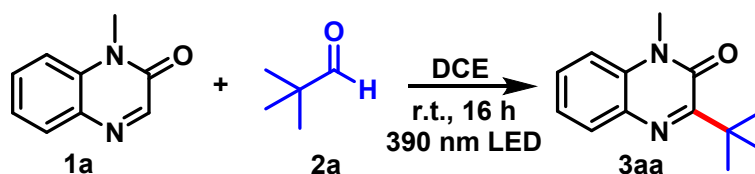
Unless otherwise noted, Reagents were purchased from commercial sources and were used as received.  $^1\text{H}$  and  $^{13}\text{C}$  Nuclear Magnetic Resonance (NMR) spectra were recorded on Bruker Avance 400 Ultrashield NMR spectrometers. Chemical shifts ( $\delta$ ) were given in parts per million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

## 2. Preparation of Quinoxalin-2(1H)-ones



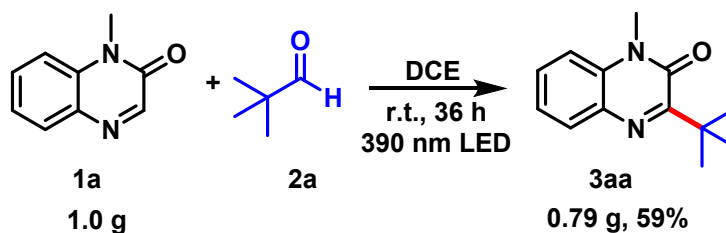
**Quinoxalin-2(1H)-one** was prepared from 1,2-phenylenediamines following the procedure of Cui and co-workers<sup>[1]</sup> on 5 mmol scale. To a solution of 1,2-phenylenediamines (5 mmol, 1.0 equiv.) in ethanol (40 mL) was added ethyl glyoxalate (6 mmol, 1.2 equiv.). The resultant reaction mixture was stirred at reflux until the raw material disappears. Then, the mixture was filtered and washed by ethanol. The solid was dried in *vacuo*. For alkylation, the corresponding halogenoalkane (1.6 equiv.) was added to a suspension of quinoxalinone (1.0 equiv.) and potassium carbonate (1.2 equiv.) in DMF (16 mL). The mixture was stirred at room temperature overnight. After complete reaction, brine was added, and then extracted three times with EtOAc. The combined organic layers were washed with a saturated solution of NH<sub>4</sub>Cl then brine, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, filtered and evaporated in *vacuo*. The residue was purified by column chromatography on silica gel to afford the desired product.

## 3. General Procedures for Rhotoredox Reactions



Quinoxalin-2(1*H*)-one (32 mg, 0.2 mmol, 1.0 equiv.), pivaldehyde (51.6 mg, 0.6 mmol, 3.0 equiv.) and DCE (2.0 mL) was added to a 10 mL oven-dried quartz tube equipped with magnetic stirring bar. The vessel placed 2 cm away from 390 nm LED (20 W). The reaction mixture was irradiated with for 16 h under air atmosphere. After irradiation, the reaction mixture was transferred to a 50 mL round-bottom flask and the solvent was concentrated in *vacuo*. The pure product was obtained by flash column chromatography on silica gel (petroleum ether/ ethyl acetate = 5/1).

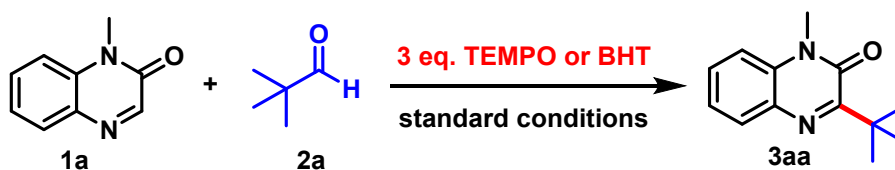
#### 4. Gram-scale reaction



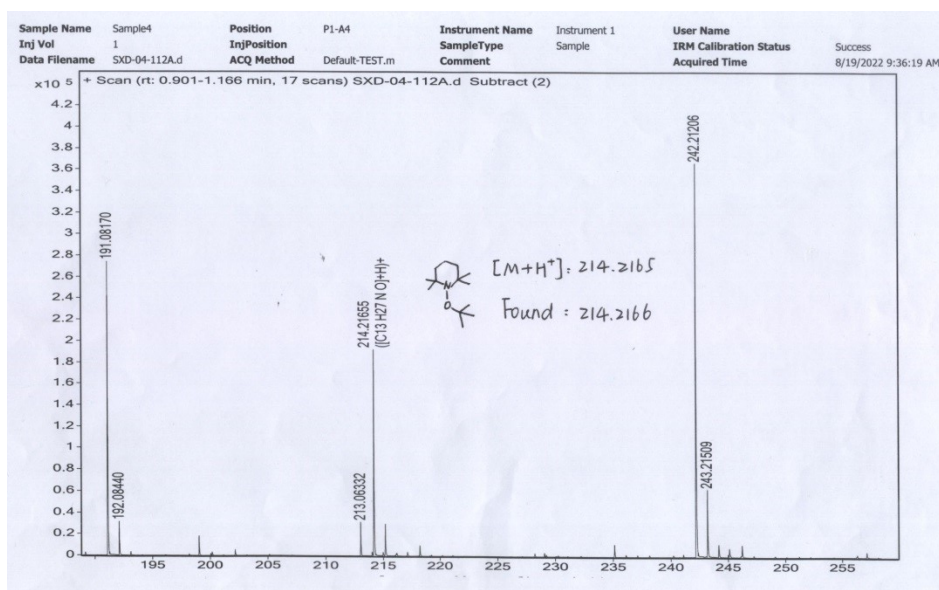
Quinoxalin-2(1*H*)-one (1.0 g, 6.2 mmol, 1.0 equiv.), pivaldehyde (1.6 g, 18.6 mmol, 3.0 equiv.) and DCE (60 mL) was added to a 100 mL round-bottom flask equipped with magnetic stirring bar. The vessel placed 2 cm away from 390 nm LED (20 W). The reaction mixture was irradiated with for 36 h under air atmosphere. After irradiation, the solvent was concentrated in *vacuo*. The pure product was obtained by flash column chromatography on silica gel (petroleum ether/ ethyl acetate = 5/1) give **3aa** as a white solid (0.79 g, 59% yield).

#### 5. Mechanistic Studies

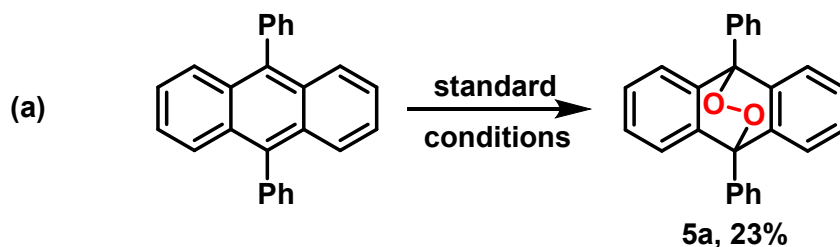
##### 5.1 Radical trapping experiment



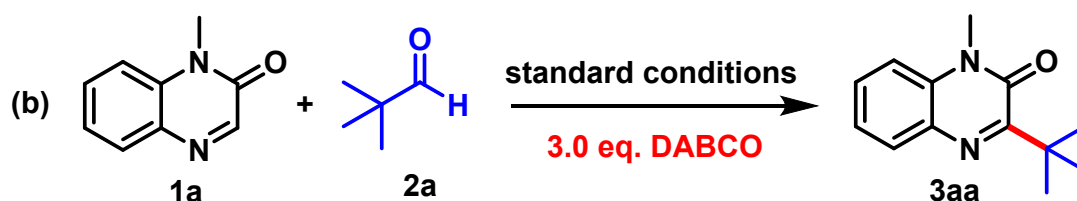
Quinoxalin-2(1*H*)-one (32 mg, 0.2 mmol, 1.0 equiv.), pivaldehyde (51.6 mg, 0.6 mmol, 3.0 equiv.), TEMPO (94 mg, 0.6 mmol, 3.0 equiv.) and DCE (2.0 mL) was added to a 10 mL oven-dried quartz tube equipped with magnetic stirring bar. The vessel placed 2 cm away from 390 nm LED (20 W). The reaction mixture was irradiated with for 16 h under air atmosphere. The reaction was almost suppressed. A similar procedure was conducted with BHT (133 mg, 0.6 mmol, 3.0 equiv.). The radical trapping product **4a** can be observed by HRMS.



## 5.2 Singlet oxygen trapping experiment



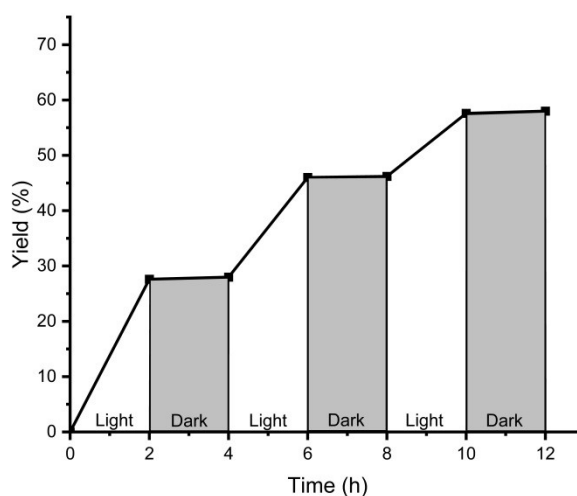
Quinoxalin-2(1*H*)-one (32 mg, 0.2 mmol, 1.0 equiv.), pivalaldehyde (51.6 mg, 0.6 mmol, 3.0 equiv.), 9,10-diphenylanthracene (66 mg, 0.2 mmol, 1.0 equiv.) and DCE (2.0 mL) was added to a 10 mL oven-dried quartz tube equipped with magnetic stirring bar. The vessel placed 2 cm away from 390 nm LED (20 W). The reaction mixture was irradiated with for 16 h under air atmosphere. The product **5a** was obtained.



Quinoxalin-2(1*H*)-one (32 mg, 0.2 mmol, 1.0 equiv.), pivaldehyde (51.6 mg, 0.6 mmol, 3.0 equiv.), DABCO (67.3 mg, 0.6 mmol, 3.0 equiv.) and DCE (2.0 mL) was added to a 10 mL oven-dried quartz tube equipped with magnetic stirring bar. The vessel placed 2 cm away from 390 nm LED (20 W). The reaction mixture was irradiated with for 16 h under air atmosphere. The reaction was almost suppressed.

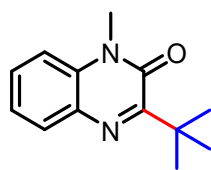
### 5.3 Light/dark experiment

Six standard reaction mixtures in 10 mL glass vials were charged with quinoxalin-2(1*H*)-one (32 mg, 0.2 mmol, 1.0 equiv.), pivaldehyde (51.6 mg, 0.6 mmol, 3.0 equiv.) and DCE (2.0 mL). The mixtures were stirred rapidly and irradiated with 390 nm LEDs (approximately 2 cm away from the light source) at room temperature. After 2 h, the 390 nm LEDs were turned off, and one vial was removed from the irradiation setup for analysis. The remaining five vials were stirred in the absence of light for an additional 2 h. Then, one tube was removed for analysis, and the 390 nm LEDs were turned back on to irradiate the remaining four reaction mixtures. After an additional 2 h of irradiation, the 390 nm LEDs were turned off, and one vial was removed for analysis. The remaining three vials were stirred in the absence of light for an additional 2 h. Then, a vial was removed for analysis, and the 390 nm LEDs were turned back on to irradiate the remaining two reaction mixtures. After 2 h, the 390 nm LEDs were turned off, and one vial was removed for analysis. The remaining one vial was stirred in the absence of light for an additional 2 h, then, it was analyzed. The yield was determined by <sup>1</sup>H NMR spectroscopy using dibromomethane as the internal standard.



## 6. Characterization Data for products

**3-(*tert*-butyl)-1-methylquinoxalin-2(1*H*)-one (3aa)**<sup>[2]</sup>

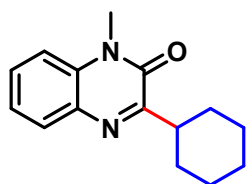


On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **3aa** as a white solid (33.7 mg, 78% yield).

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  7.83 (d, *J* = 7.9 Hz, 1H), 7.50 (t, *J* = 8.0 Hz, 1H), 7.33 – 7.25 (m, 2H), 3.67 (s, 3H), 1.49 (s, 9H).

**<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)**  $\delta$  165.29, 153.75, 133.33, 132.19, 130.12, 129.52, 123.18, 113.26, 77.37, 77.06, 76.74, 39.47, 28.76, 27.89.

**3-cyclohexyl-1-methylquinoxalin-2(1*H*)-one (3ab)**<sup>[3]</sup>

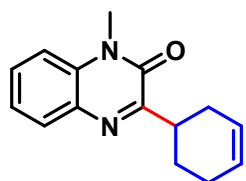


On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **3ab** as a white solid (32.5 mg, 67% yield).

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  7.83 (dd, *J* = 7.9, 1.5 Hz, 1H), 7.50 (t, *J* = 8.4, 7.3, 1.5 Hz, 1H), 7.35 – 7.26 (m, 2H), 3.70 (s, 3H), 3.38 – 3.29 (tt, 1H), 1.98 – 1.92 (m, 2H), 1.87 (dt, *J* = 12.9, 3.3 Hz, 2H), 1.79 – 1.74 (m, 1H), 1.63 – 1.53 (m, 2H), 1.52 – 1.42 (m, 2H), 1.36 – 1.25 (m, 1H).

**<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)**  $\delta$  164.28, 154.56, 132.90, 132.86, 129.77, 129.39, 123.40, 113.47, 40.78, 30.53, 29.07, 26.32, 26.16.

**3-(cyclohex-3-en-1-yl)-1-methylquinoxalin-2(1*H*)-one (3ac)**<sup>[4]</sup>



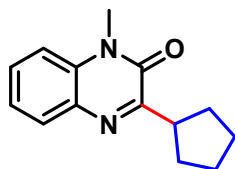
On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material

was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **3ac** as a white solid (23.1 mg, 48% yield).

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  7.84 (d,  $J$  = 8.0 Hz, 1H), 7.52 (t,  $J$  = 7.9 Hz, 1H), 7.35 – 7.26 (m, 2H), 5.78 (d,  $J$  = 7.8 Hz, 2H), 3.71 (s, 3H), 3.63 – 3.55 (m, 1H), 2.51 – 2.42 (m, 1H), 2.35 – 2.15 (m, 4H), 2.05 (d,  $J$  = 10.1 Hz, 1H), 1.85 – 1.71 (m, 2H).

**<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)**  $\delta$  163.69, 154.62, 132.89, 132.84, 129.85, 129.55, 126.55, 126.33, 123.47, 113.51, 77.38, 77.06, 76.75, 36.91, 29.09, 28.88, 26.87, 25.61.

### 3-cyclopentyl-1-methylquinoxalin-2(1H)-one (**3ad**)<sup>[2]</sup>

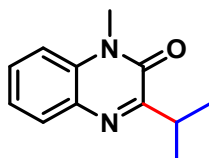


On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **3ad** as a white solid (22.4 mg, 49% yield).

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  7.82 (d,  $J$  = 7.9 Hz, 1H), 7.51 (t,  $J$  = 7.8 Hz, 1H), 7.36 – 7.25 (m, 2H), 3.70 (s, 4H), 2.06 (dq,  $J$  = 11.8, 5.6 Hz, 3H), 1.92 (dq,  $J$  = 13.4, 7.5 Hz, 3H), 1.82 (t,  $J$  = 5.5 Hz, 2H), 1.70 (td,  $J$  = 7.0, 3.7 Hz, 3H), 1.63 (q,  $J$  = 5.9 Hz, 1H).

**<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)**  $\delta$  163.73, 155.03, 132.96, 132.74, 129.76, 129.34, 123.41, 113.46, 77.38, 77.06, 76.75, 42.74, 30.87, 30.85, 29.07, 25.96, 25.94.

### 3-isopropyl-1-methylquinoxalin-2(1H)-one (**3ae**)<sup>[2]</sup>

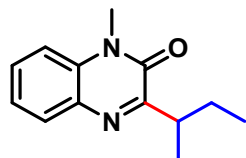


On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **3ae** as a white solid (18.2 mg, 45% yield).

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  7.85 (dd,  $J$  = 7.9, 1.6 Hz, 1H), 7.54 – 7.47 (m, 1H), 7.36 – 7.26 (m, 2H), 3.70 (s, 3H), 3.63 (p,  $J$  = 6.8 Hz, 1H), 1.33 (s, 3H), 1.31 (s, 3H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  165.01, 154.55, 133.00, 132.81, 129.84, 129.46, 123.43, 113.49, 77.38, 77.06, 76.74, 31.22, 29.06, 20.21.

**3-(*sec*-butyl)-1-methylquinoxalin-2(1*H*)-one (3af)<sup>[2]</sup>**

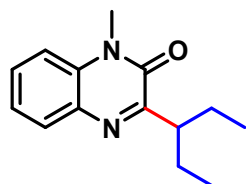


On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **3af** as a yellow oil (28.1 mg, 65% yield).

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.85 (d,  $J$  = 8.0 Hz, 1H), 7.52 (t,  $J$  = 7.8 Hz, 1H), 7.36 – 7.27 (m, 2H), 3.71 (s, 3H), 3.46 (q,  $J$  = 6.9 Hz, 1H), 1.93 (dt,  $J$  = 13.9, 7.1 Hz, 1H), 1.60 (dt,  $J$  = 14.4, 7.2 Hz, 1H), 1.29 (d,  $J$  = 6.9 Hz, 3H), 0.94 (t,  $J$  = 7.4 Hz, 3H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  164.58, 154.75, 132.90, 132.83, 129.82, 129.46, 123.42, 113.50, 77.38, 77.06, 76.74, 37.77, 29.11, 27.56, 17.89, 12.09.

**1-methyl-3-(pentan-3-yl)quinoxalin-2(1*H*)-one (3ag)<sup>[2]</sup>**



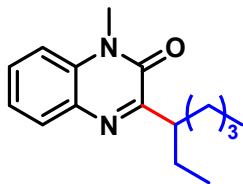
On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **3ag** as a yellow oil (24.4 mg, 53% yield).

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.86 (d,  $J$  = 8.0 Hz, 1H), 7.52 (t,  $J$  = 7.8 Hz, 1H), 7.31 (dt,  $J$  = 17.0, 8.5 Hz, 2H), 3.71 (s, 3H), 3.39 – 3.30 (m, 1H), 1.87 (dq,  $J$  = 15.0, 7.5 Hz, 2H), 1.75 – 1.65 (m, 2H), 0.88 (t,  $J$  = 7.4 Hz, 6H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  163.84, 155.11, 132.80, 129.83, 129.45, 123.39, 113.49, 77.37, 77.06, 76.74, 44.63, 29.14, 25.75, 12.00.

**3-(heptan-3-yl)-1-methylquinoxalin-2(1*H*)-one (3ah)<sup>[4]</sup>**



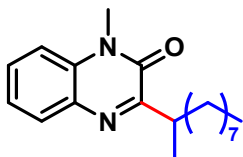


On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **3ah** as a yellow oil (24.8 mg, 48% yield).

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  7.79 (d,  $J$  = 8.1 Hz, 1H), 7.48 – 7.42 (m, 1H), 7.30 – 7.20 (m, 2H), 3.64 (s, 3H), 3.40 – 3.28 (m, 1H), 1.85 – 1.72 (m, 2H), 1.63 – 1.53 (m, 2H), 1.27 – 1.17 (m, 4H), 0.80 (dt,  $J$  = 9.6, 7.1 Hz, 6H).

**<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)**  $\delta$  164.13, 155.10, 132.85, 129.85, 129.44, 123.39, 113.50, 77.38, 77.06, 76.74, 43.14, 32.78, 29.82, 29.17, 26.24, 22.95, 14.06, 12.06.

### 3-(decan-2-yl)-1-methylquinoxalin-2(1H)-one (**3ai**)



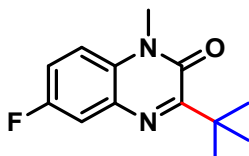
On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **3ai** as a yellow oil (30.0 mg, 50% yield).

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  7.85 (d,  $J$  = 8.0 Hz, 1H), 7.51 (t,  $J$  = 7.8 Hz, 1H), 7.36 – 7.27 (m, 2H), 3.70 (s, 3H), 3.52 (q,  $J$  = 6.9 Hz, 1H), 1.26 (dd,  $J$  = 12.5, 6.7 Hz, 17H), 0.86 (t,  $J$  = 6.7 Hz, 3H).

**<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)**  $\delta$  164.82, 154.73, 132.88, 129.83, 129.44, 123.41, 113.49, 77.38, 77.06, 76.74, 36.23, 34.73, 31.92, 29.82, 29.57, 29.33, 29.11, 27.62, 22.70, 18.30, 14.14.

**HRMS (ESI):** Calcd for C<sub>19</sub>H<sub>29</sub>N<sub>2</sub>O [M+H]<sup>+</sup>: 301.2274; found: 301.2273.

### 3-(*tert*-butyl)-6-fluoro-1-methylquinoxalin-2(1H)-one (**4aa**)<sup>[2]</sup>

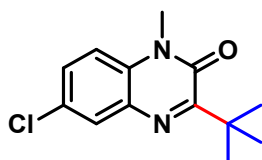


On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **4aa** as a white solid (32.3 mg, 69% yield).

$^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.53 (d,  $J = 8.9$  Hz, 1H), 7.26 (d,  $J = 5.8$  Hz, 1H), 7.23 (d,  $J = 5.2$  Hz, 1H), 3.67 (s, 3H), 1.48 (s, 9H).

$^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  166.91, 158.54 (d,  $J = 243.1$  Hz), 153.39, 132.83, 130.03, 117.19 (d,  $J = 23.9$  Hz), 115.51 (d,  $J = 22.2$  Hz), 114.29 (d,  $J = 8.8$  Hz), 39.69, 29.05, 27.83.

**3-(tert-butyl)-6-chloro-1-methylquinoxalin-2(1H)-one (4ab)**<sup>[2]</sup>

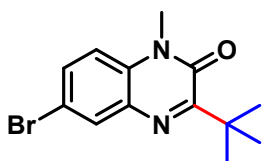


On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **4ab** as a white solid (35.6 mg, 71% yield).

$^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.84 (s, 1H), 7.45 (d,  $J = 8.9$  Hz, 1H), 7.20 (d,  $J = 8.9$  Hz, 1H), 3.65 (s, 3H), 1.47 (s, 9H).

$^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  166.80, 153.39, 132.74, 132.07, 129.48, 128.47, 114.40, 39.69, 28.96, 27.80.

**6-bromo-3-(tert-butyl)-1-methylquinoxalin-2(1H)-one (4ac)**<sup>[2]</sup>

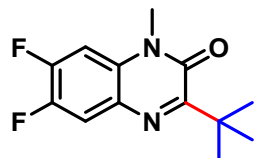


On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **4ac** as a white solid (37.8 mg, 64% yield).

$^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.99 (d,  $J = 2.3$  Hz, 1H), 7.58 (dd,  $J = 8.9, 2.3$  Hz, 1H), 7.14 (d,  $J = 8.9$  Hz, 1H), 3.65 (s, 3H), 1.47 (s, 9H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  166.76, 153.38, 133.05, 132.52, 132.23, 115.70, 114.73, 77.38, 77.06, 76.74, 39.70, 28.95, 27.82.

**3-(*tert*-butyl)-6,7-difluoro-1-methylquinoxalin-2(1*H*)-one (4ad)<sup>[4]</sup>**

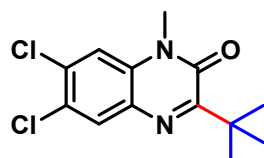


On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **4ad** as a white solid (34.3 mg, 68% yield).

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.67 – 7.57 (dd, 1H), 7.06 (dd,  $J = 12.3, 5.8$  Hz, 1H), 3.62 (s, 3H), 1.46 (s, 9H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  165.95, 153.30, 152.26 (d,  $J = 14.7$  Hz), 149.76 (d,  $J = 14.5$  Hz), 146.46 (d,  $J = 232.2$  Hz), 129.53 (d,  $J = 209.9$  Hz), 117.70 (d,  $J = 17.7$  Hz), 101.84 (d,  $J = 22.9$  Hz), 39.63, 29.31, 27.78.

**3-(*tert*-butyl)-6,7-dichloro-1-methylquinoxalin-2(1*H*)-one (4ae)<sup>[2]</sup>**

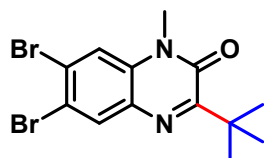


On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **4ae** as a white solid (41.2 mg, 72% yield).

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.91 (s, 1H), 7.34 (s, 1H), 3.62 (s, 3H), 1.46 (s, 9H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  166.98, 153.09, 133.45, 132.79, 131.30, 130.94, 126.87, 114.75, 77.38, 77.06, 76.74, 39.79, 29.06, 27.79.

**6,7-dibromo-3-(*tert*-butyl)-1-methylquinoxalin-2(1*H*)-one (4af)<sup>[5]</sup>**



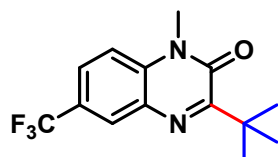
On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **4af** as a white solid (47.1 mg, 63% yield).

$^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.06 (s, 1H), 7.52 (s, 1H), 3.61 (s, 3H), 1.45 (s, 9H).

$^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  167.15, 153.04, 134.03, 133.33, 131.96, 125.58, 118.17, 117.90, 77.37, 77.06, 76.74, 39.82, 29.00, 27.76.

HRMS (ESI): Calcd for  $\text{C}_{13}\text{H}_{15}\text{Br}_2\text{N}_2\text{O}$   $[\text{M}+\text{H}]^+$ : 372.9546; found: 372.9546.

### 3-(*tert*-butyl)-1-methyl-6-(trifluoromethyl)quinoxalin-2(1H)-one (**4ag**)<sup>[2]</sup>

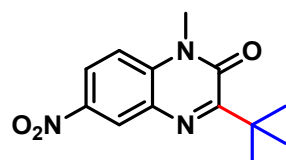


On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **4ag** as a white solid (41.4 mg, 73% yield).

$^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.13 (s, 1H), 7.72 (d,  $J$  = 10.9 Hz, 1H), 7.36 (d,  $J$  = 8.7 Hz, 1H), 3.70 (s, 3H), 1.49 (s, 9H).

$^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  167.14, 153.51, 135.71, 131.58, 127.94 – 125.89 (m), 125.52 (d,  $J$  = 33.6 Hz), 122.56, 113.91, 39.76, 29.06, 27.79.

### 3-(*tert*-butyl)-1-methyl-6-nitroquinoxalin-2(1H)-one (**4ah**)<sup>[4]</sup>

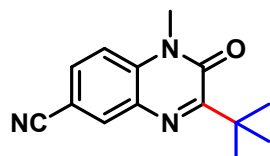


On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **4ah** as a white solid (49.6 mg, 95% yield).

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)** δ 8.71 (d, *J* = 2.6 Hz, 1H), 8.36 (dd, *J* = 9.2, 2.6 Hz, 1H), 7.37 (d, *J* = 9.2 Hz, 1H), 3.73 (s, 3H), 1.49 (s, 9H).

**<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)** δ 168.10, 153.18, 143.07, 138.07, 131.30, 125.84, 124.18, 113.86, 39.93, 29.39, 27.71.

**3-(*tert*-butyl)-1-methyl-2-oxo-1,2-dihydroquinoxaline-6-carbonitrile (4ai)<sup>[2]</sup>**

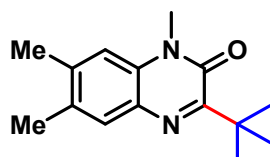


On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **4ai** as a white solid (34.3 mg, 71% yield).

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)** δ 8.16 (s, 1H), 7.74 (dd, *J* = 8.7, 1.9 Hz, 1H), 7.34 (d, *J* = 8.6 Hz, 1H), 3.69 (s, 3H), 1.47 (s, 9H).

**<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)** δ 167.75, 153.24, 136.69, 134.48, 132.18, 131.79, 118.21, 114.42, 106.65, 77.38, 77.06, 76.74, 39.87, 29.11, 27.74.

**3-(*tert*-butyl)-1,6,7-trimethylquinoxalin-2(1H)-one (4aj)<sup>[2]</sup>**

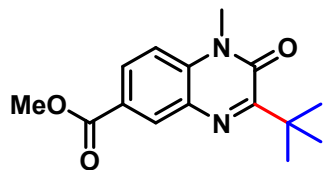


On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **4aj** as a white solid (23.4 mg, 48% yield).

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)** δ 7.59 (s, 1H), 7.02 (s, 1H), 3.64 (s, 3H), 2.40 (s, 3H), 2.34 (s, 3H), 1.47 (s, 9H).

**<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)** δ 164.05, 153.89, 139.16, 132.03, 131.32, 130.61, 130.22, 113.90, 77.38, 77.06, 76.74, 39.30, 28.68, 27.95, 20.52, 19.09.

**methyl 3-(*tert*-butyl)-1-methyl-2-oxo-1,2-dihydroquinoxaline-6-carboxylate (4ak)<sup>[4]</sup>**

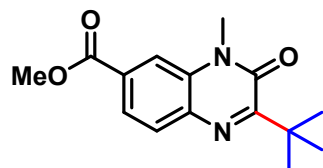


On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **4ak** as a white solid (44.4 mg, 81% yield).

$^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.96 (d,  $J$  = 11.6 Hz, 2H), 7.87 (d,  $J$  = 8.2 Hz, 1H), 3.98 (s, 3H), 3.72 (s, 3H), 1.49 (s, 9H).

$^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  168.03, 166.35, 153.51, 134.88, 133.16, 130.51, 130.17, 123.94, 115.08, 77.38, 77.06, 76.74, 52.61, 39.88, 29.01, 27.84.

**methyl 2-(tert-butyl)-4-methyl-3-oxo-3,4-dihydroquinoxaline-6-carboxylate (4al)<sup>[2]</sup>**

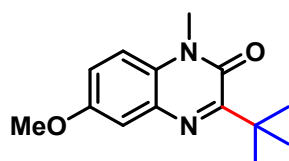


On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **4al** as a white solid (48.2 mg, 88% yield).

$^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.52 (t,  $J$  = 1.5 Hz, 1H), 8.16 (dt,  $J$  = 8.8, 1.5 Hz, 1H), 7.31 (d,  $J$  = 8.7 Hz, 1H), 3.96 (s, 3H), 3.69 (s, 3H), 1.48 (s, 9H).

$^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  166.32, 153.59, 136.72, 131.98, 131.55, 130.38, 125.10, 113.36, 77.38, 77.06, 76.74, 52.31, 39.66, 29.07, 27.83.

**3-(tert-butyl)-6-methoxy-1-methylquinoxalin-2(1H)-one (4am)<sup>[2]</sup>**



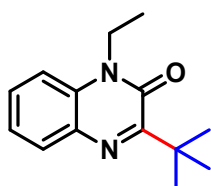
On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to

give **4am** as a white solid (34.0 mg, 69% yield).

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  7.31 (d,  $J$  = 2.8 Hz, 1H), 7.19 (d,  $J$  = 9.1 Hz, 1H), 7.13 (dd,  $J$  = 9.1, 2.8 Hz, 1H), 3.90 (s, 3H), 3.66 (s, 3H), 1.49 (s, 9H).

**<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)**  $\delta$  165.94, 155.75, 153.48, 132.93, 127.63, 118.77, 114.23, 111.58, 77.38, 77.06, 76.74, 55.82, 39.55, 28.93, 27.93.

### 3-(*tert*-butyl)-1-ethylquinoxalin-2(1*H*)-one (**4an**)<sup>[4]</sup>

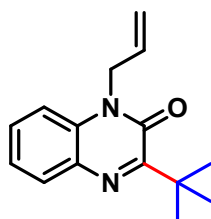


On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **4an** as a white solid (34.5 mg, 75% yield).

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  7.84 (d,  $J$  = 7.8 Hz, 1H), 7.50 (t,  $J$  = 7.8 Hz, 1H), 7.29 (t,  $J$  = 7.8 Hz, 2H), 4.29 (q,  $J$  = 7.2 Hz, 2H), 1.49 (s, 9H), 1.37 (t,  $J$  = 7.1 Hz, 3H).

**<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)**  $\delta$  165.32, 153.16, 132.49, 132.26, 130.38, 129.47, 122.95, 113.10, 77.38, 77.06, 76.74, 39.43, 37.00, 27.93, 12.46.

### 1-allyl-3-(*tert*-butyl)quinoxalin-2(1*H*)-one (**4ao**)<sup>[4]</sup>

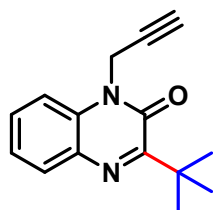


On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **4ao** as a white solid (37.3 mg, 77% yield).

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  7.84 (d,  $J$  = 8.0 Hz, 1H), 7.50 – 7.43 (m, 1H), 7.29 (t,  $J$  = 7.6 Hz, 1H), 7.23 (d,  $J$  = 8.2 Hz, 1H), 6.00 – 5.87 (m, 1H), 5.25 (d,  $J$  = 10.6 Hz, 1H), 5.14 (d,  $J$  = 17.2 Hz, 1H), 4.88 (d,  $J$  = 5.1 Hz, 2H), 1.49 (s, 9H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  165.37, 153.21, 132.57, 132.35, 131.00, 130.20, 129.44, 123.17, 117.72, 113.81, 77.38, 77.06, 76.74, 44.22, 39.50, 27.92.

**3-(*tert*-butyl)-1-(prop-2-yn-1-yl)quinoxalin-2(1*H*)-one (4ap)<sup>[4]</sup>**

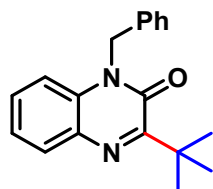


On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **4ap** as a white solid (37.4 mg, 78% yield).

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.85 (d,  $J$  = 8.0 Hz, 1H), 7.53 (t,  $J$  = 7.8 Hz, 1H), 7.42 (d,  $J$  = 8.4 Hz, 1H), 7.34 (t,  $J$  = 7.5 Hz, 1H), 5.03 (s, 2H), 2.28 (s, 1H), 1.49 (s, 9H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  165.26, 152.64, 132.39, 131.89, 130.28, 129.65, 123.60, 113.77, 77.38, 77.06, 76.74, 72.93, 39.62, 31.16, 27.92.

**1-benzyl-3-(*tert*-butyl)quinoxalin-2(1*H*)-one (4aq)<sup>[5]</sup>**



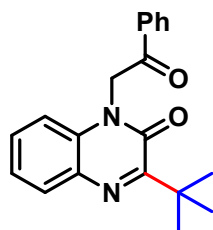
On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **4aq** as a white solid (52.6 mg, 90% yield).

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.83 (d,  $J$  = 7.9 Hz, 1H), 7.38 – 7.27 (m, 4H), 7.20 (dd,  $J$  = 17.8, 9.2 Hz, 4H), 5.48 (s, 2H), 1.53 (s, 9H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  165.50, 153.70, 135.63, 132.70, 132.45, 130.23, 129.52, 128.90, 127.54, 126.78, 123.25, 114.07, 77.38, 77.06, 76.74, 45.54, 39.58, 27.97.

**3-(*tert*-butyl)-1-(2-oxo-2-phenylethyl)quinoxalin-2(1*H*)-one (4ar)<sup>[4]</sup>**



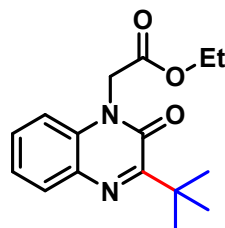


On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **4ar** as a white solid (45.5 mg, 71% yield).

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  8.08 (d,  $J$  = 7.2 Hz, 2H), 7.87 (d,  $J$  = 7.9 Hz, 1H), 7.66 (t,  $J$  = 7.5 Hz, 1H), 7.54 (t,  $J$  = 7.8 Hz, 2H), 7.42 – 7.37 (m, 1H), 7.29 (t,  $J$  = 7.6 Hz, 1H), 6.92 (d,  $J$  = 8.3 Hz, 1H), 5.72 (s, 2H), 1.50 (s, 9H).

**<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)**  $\delta$  191.59, 165.01, 153.41, 134.66, 134.24, 132.79, 132.39, 130.41, 129.62, 129.02, 128.18, 123.39, 113.16, 77.38, 77.06, 76.74, 48.28, 39.51, 27.91.

**ethyl 2-(3-(*tert*-butyl)-2-oxoquinoxalin-1(2*H*)-yl)acetate (4as)<sup>[4]</sup>**

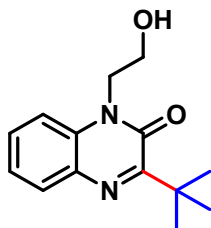


On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **4as** as a white solid (47.3 mg, 82% yield).

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  7.85 (d,  $J$  = 8.0 Hz, 1H), 7.49 – 7.43 (m, 1H), 7.33 – 7.28 (m, 1H), 7.03 (d,  $J$  = 8.3 Hz, 1H), 5.00 (s, 2H), 4.24 (q,  $J$  = 7.1 Hz, 2H), 1.49 (s, 9H), 1.27 (t,  $J$  = 7.1 Hz, 3H).

**<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)**  $\delta$  167.38, 165.12, 153.23, 132.52, 132.28, 130.45, 129.68, 123.49, 112.71, 77.38, 77.06, 76.74, 61.93, 43.33, 39.49, 27.86, 14.11.

**3-(*tert*-butyl)-1-(2-hydroxyethyl)quinoxalin-2(1*H*)-one (4at)**



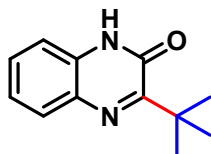
On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **4at** as a colorless oil (24.6 mg, 50% yield).

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  7.97 (d,  $J$  = 8.1 Hz, 1H), 7.75 (d,  $J$  = 8.2 Hz, 1H), 7.62 – 7.56 (m, 1H), 7.56 – 7.50 (m, 1H), 4.72 – 4.68 (m, 2H), 4.09 – 4.04 (m, 2H), 1.51 (s, 9H).

**<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)**  $\delta$  156.19, 155.89, 138.78, 138.22, 129.16, 128.81, 126.52, 125.98, 77.38, 77.06, 76.74, 68.75, 62.14, 38.32, 28.25.

**HRMS (ESI):** Calcd for C<sub>14</sub>H<sub>19</sub>N<sub>2</sub>O<sub>2</sub> [M+H]<sup>+</sup>: 247.1441; found: 247.1439.

### 3-(*tert*-butyl)quinoxalin-2(1H)-one (**4au**)<sup>[4]</sup>

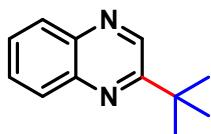


On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **4au** as a white solid (22.6 mg, 56% yield).

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  12.55 (s, 1H), 7.84 (d,  $J$  = 8.2 Hz, 1H), 7.50 – 7.43 (m, 1H), 7.35 – 7.28 (m, 2H), 1.55 (s, 9H).

**<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)**  $\delta$  165.78, 156.09, 132.39, 131.33, 129.66, 129.20, 123.81, 115.09, 77.38, 77.06, 76.74, 39.31, 27.84.

### 2-(*tert*-butyl)quinoxaline (**4av**)<sup>[6]</sup>



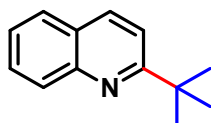
On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material

was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **4av** as a colorless oil (34.3 mg, 92% yield).

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)** δ 8.99 (d, *J* = 6.2 Hz, 1H), 8.16 – 7.99 (m, 2H), 7.86 – 7.62 (m, 2H), 1.52 (d, *J* = 7.7 Hz, 9H).

**<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)** δ 163.73, 143.43, 141.64, 140.76, 129.67, 129.30, 128.90, 37.27, 29.76.

#### 2-(*tert*-butyl)quinolone (**4aw**)<sup>[7]</sup>

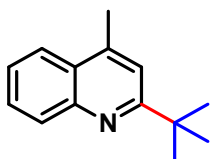


On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **4aw** as a colorless oil (30.4 mg, 82% yield).

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)** δ 8.06 (d, *J* = 8.5 Hz, 2H), 7.75 (d, *J* = 8.1 Hz, 1H), 7.66 (t, *J* = 7.6 Hz, 1H), 7.52 (d, *J* = 8.6 Hz, 1H), 7.46 (t, *J* = 7.4 Hz, 1H), 1.47 (s, 9H).

**<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)** δ 169.29, 147.45, 135.90, 129.42, 129.02, 127.25, 126.48, 125.65, 118.26, 77.38, 77.06, 76.74, 38.16, 30.18.

#### 2-(*tert*-butyl)-4-methylquinoline (**4ax**)<sup>[8]</sup>

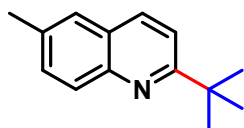


On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **4ax** as a colorless oil (24.3 mg, 61% yield).

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)** δ 8.06 (d, *J* = 8.1 Hz, 1H), 7.93 (d, *J* = 8.3 Hz, 1H), 7.65 (t, *J* = 7.7 Hz, 1H), 7.48 (t, *J* = 7.6 Hz, 1H), 7.35 (s, 1H), 2.68 (s, 3H), 1.45 (s, 9H).

**<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)** δ 168.97, 147.33, 143.66, 129.95, 128.73, 126.58, 125.42, 123.41, 118.93, 77.38, 77.06, 76.74, 37.95, 30.16, 18.99.

**2-(*tert*-butyl)-6-methylquinoline (4ay)**<sup>[7]</sup>

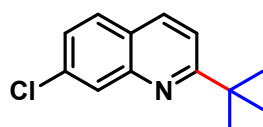


On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **4ay** as a colorless oil (37.1 mg, 93% yield).

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  7.96 (t,  $J$  = 8.1 Hz, 2H), 7.48 (t,  $J$  = 9.3 Hz, 3H), 2.51 (s, 3H), 1.46 (s, 9H).

**<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)**  $\delta$  168.35, 146.00, 135.31, 135.27, 131.24, 129.07, 126.46, 126.15, 118.21, 77.38, 77.06, 76.74, 38.02, 30.21, 21.52.

**2-(*tert*-butyl)-7-chloroquinoline (4az)**<sup>[9]</sup>

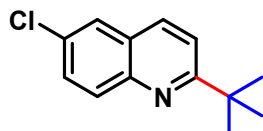


On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **4az** as a colorless oil (39.9 mg, 91% yield).

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  8.07 (s, 1H), 8.03 (d,  $J$  = 8.7 Hz, 1H), 7.68 (d,  $J$  = 8.7 Hz, 1H), 7.51 (d,  $J$  = 8.6 Hz, 1H), 7.42 (dd,  $J$  = 8.7, 2.1 Hz, 1H), 1.45 (s, 9H).

**<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)**  $\delta$  170.48, 147.84, 135.68, 134.79, 128.50, 128.44, 126.65, 124.81, 118.46, 77.38, 77.06, 76.74, 38.27, 30.08.

**2-(*tert*-butyl)-6-chloroquinoline (4ba)**<sup>[10]</sup>

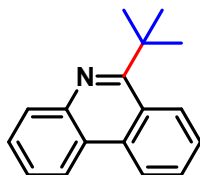


On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **4ba** as a colorless oil (41.7 mg, 95% yield).

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)** δ 8.00 – 7.95 (m, 2H), 7.73 (s, 1H), 7.59 (d, *J* = 9.0 Hz, 1H), 7.53 (d, *J* = 8.7 Hz, 1H), 1.45 (s, 9H).

**<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)** δ 169.66, 145.83, 135.00, 131.22, 131.07, 129.85, 127.05, 125.92, 119.15, 77.38, 77.06, 76.74, 38.22, 30.09.

#### 6-(*tert*-butyl)phenanthridine (**4bb**)<sup>[2]</sup>

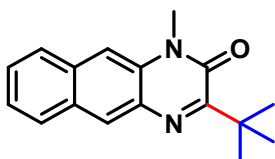


On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **4bb** as a white solid (34.3 mg, 73% yield).

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)** δ 8.66 (d, *J* = 8.3 Hz, 1H), 8.61 (d, *J* = 8.5 Hz, 1H), 8.50 (d, *J* = 8.2 Hz, 1H), 8.12 (d, *J* = 8.0 Hz, 1H), 7.78 – 7.72 (m, 1H), 7.68 (t, *J* = 7.6 Hz, 1H), 7.65 – 7.56 (m, 2H), 1.74 – 1.70 (m, 9H).

**<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)** δ 166.67, 142.98, 134.04, 130.31, 129.27, 128.39, 128.27, 126.46, 125.95, 124.35, 123.45, 123.00, 121.63, 77.38, 77.06, 76.74, 40.22, 31.23.

#### 3-(*tert*-butyl)-1-methylbenzo[*g*]quinoxalin-2(1H)-one (**4bc**)<sup>[2]</sup>

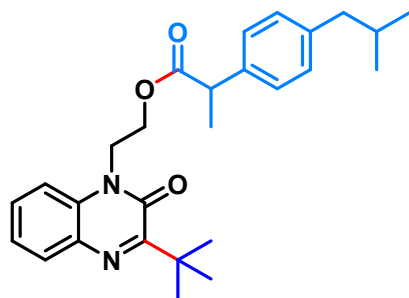


On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **4bc** as a white solid (21.3 mg, 40% yield).

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)** δ 8.34 (s, 1H), 7.95 (d, *J* = 8.3 Hz, 1H), 7.89 (d, *J* = 8.3 Hz, 1H), 7.53 (d, *J* = 8.9 Hz, 2H), 7.46 (t, *J* = 7.5 Hz, 1H), 3.73 (s, 3H), 1.53 (s, 9H).

**<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)** δ 165.96, 153.61, 133.42, 132.08, 131.67, 129.66, 129.06, 128.35, 127.51, 127.13, 124.99, 109.40, 77.36, 77.04, 76.73, 39.72, 28.77, 28.12.

**2-(3-(*tert*-butyl)-2-oxoquinoxalin-1(2H)-yl)ethyl 2-(4-isobutylphenyl)propanoate (4bd)**



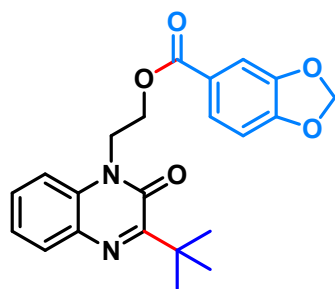
On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **4bd** as a colorless oil (53.0 mg, 61% yield).

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  7.99 (d,  $J$  = 8.1 Hz, 1H), 7.77 (d,  $J$  = 8.1 Hz, 1H), 7.61 (t,  $J$  = 7.6 Hz, 1H), 7.57 – 7.51 (m, 1H), 7.20 (d,  $J$  = 7.8 Hz, 2H), 7.05 (d,  $J$  = 7.7 Hz, 2H), 4.73 (dp,  $J$  = 12.5, 3.9 Hz, 2H), 4.59 (dt,  $J$  = 9.6, 4.5 Hz, 1H), 4.55 – 4.46 (m, 1H), 3.74 (q,  $J$  = 7.2 Hz, 1H), 2.43 (d,  $J$  = 7.1 Hz, 2H), 1.86 – 1.79 (m, 1H), 1.52 (d,  $J$  = 7.2 Hz, 3H), 1.46 (s, 9H), 0.90 (d,  $J$  = 6.6 Hz, 6H).

**<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)**  $\delta$  174.67, 156.01, 155.29, 140.57, 139.21, 138.22, 137.43, 129.33, 129.29, 128.95, 128.78, 127.14, 127.12, 126.34, 126.30, 77.36, 77.04, 76.73, 63.74, 62.81, 45.14, 45.01, 38.16, 30.13, 28.11, 22.40, 18.52.

**HRMS (ESI):** Calcd for C<sub>27</sub>H<sub>35</sub>N<sub>2</sub>O<sub>3</sub> [M+H]<sup>+</sup>: 435.2642; found: 435.2641.

**2-(3-(*tert*-butyl)-2-oxoquinoxalin-1(2H)-yl)ethyl benzo[d][1,3]dioxole-5-carboxylate (4be)**



On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **4be** as a white solid (22.1 mg, 28% yield, M.p. = 100–101 °C).

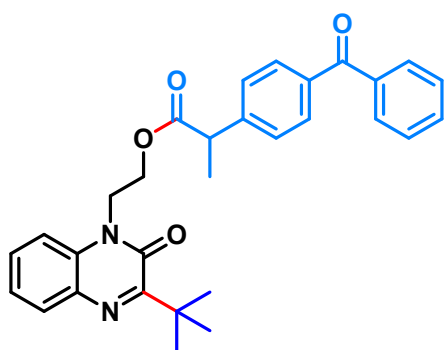
**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  7.97 (dt,  $J$  = 8.2, 1.6 Hz, 1H), 7.77 (dt,  $J$  = 8.2, 1.7 Hz, 1H), 7.66 (dt,  $J$  = 8.2, 1.7 Hz, 1H), 7.59 (ddt,  $J$  = 8.3, 7.0, 1.7 Hz, 1H), 7.56 – 7.49 (m, 1H), 7.47 (t,  $J$  = 1.7

Hz, 1H), 6.81 (dd,  $J = 8.2, 1.6$  Hz, 1H), 6.03 (d,  $J = 1.7$  Hz, 2H), 4.85 (dd,  $J = 4.7, 2.0$  Hz, 2H), 4.80 – 4.71 (m, 2H), 1.63 (d,  $J = 1.7$  Hz, 3H), 1.49 (d,  $J = 1.7$  Hz, 9H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  165.83, 156.05, 155.36, 151.79, 147.77, 139.24, 138.26, 129.01, 128.79, 126.40, 126.33, 125.55, 123.88, 109.55, 108.02, 101.85, 77.38, 77.06, 76.74, 63.95, 62.90, 38.26, 28.15.

HRMS (ESI): Calcd for  $\text{C}_{22}\text{H}_{23}\text{N}_2\text{O}_5$   $[\text{M}+\text{H}]^+$ : 395.1601; found: 395.1601.

#### 2-(3-(*tert*-butyl)-2-oxoquinoxalin-1(2H)-yl)ethyl 2-(4-benzoylphenyl)propanoate (4bf)



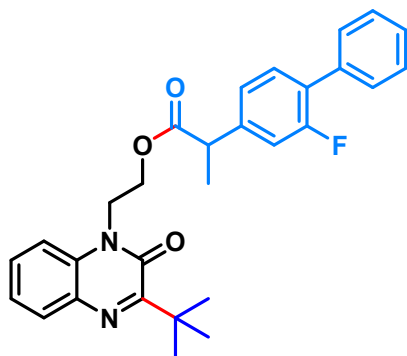
On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **4bf** as a colorless oil (64.6 mg, 67% yield).

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.96 (dd,  $J = 8.1, 1.6$  Hz, 1H), 7.79 – 7.72 (m, 4H), 7.64 (dt,  $J = 7.6, 1.5$  Hz, 1H), 7.60 – 7.48 (m, 4H), 7.48 – 7.42 (m, 2H), 7.37 (t,  $J = 7.7$  Hz, 1H), 4.72 (dt,  $J = 5.4, 3.2$  Hz, 2H), 4.59 (ddd,  $J = 12.2, 5.5, 3.8$  Hz, 1H), 4.51 (ddd,  $J = 12.2, 5.7, 3.6$  Hz, 1H), 3.83 (q,  $J = 7.2$  Hz, 1H), 1.54 (d,  $J = 7.2$  Hz, 3H), 1.41 (s, 9H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  196.37, 173.95, 155.87, 155.17, 140.53, 139.14, 138.19, 137.89, 137.44, 132.48, 131.50, 130.01, 129.19, 129.08, 128.98, 128.75, 128.56, 128.28, 126.38, 126.27, 77.38, 77.06, 76.74, 63.63, 63.03, 45.37, 38.13, 28.05, 18.50.

HRMS (ESI): Calcd for  $\text{C}_{30}\text{H}_{31}\text{N}_2\text{O}_4$   $[\text{M}+\text{H}]^+$ : 483.2278; found: 483.2280.

#### 2-(3-(*tert*-butyl)-2-oxoquinoxalin-1(2H)-yl)ethyl 2-(2-fluoro-[1,1'-biphenyl]-4-yl)propanoate (4bg)



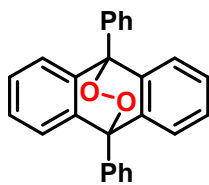
On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 5/1) to give **4bg** as a colorless oil (75.6 mg, 80% yield).

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  7.95 (d,  $J$  = 8.2 Hz, 1H), 7.75 (d,  $J$  = 8.2 Hz, 1H), 7.57 (t,  $J$  = 7.6 Hz, 1H), 7.53 – 7.46 (m, 3H), 7.42 (t,  $J$  = 7.6 Hz, 2H), 7.38 – 7.20 (m, 3H), 7.10 – 7.06 (m, 1H), 4.74 (t,  $J$  = 4.3 Hz, 2H), 4.56 (dq,  $J$  = 11.8, 6.9 Hz, 2H), 3.77 (d,  $J$  = 7.3 Hz, 1H), 1.53 (d,  $J$  = 7.1 Hz, 3H), 1.43 (s, 9H).

**<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)**  $\delta$  173.90, 159.70 (d,  $J$  = 248.5 Hz), 155.95, 155.25, 141.53 (d,  $J$  = 7.9 Hz), 139.20, 138.25, 135.47, 130.83 (d,  $J$  = 4.0 Hz), 128.97 (d,  $J$  = 7.7 Hz), 128.97, 128.81, 128.46, 127.91 (d,  $J$  = 13.4 Hz), 127.69, 126.41, 126.30, 123.53 (d,  $J$  = 3.3 Hz), 115.27 (d,  $J$  = 23.5 Hz), 63.64, 63.16, 45.05, 38.19, 28.10, 18.38.

**HRMS (ESI):** Calcd for C<sub>29</sub>H<sub>30</sub>FN<sub>2</sub>O<sub>3</sub> [M+H]<sup>+</sup>: 473.2235; found: 473.2235.

#### 9,10-diphenyl-9,10-dihydro-9,10-epidioxyanthracene (**5a**)<sup>[11]</sup>



On 0.2 mmol scale. Photoredox was conducted following the general procedure. The crude material was purified by flash column chromatography (silica gel, petroleum ether/ ethyl acetate = 20/1) to give **5a** as a white solid (16.9 mg, 23% yield).

**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  7.70 (d,  $J$  = 7.3 Hz, 4H), 7.63 (t,  $J$  = 7.6 Hz, 4H), 7.54 (t,  $J$  = 7.3 Hz, 2H), 7.19 (ddt,  $J$  = 9.1, 6.2, 3.2 Hz, 8H).

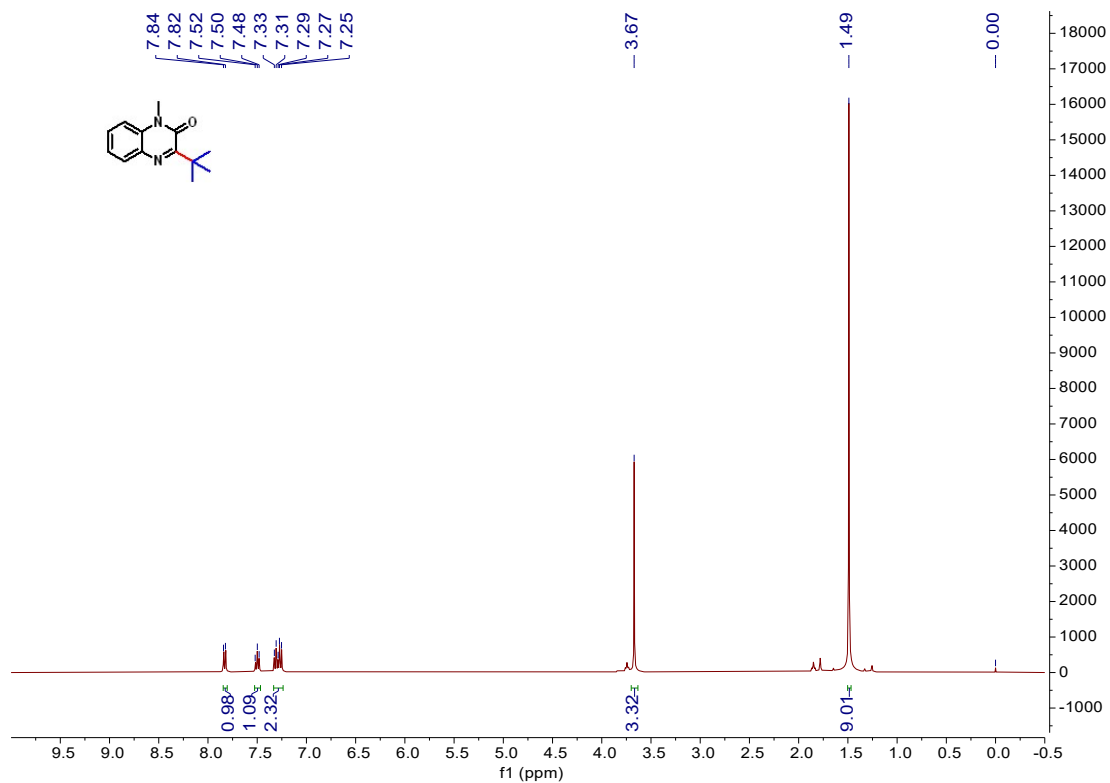
**<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)**  $\delta$  140.27, 133.03, 128.38, 128.30, 127.68, 127.56, 123.54, 84.12,



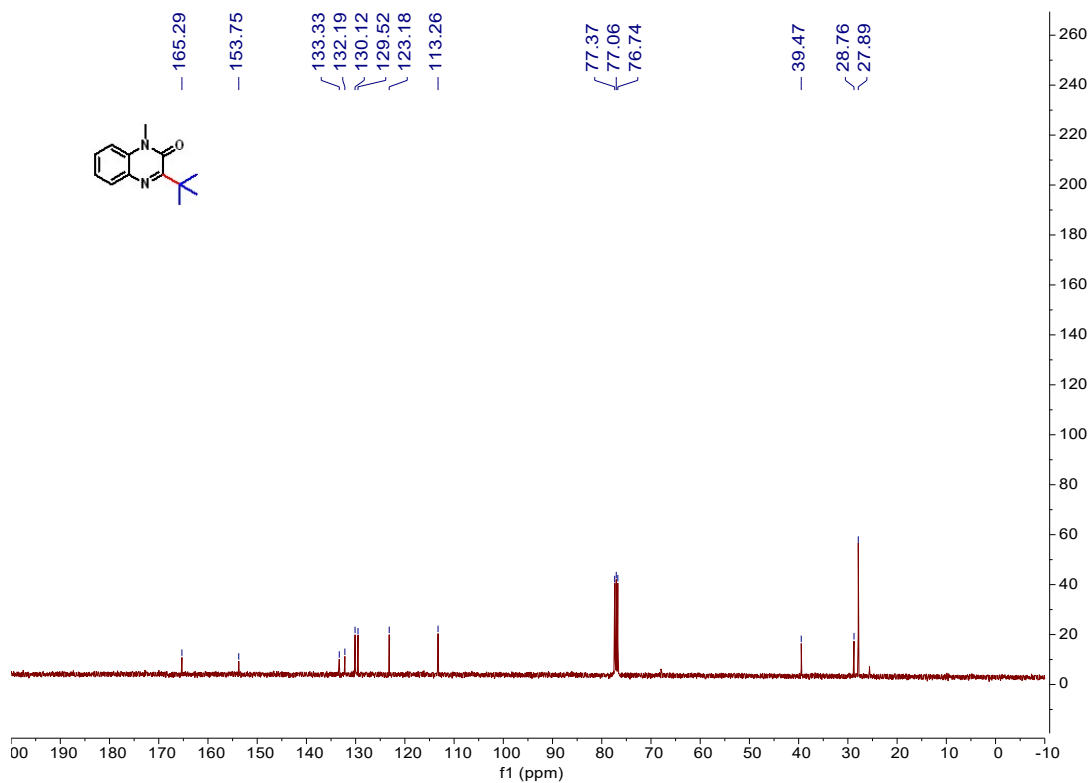
77.38, 77.06, 76.74.

## Spectra of prepared compounds

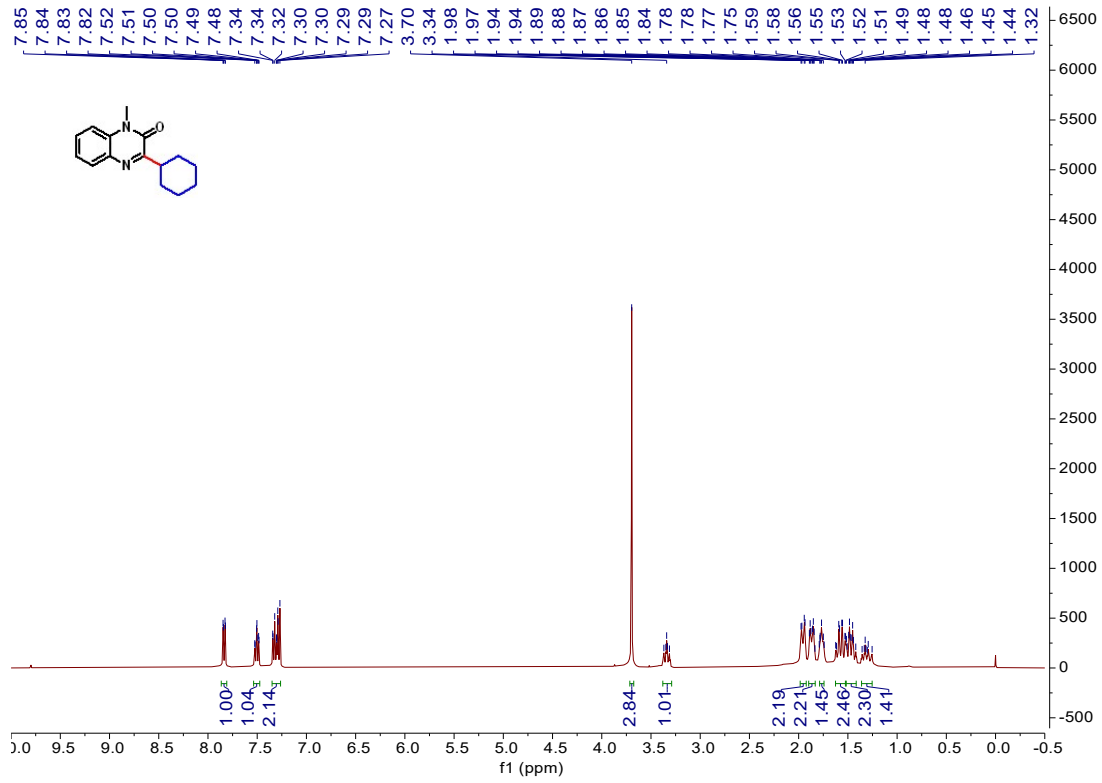
### <sup>1</sup>H NMR spectrum of compound **3aa**



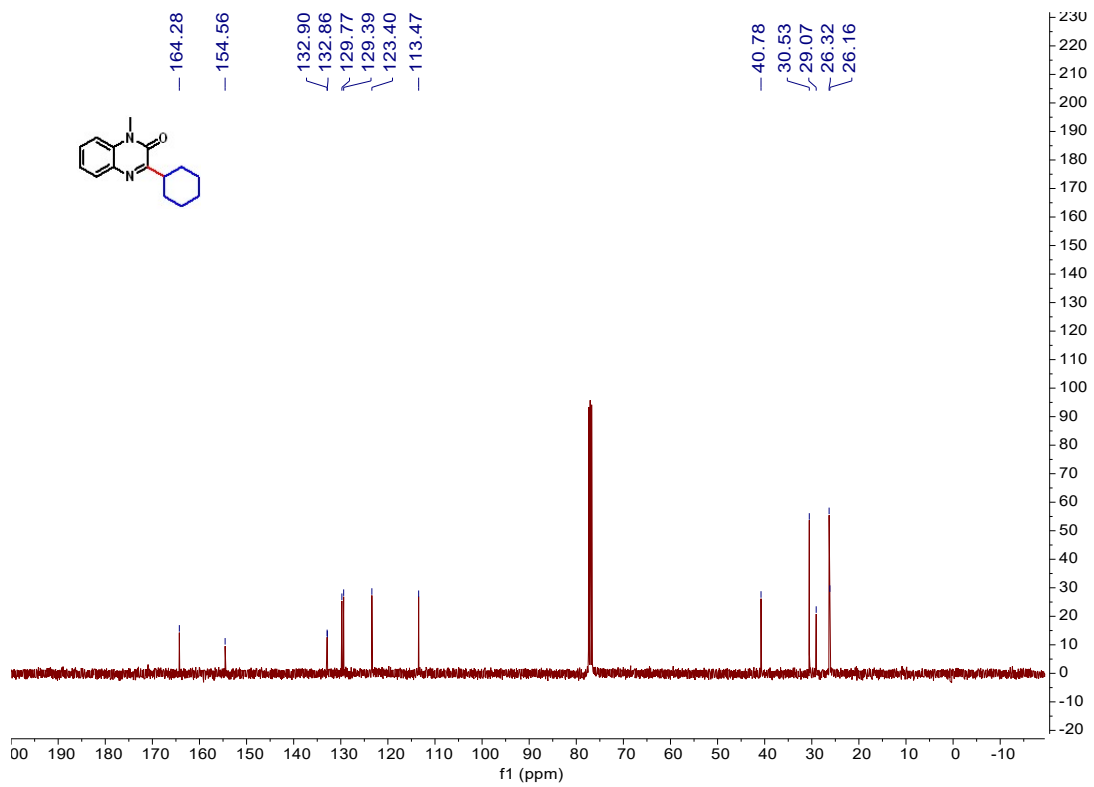
### <sup>13</sup>C NMR spectrum of compound **3aa**



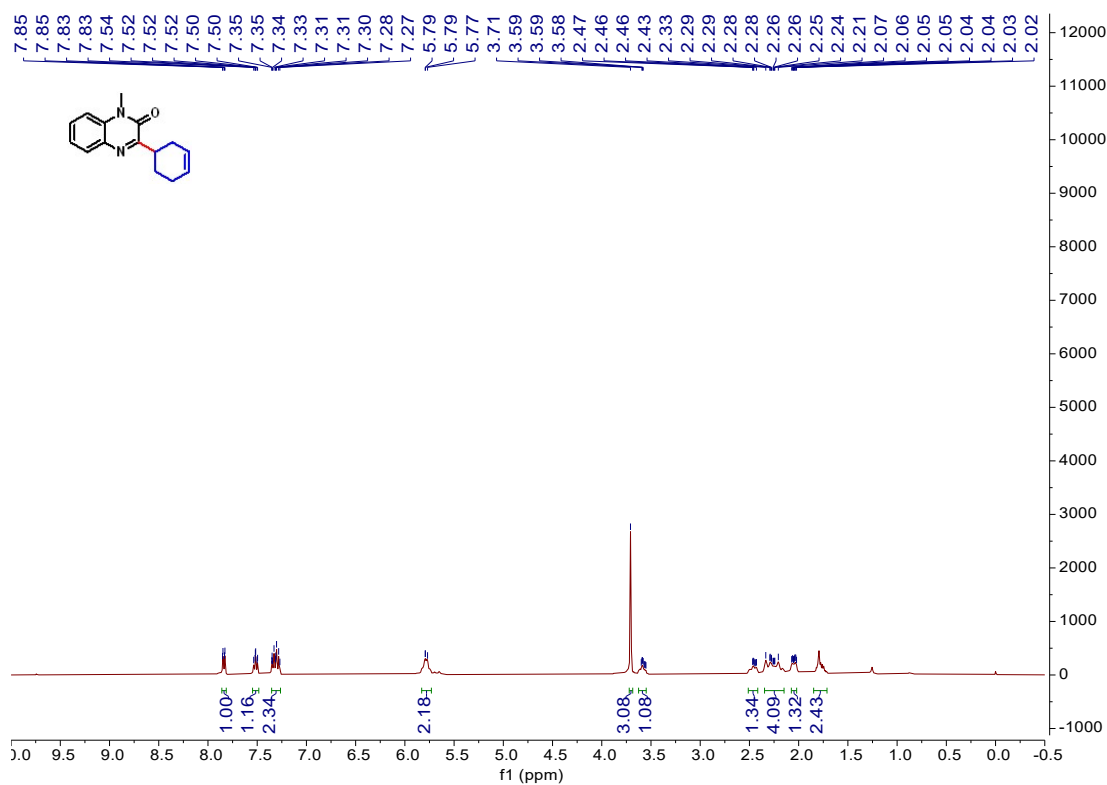
### <sup>1</sup>H NMR spectrum of compound **3ab**



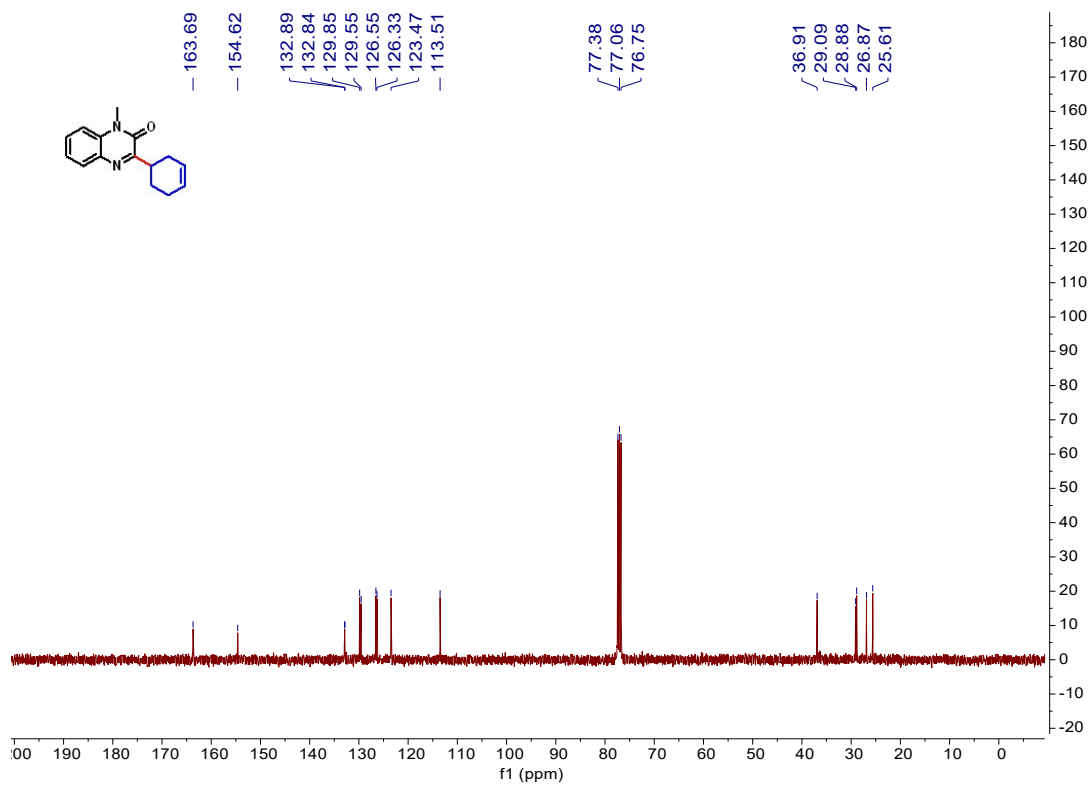
### <sup>13</sup>C NMR spectrum of compound **3ab**



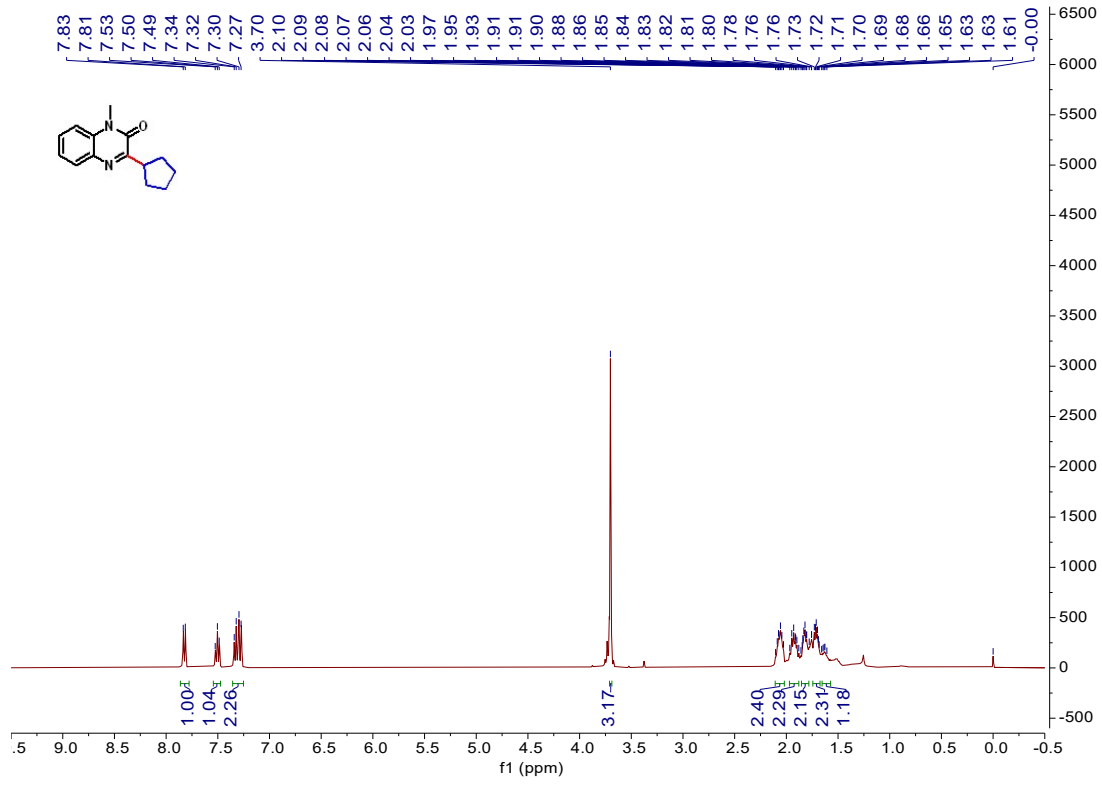
### <sup>1</sup>H NMR spectrum of compound **3ac**



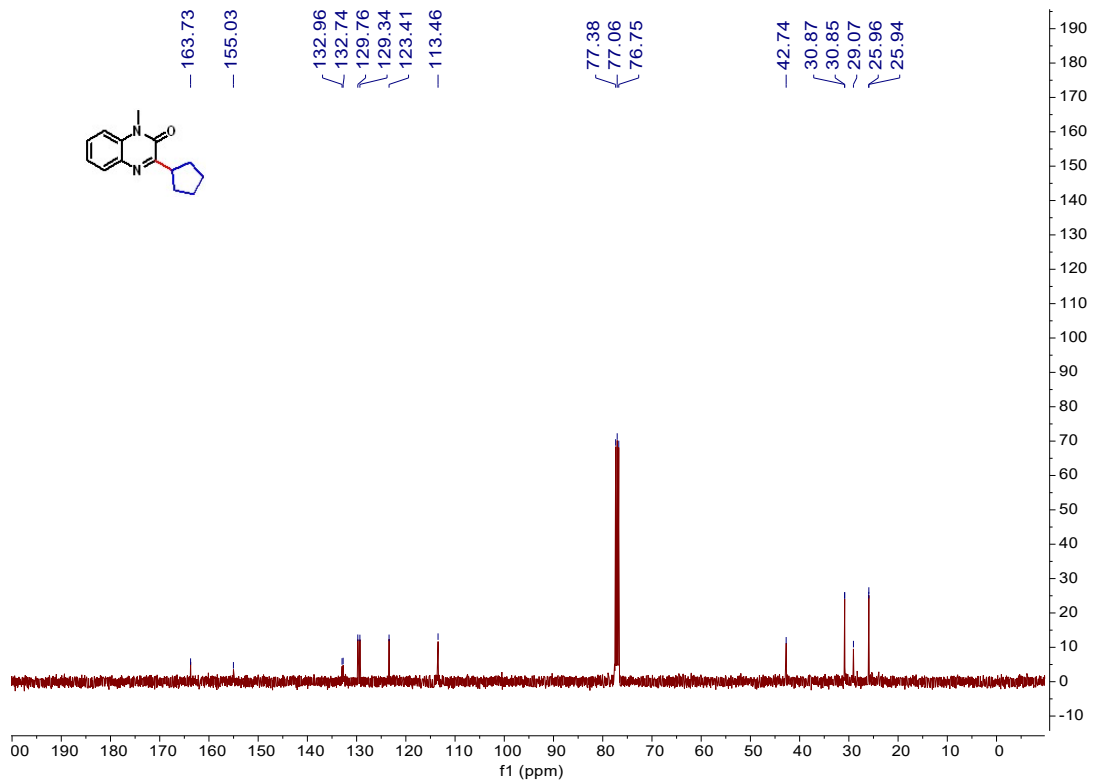
### <sup>13</sup>C NMR spectrum of compound **3ac**



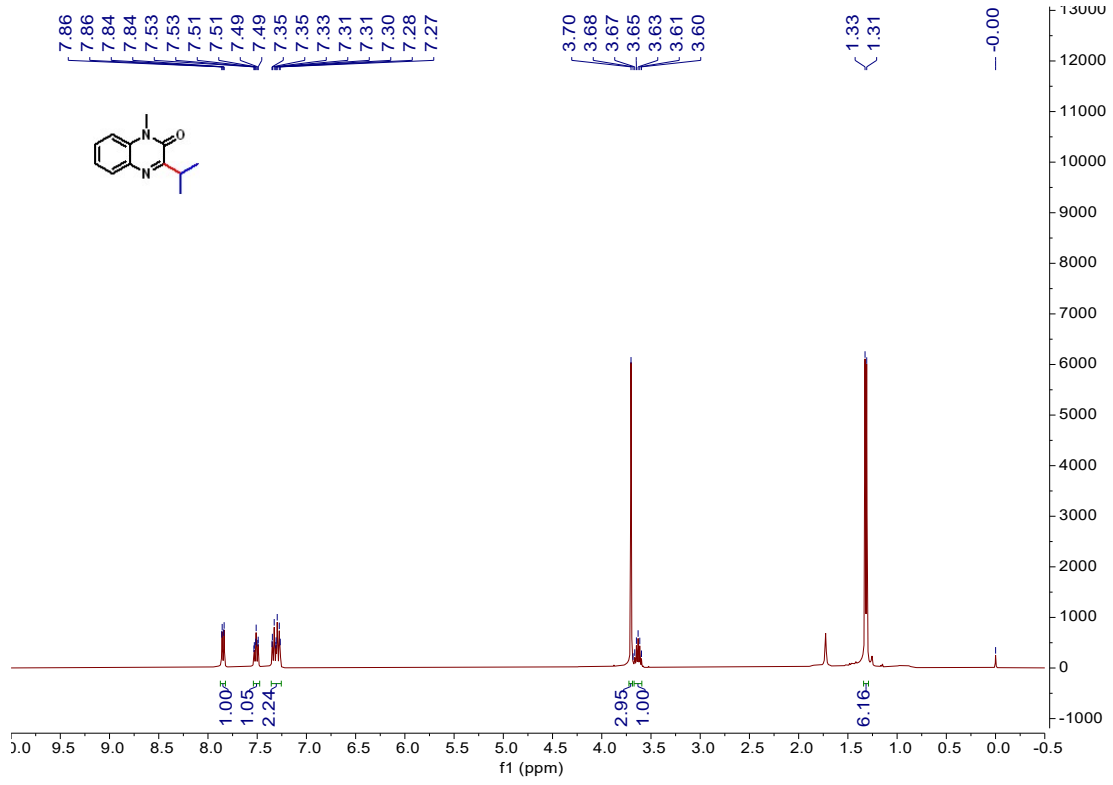
### <sup>1</sup>H NMR spectrum of compound 3ad



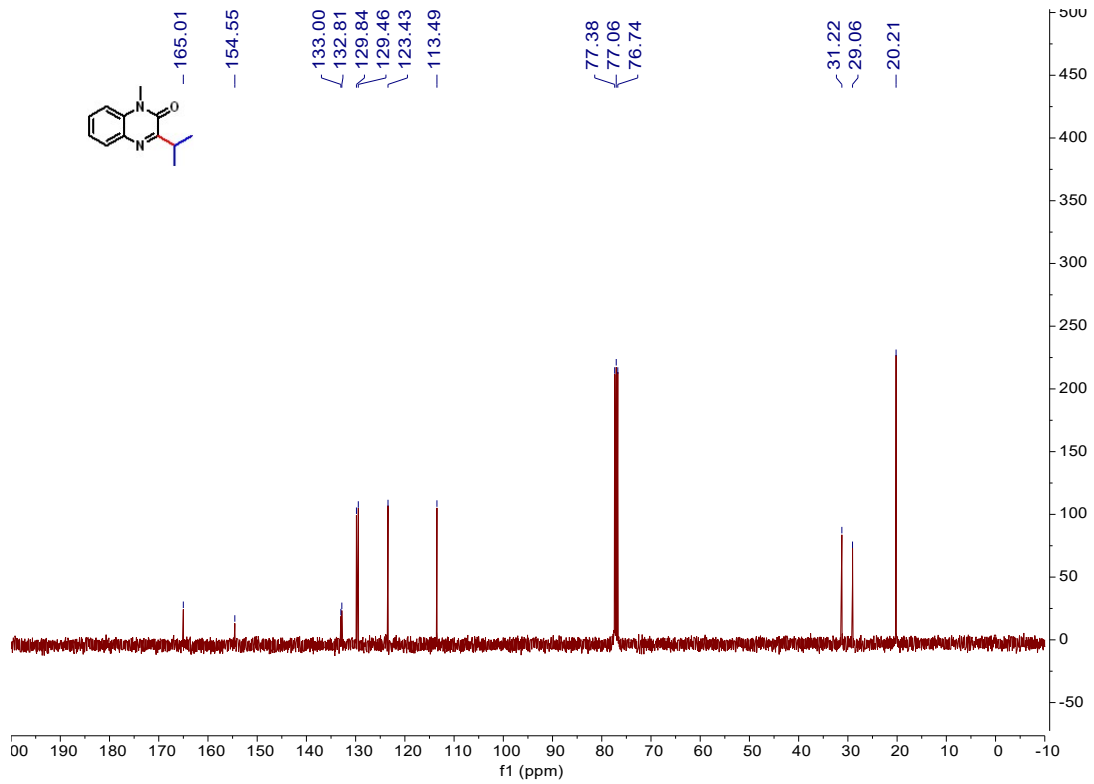
### <sup>13</sup>C NMR spectrum of compound 3ad



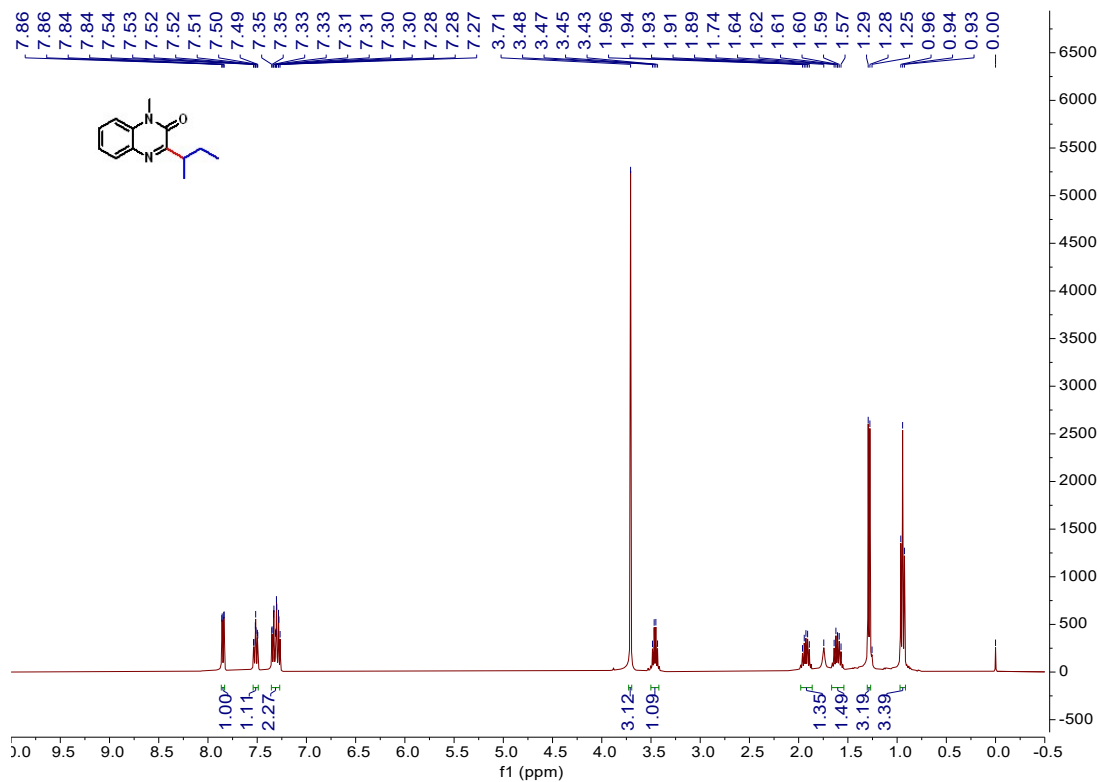
### <sup>1</sup>H NMR spectrum of compound **3ae**



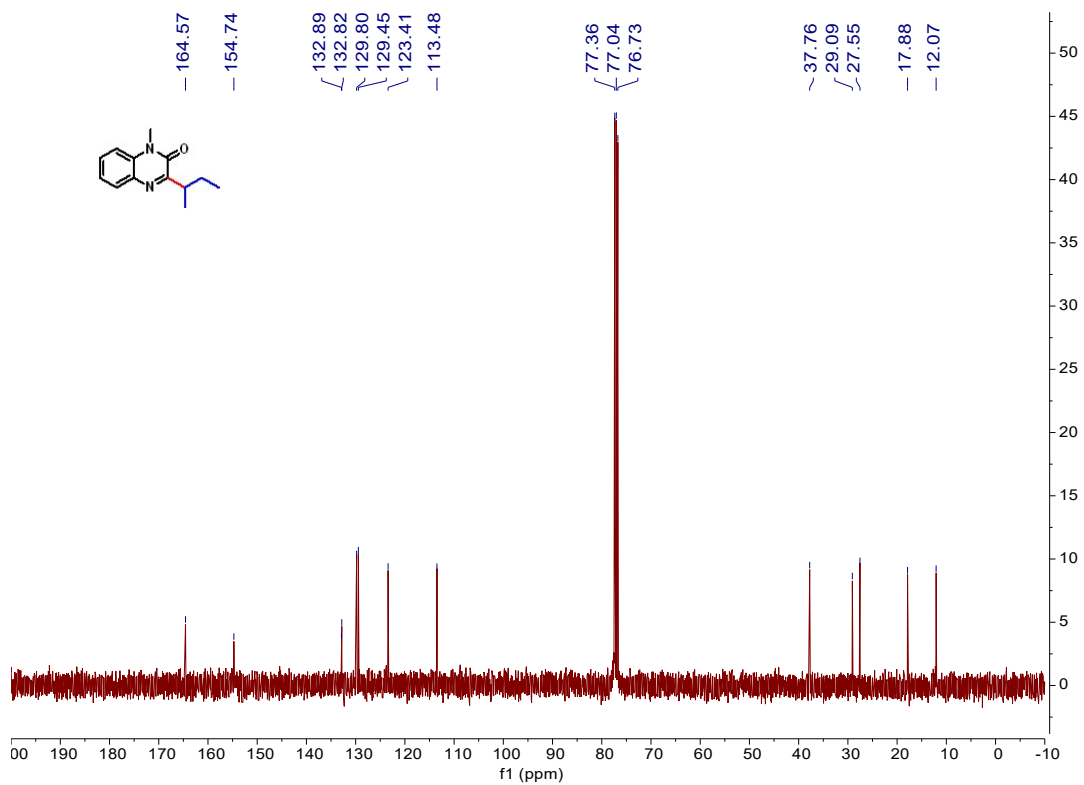
### <sup>13</sup>C NMR spectrum of compound **3ae**



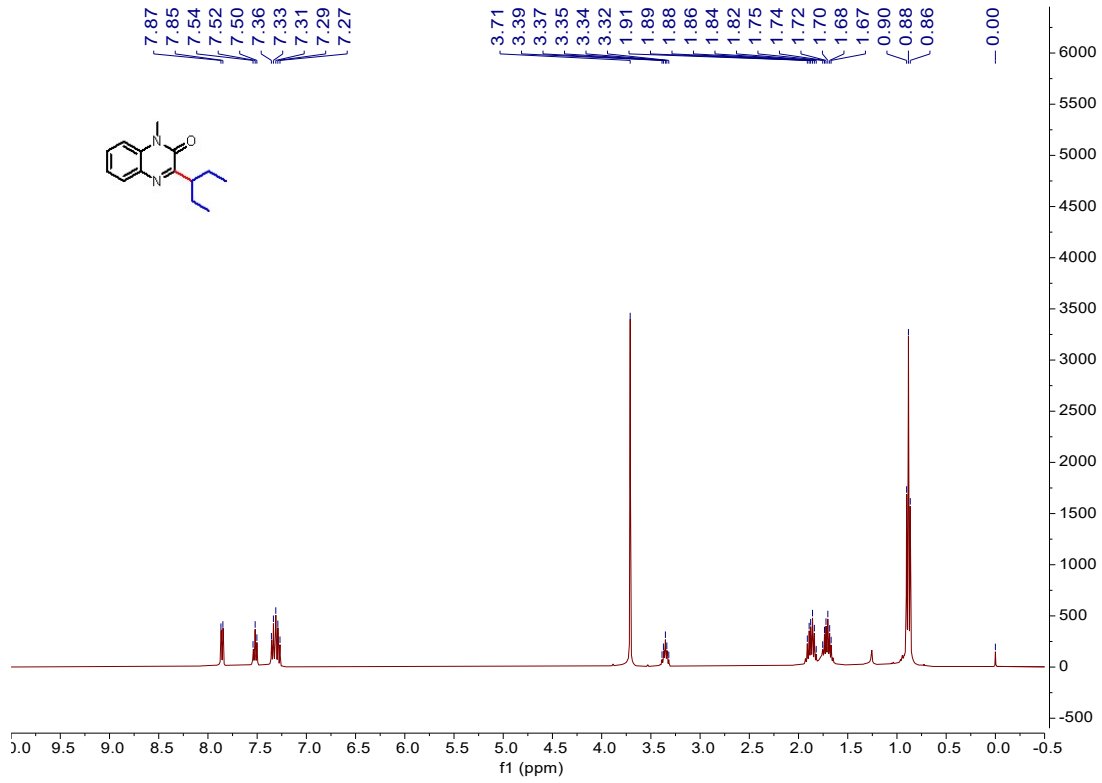
### <sup>1</sup>H NMR spectrum of compound **3af**



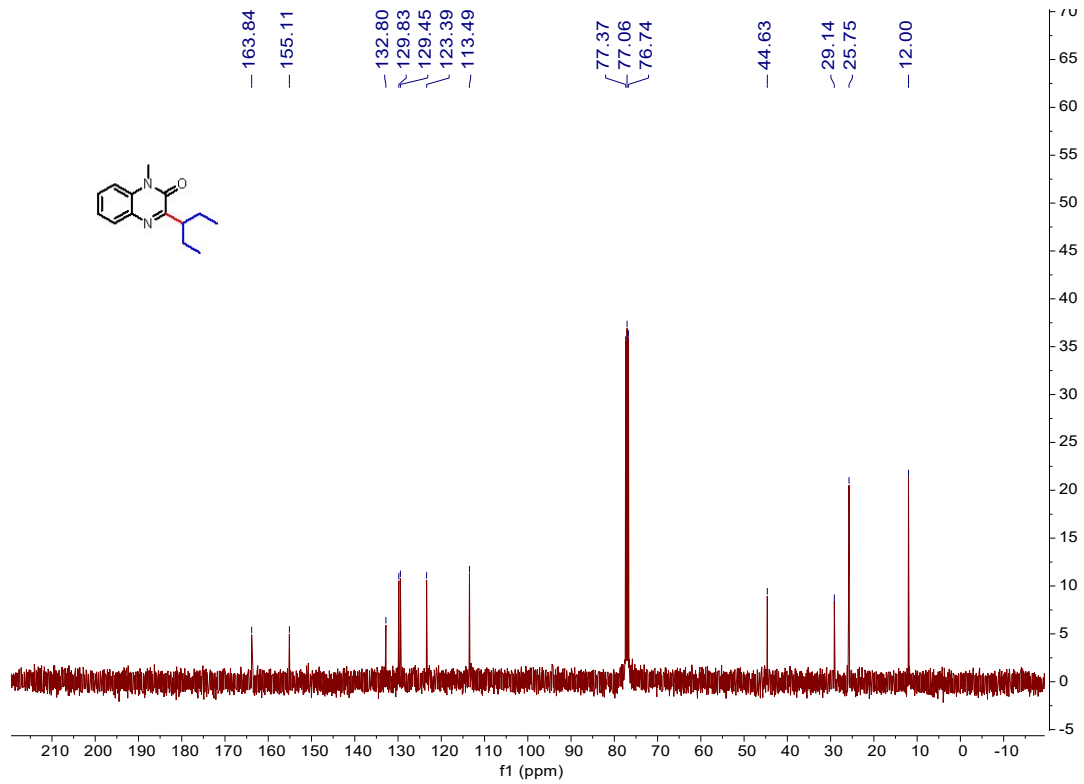
### <sup>13</sup>C NMR spectrum of compound **3af**



### <sup>1</sup>H NMR spectrum of compound **3ag**

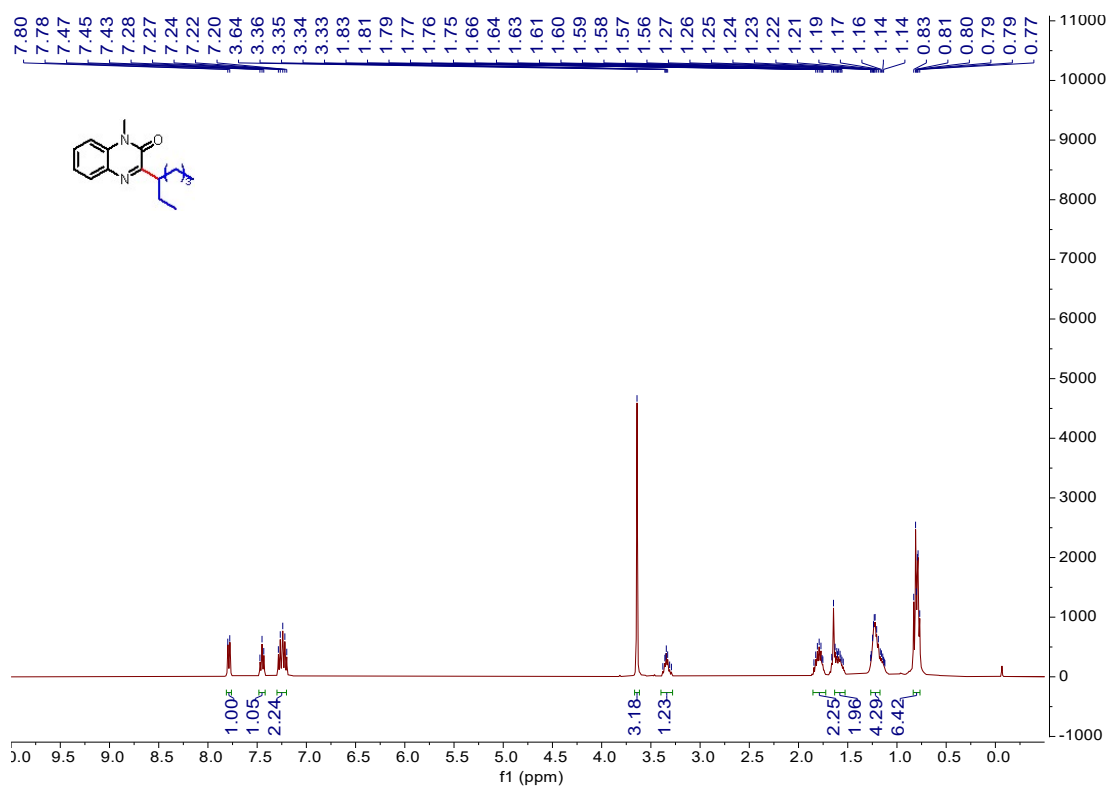


### <sup>13</sup>C NMR spectrum of compound **3ag**

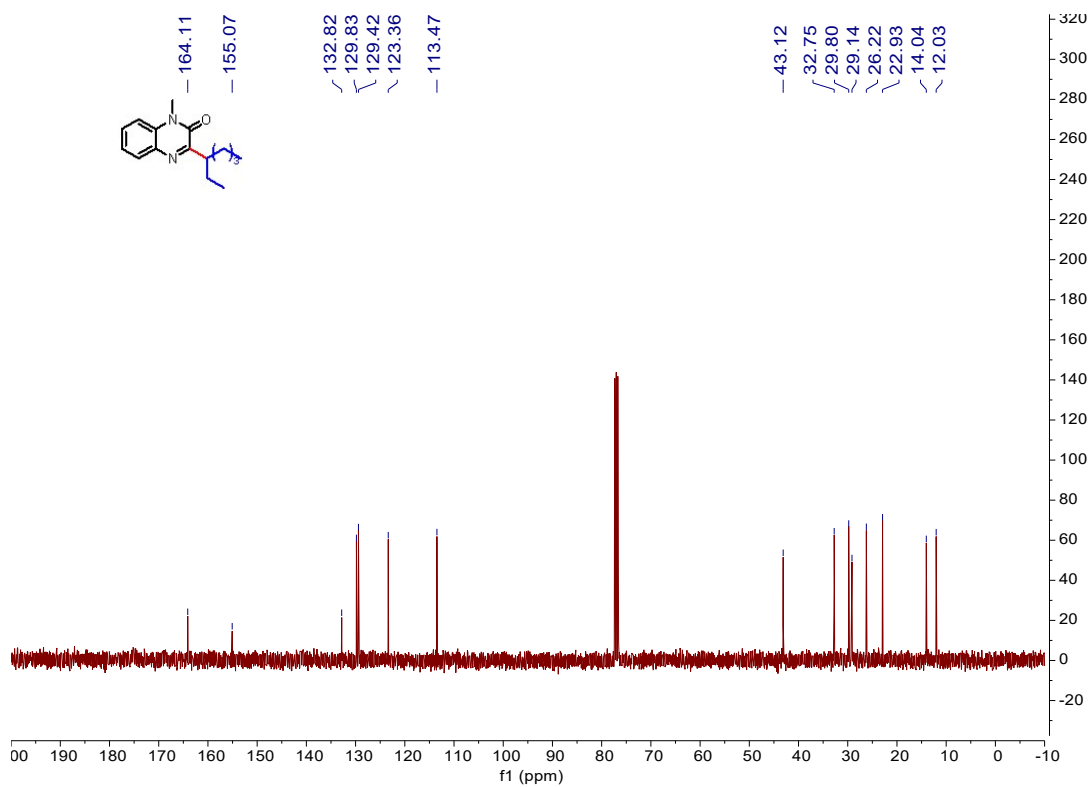




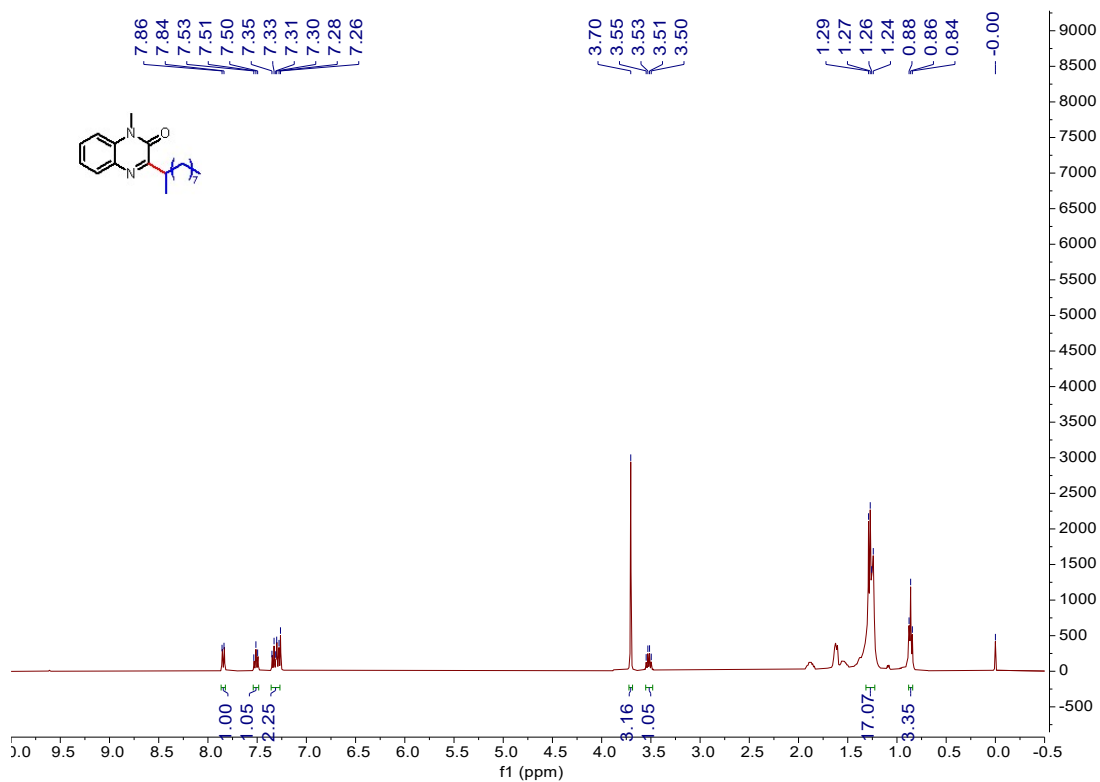
### <sup>1</sup>H NMR spectrum of compound **3ah**



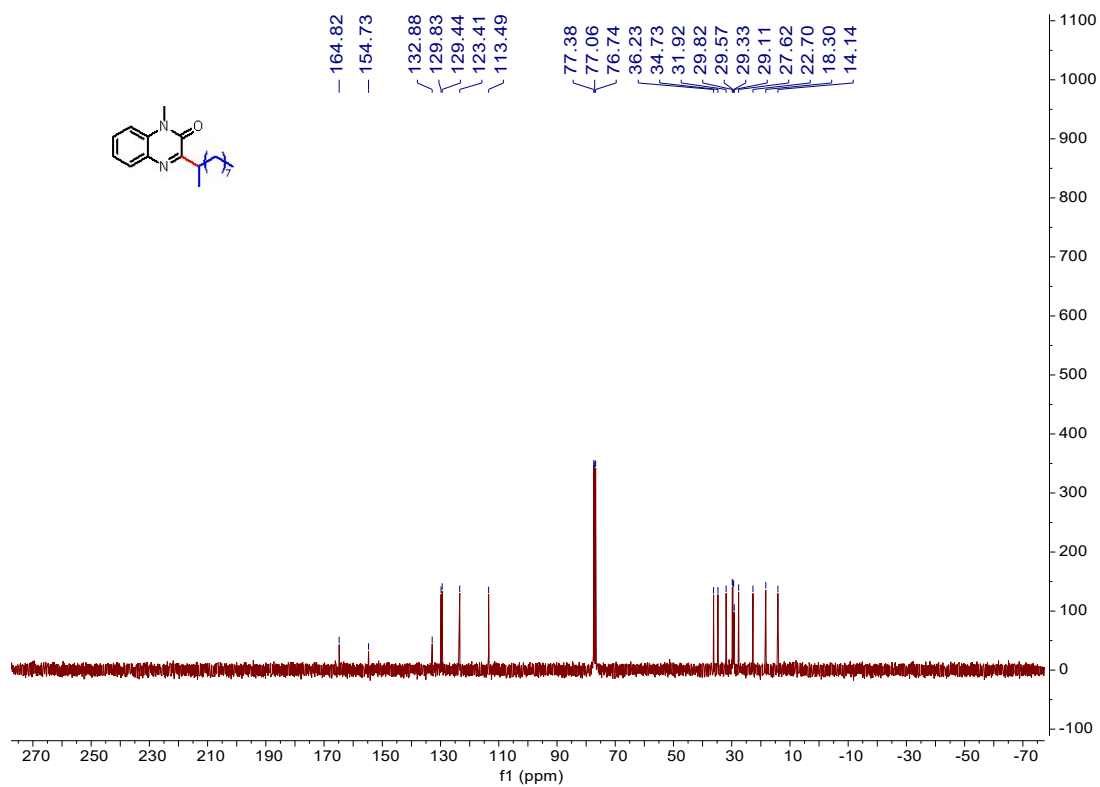
### <sup>13</sup>C NMR spectrum of compound **3ah**



### <sup>1</sup>H NMR spectrum of compound 3ai

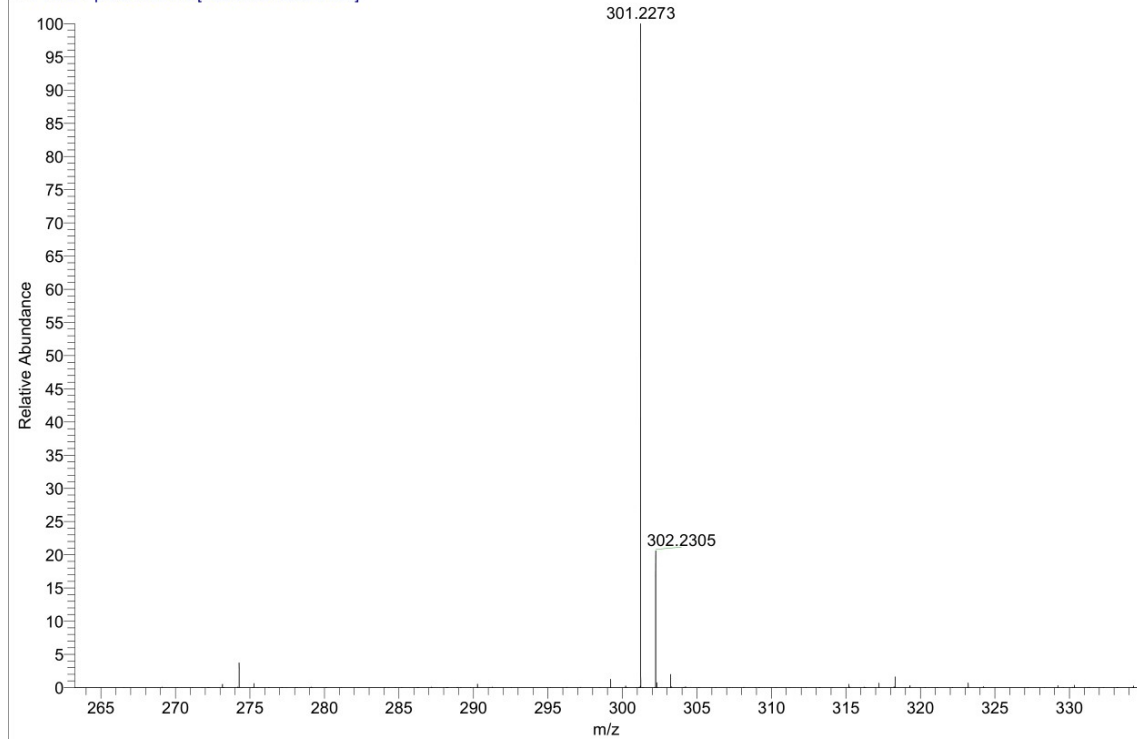


### <sup>13</sup>C NMR spectrum of compound 3ai

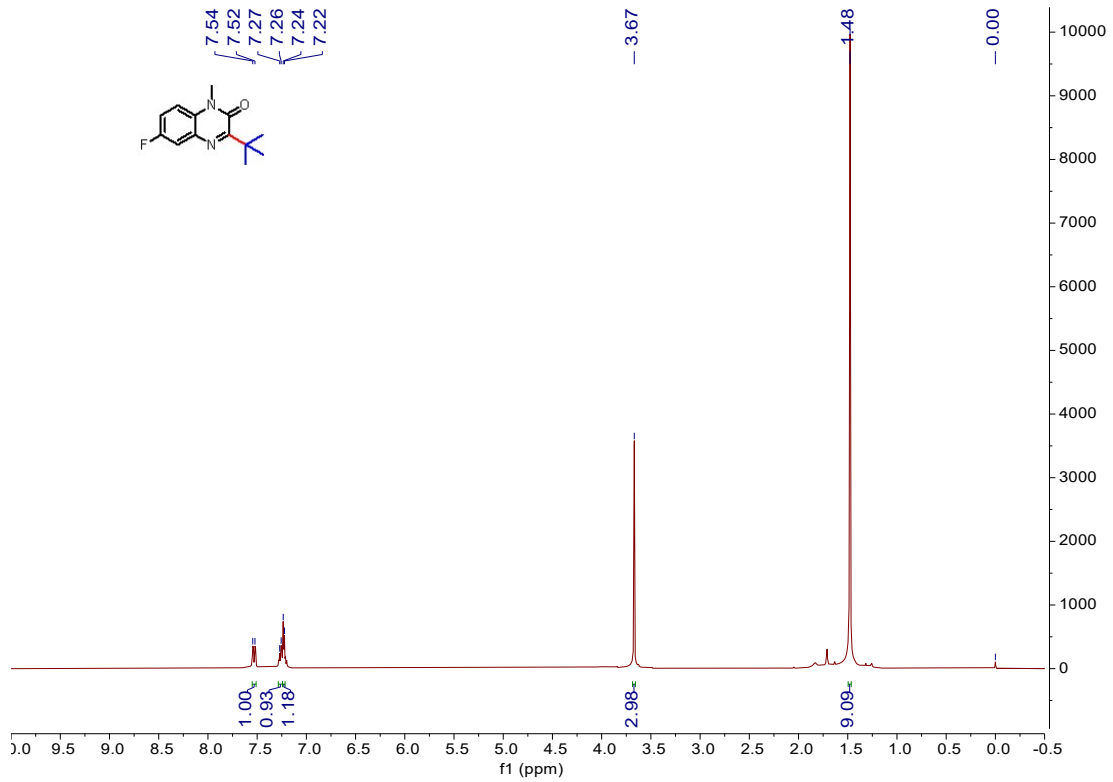


### HRMS of compound **3ai**

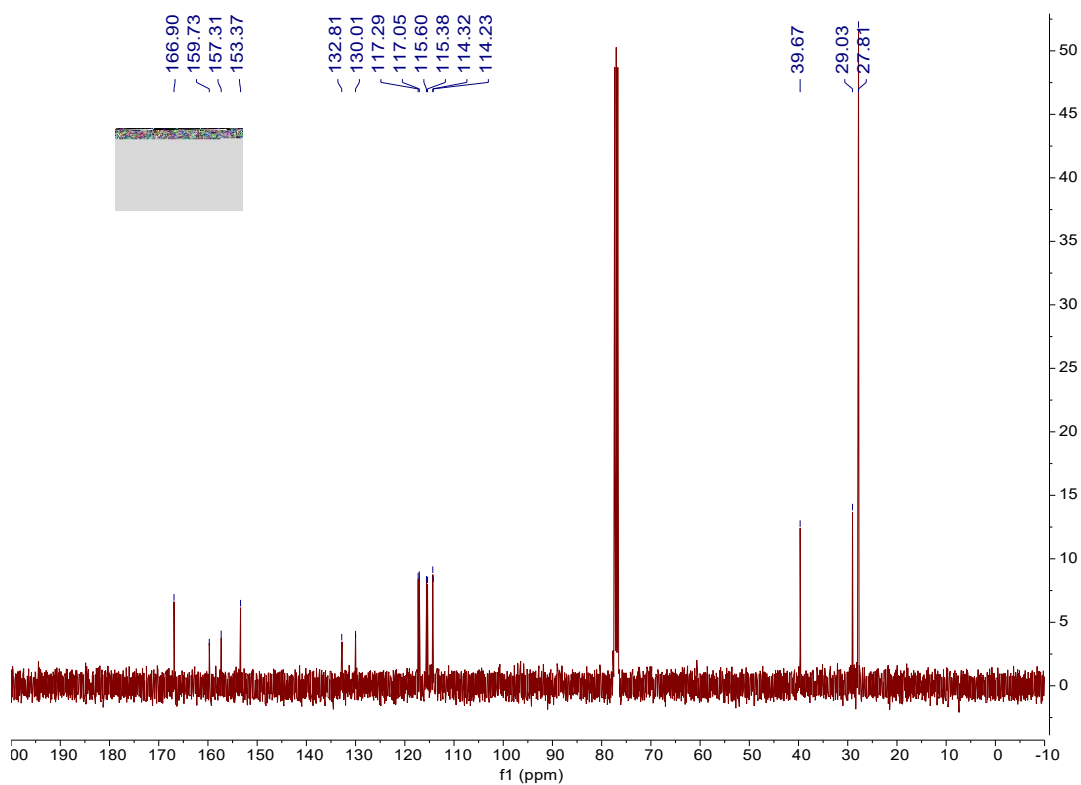
SXD-04-61 #22-31 RT: 0.10-0.14 AV: 10 NL: 3.35E9  
T: FTMS + p ESI Full ms [100.0000-1500.0000]



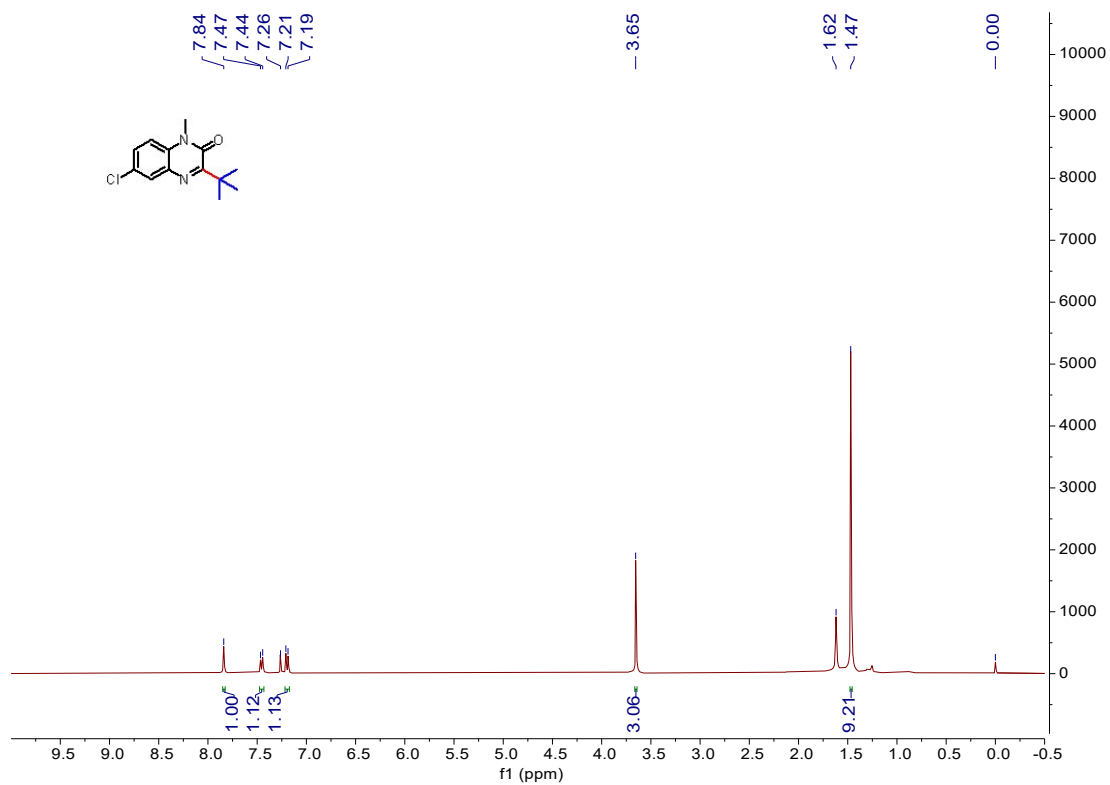
### <sup>1</sup>H NMR spectrum of compound **4aa**



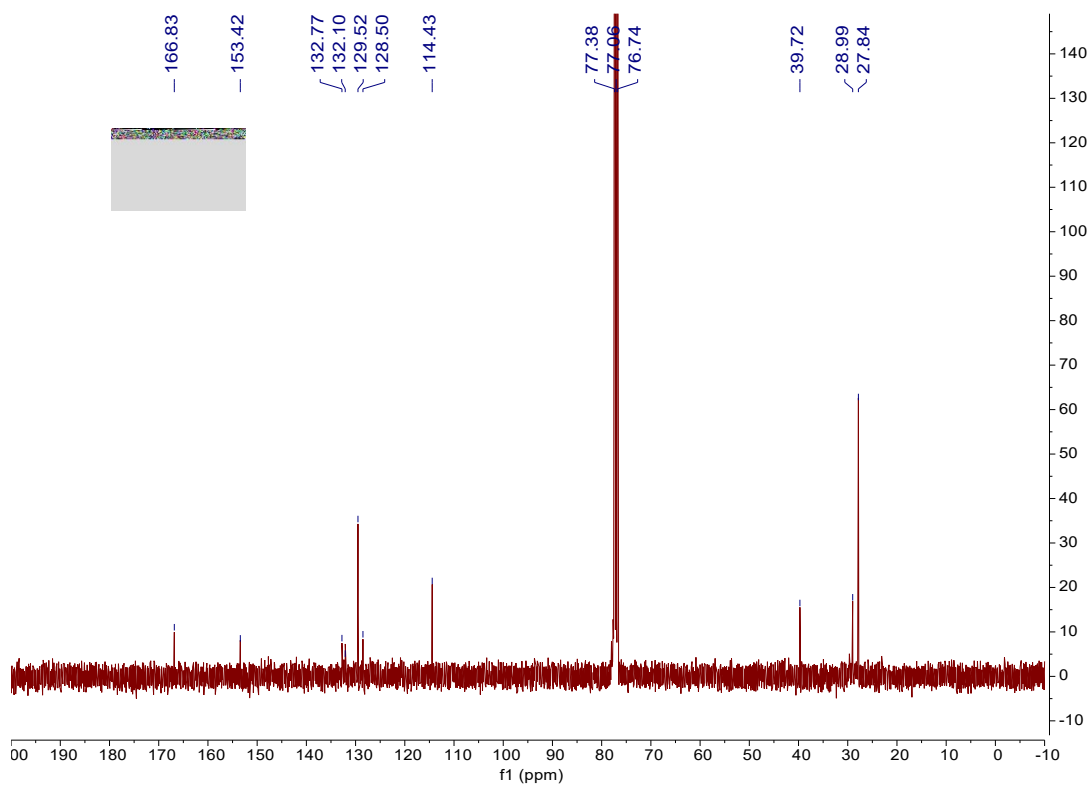
<sup>13</sup>C NMR spectrum of compound **4aa**



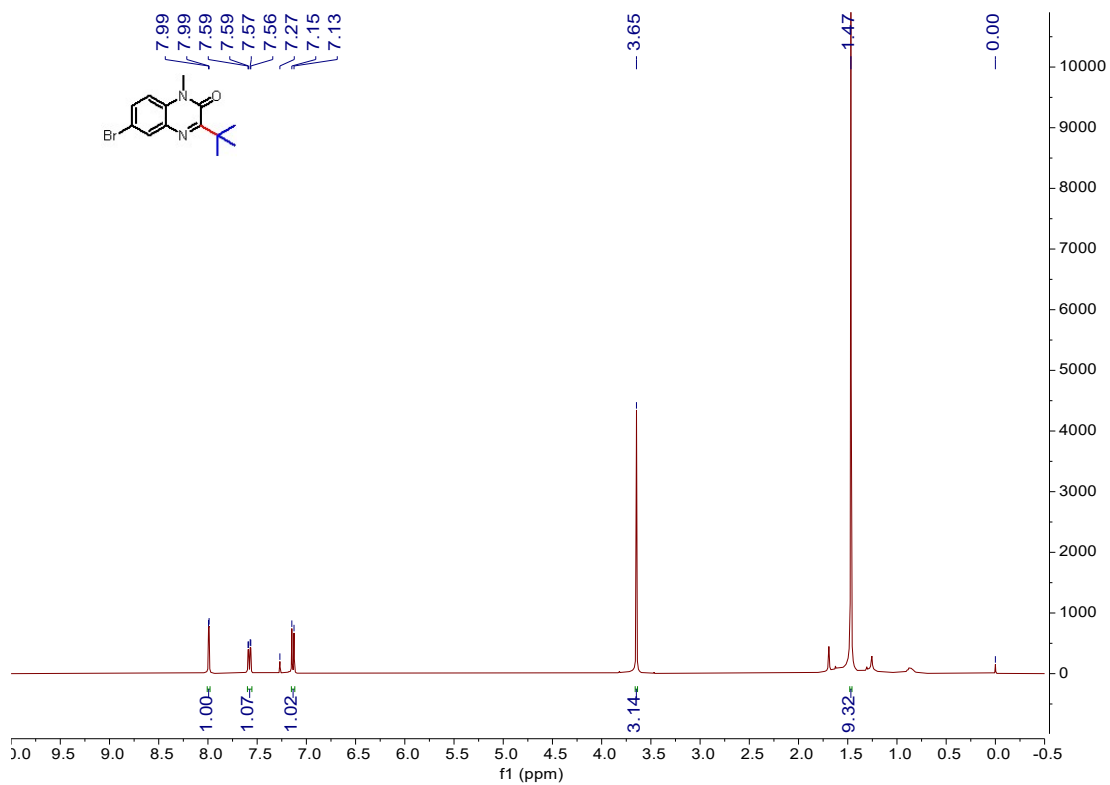
<sup>1</sup>H NMR spectrum of compound **4ab**



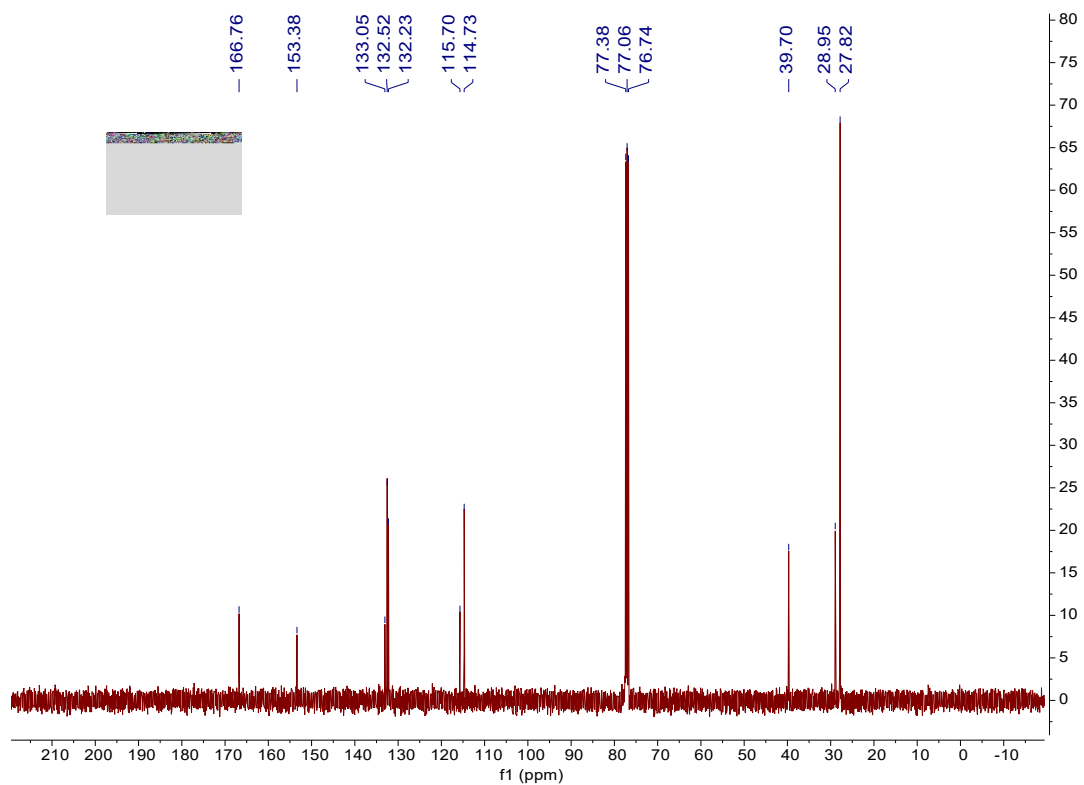
<sup>13</sup>C NMR spectrum of compound **4ab**



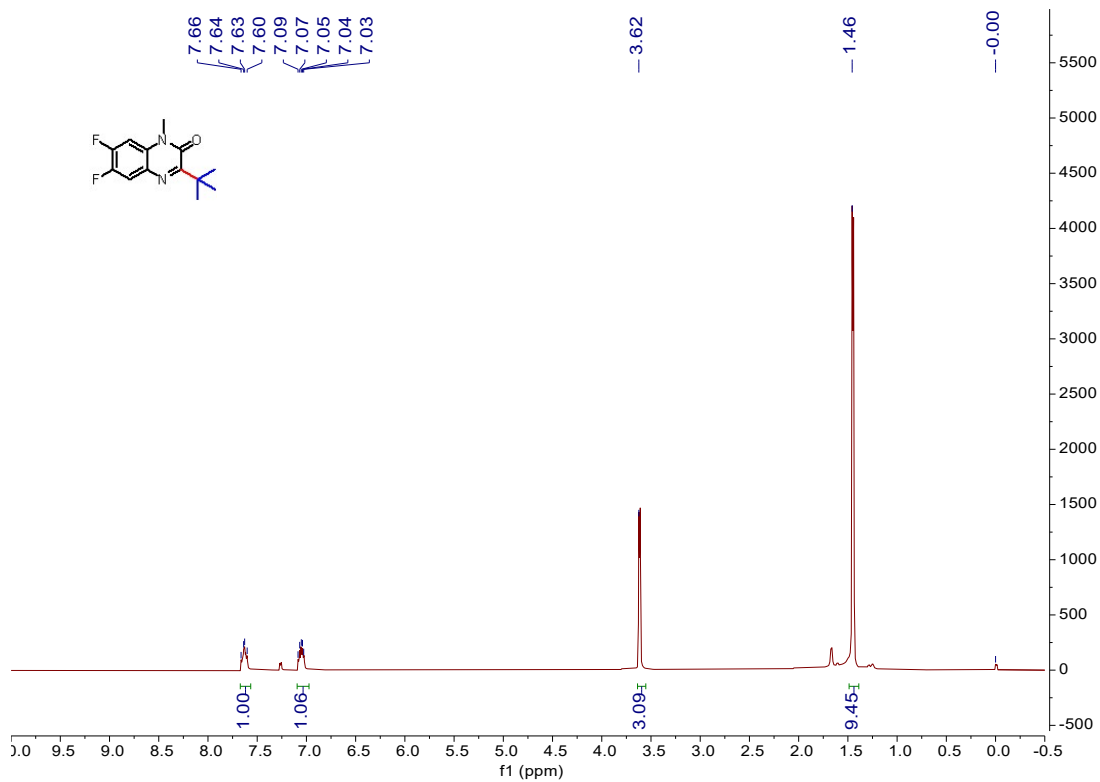
<sup>1</sup>H NMR spectrum of compound **4ac**



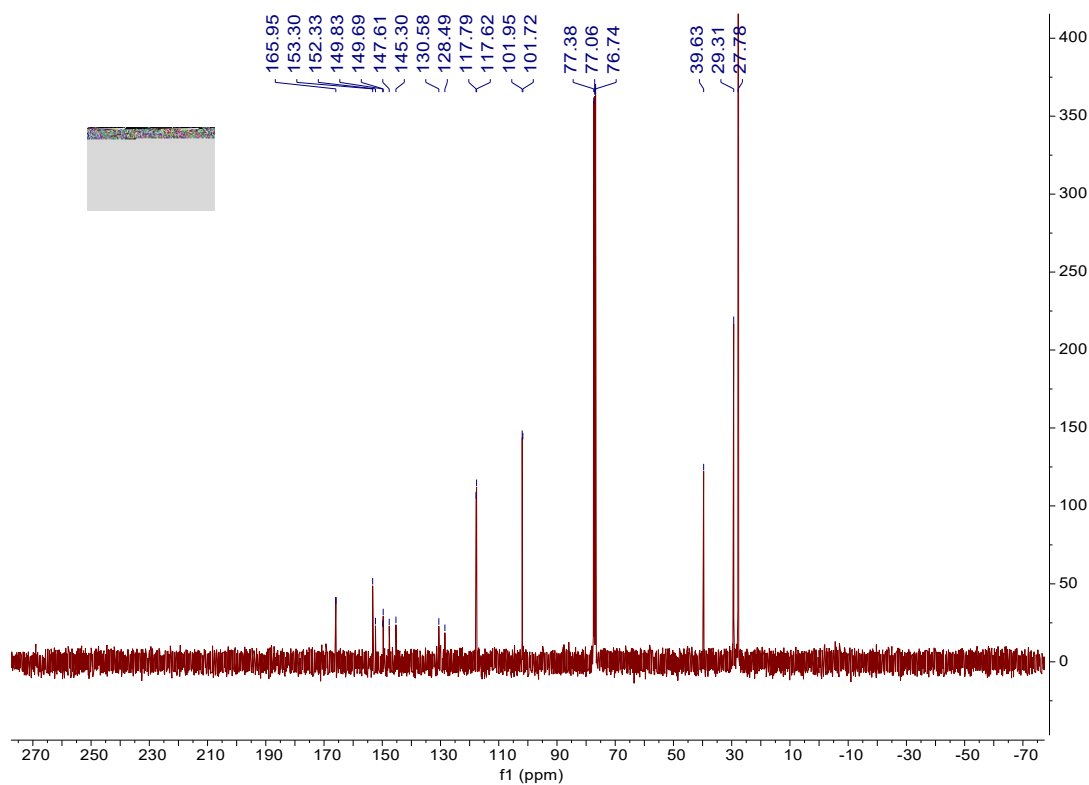
<sup>13</sup>C NMR spectrum of compound **4ac**



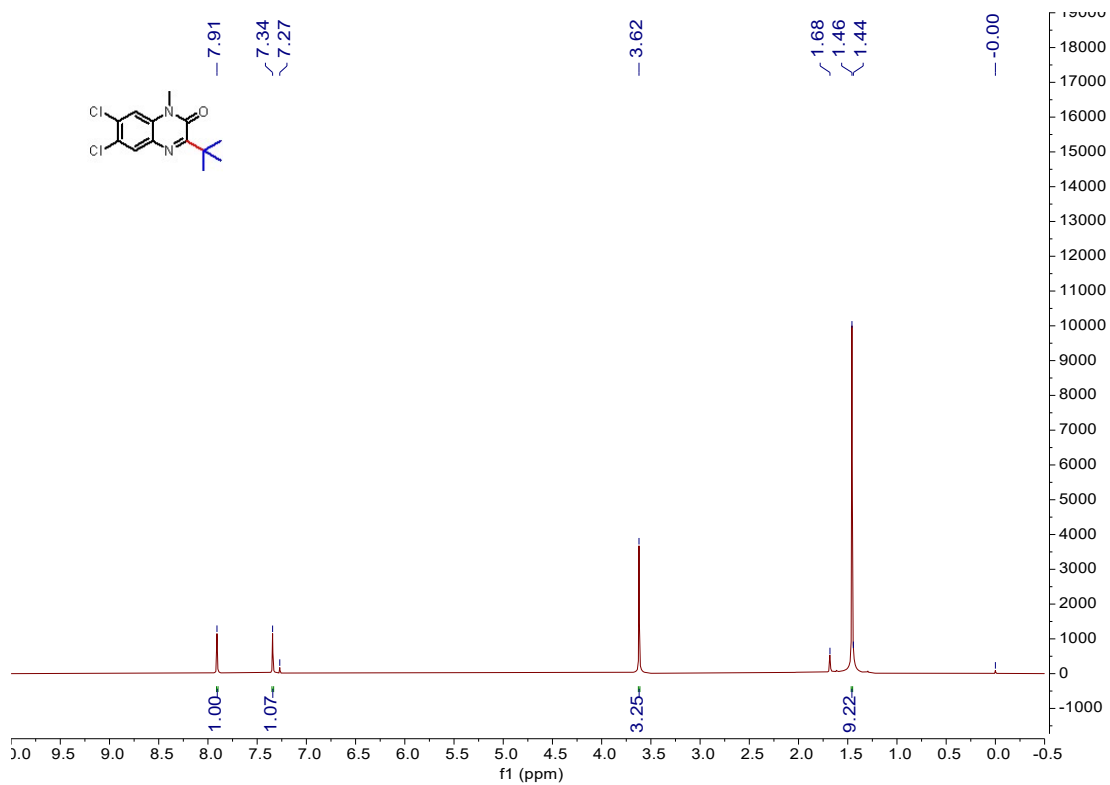
<sup>1</sup>H NMR spectrum of compound **4ad**



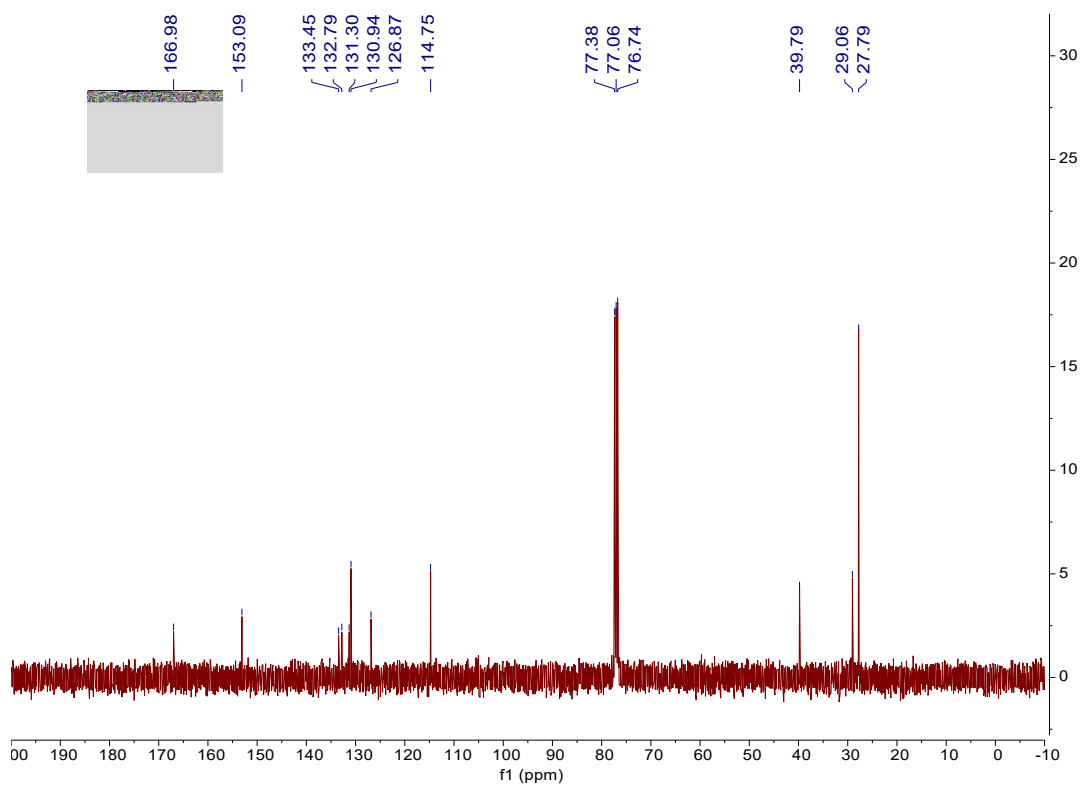
<sup>13</sup>C NMR spectrum of compound **4ad**



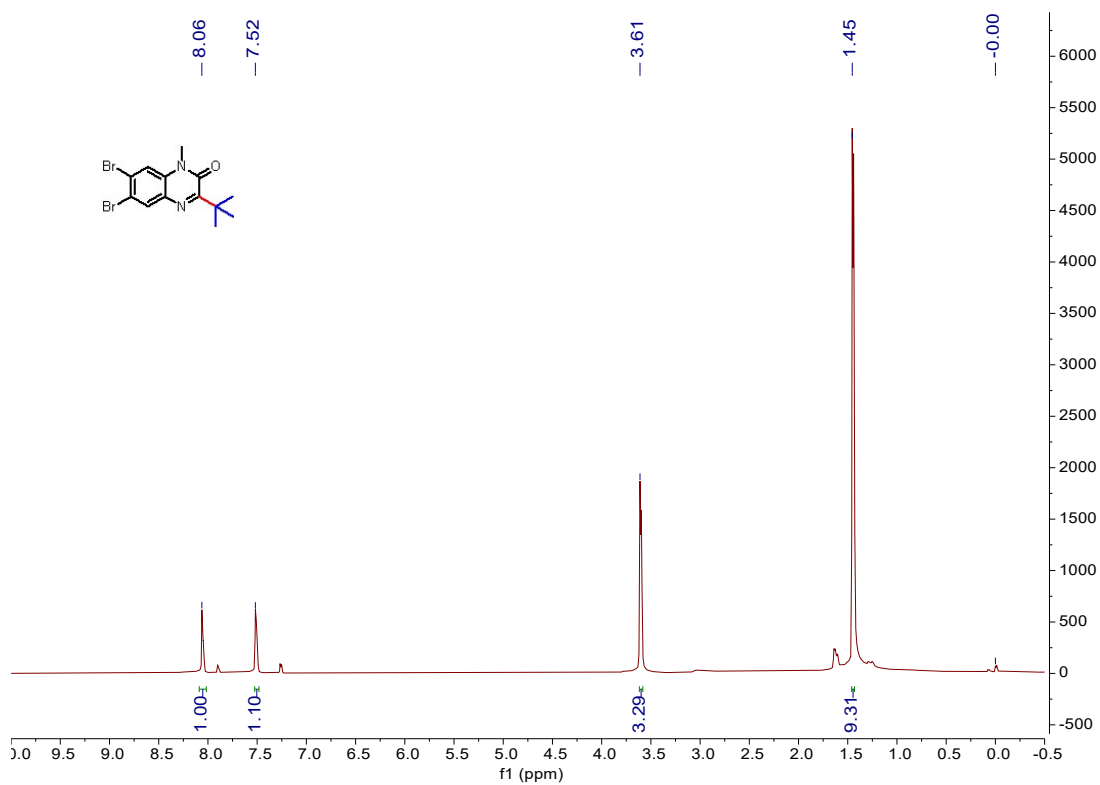
<sup>1</sup>H NMR spectrum of compound **4ae**



<sup>13</sup>C NMR spectrum of compound **4ae**

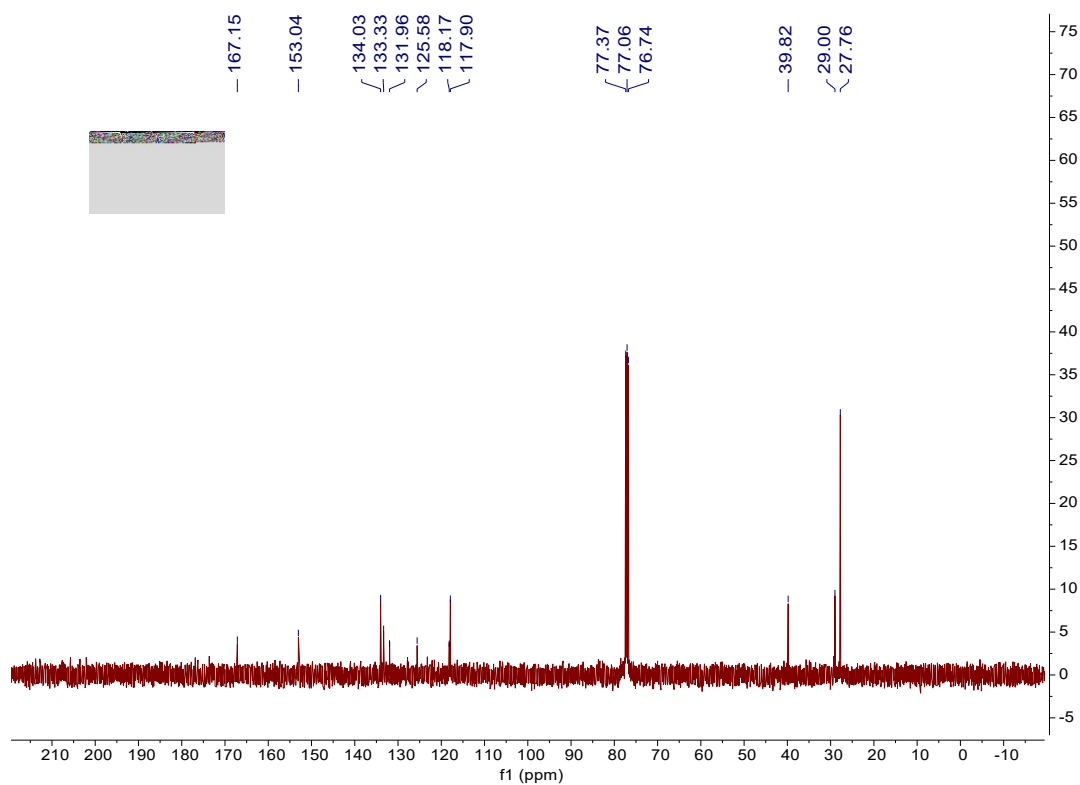


<sup>1</sup>H NMR spectrum of compound **4af**

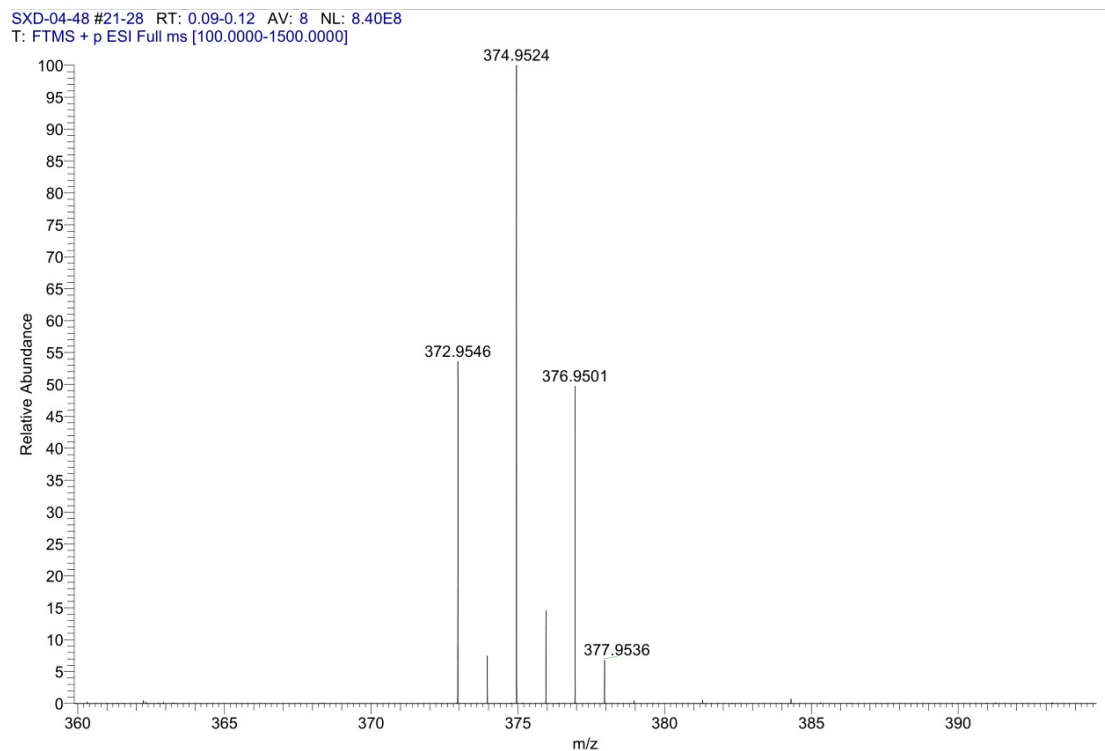




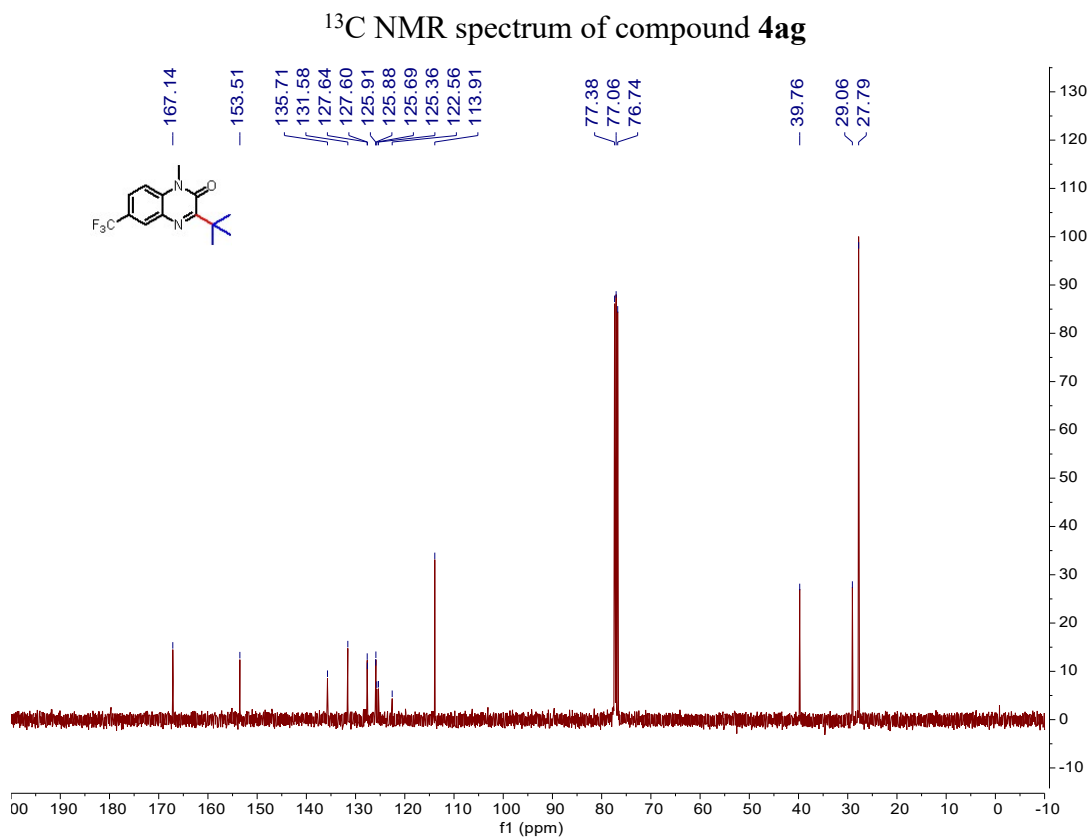
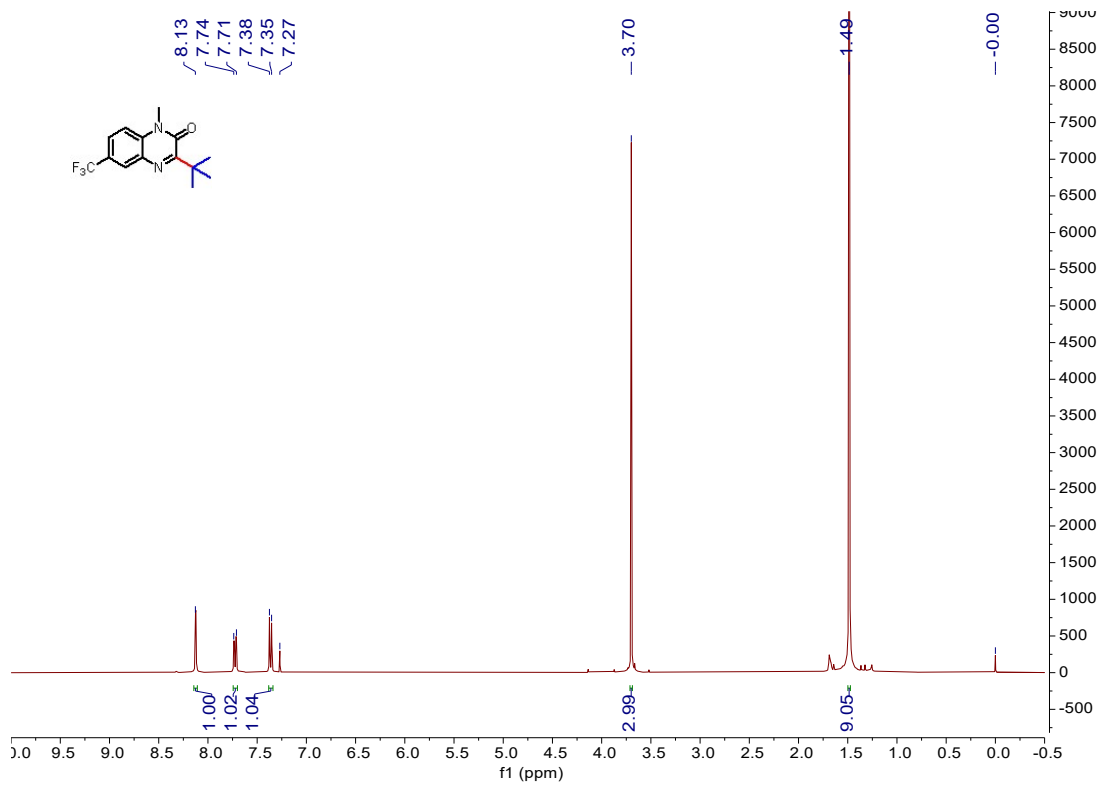
### <sup>13</sup>C NMR spectrum of compound **4af**



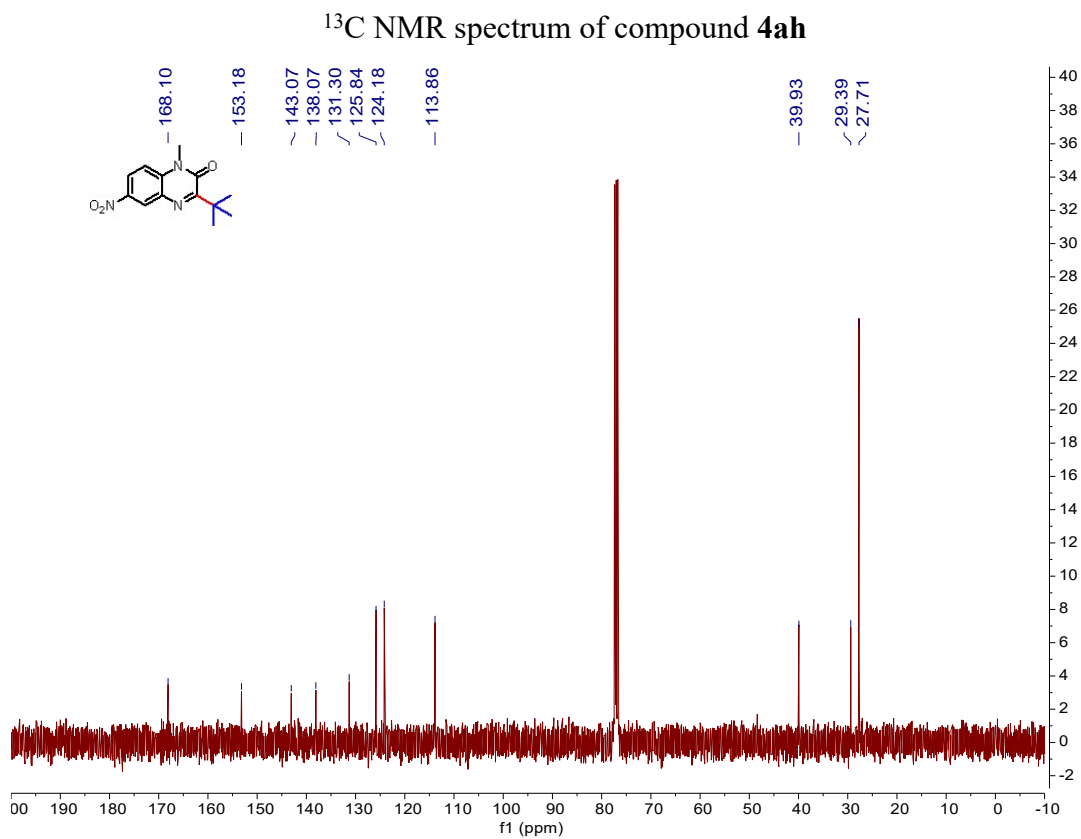
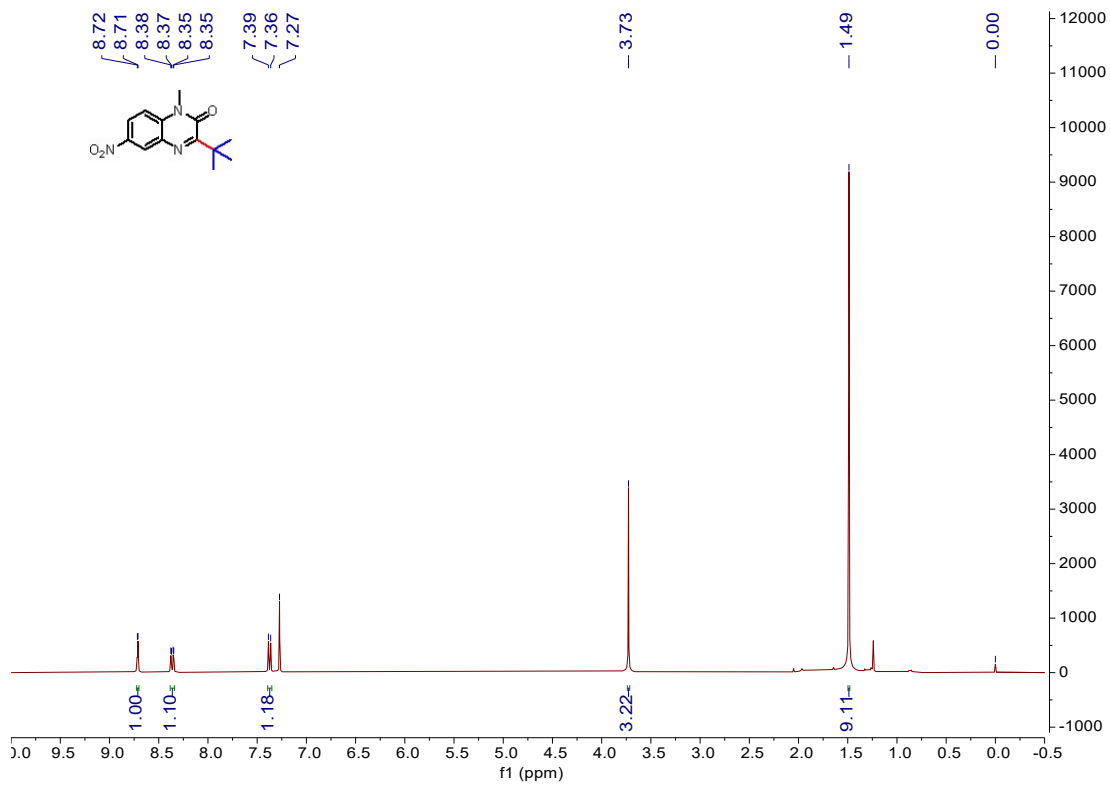
### HRMS of compound **4af**



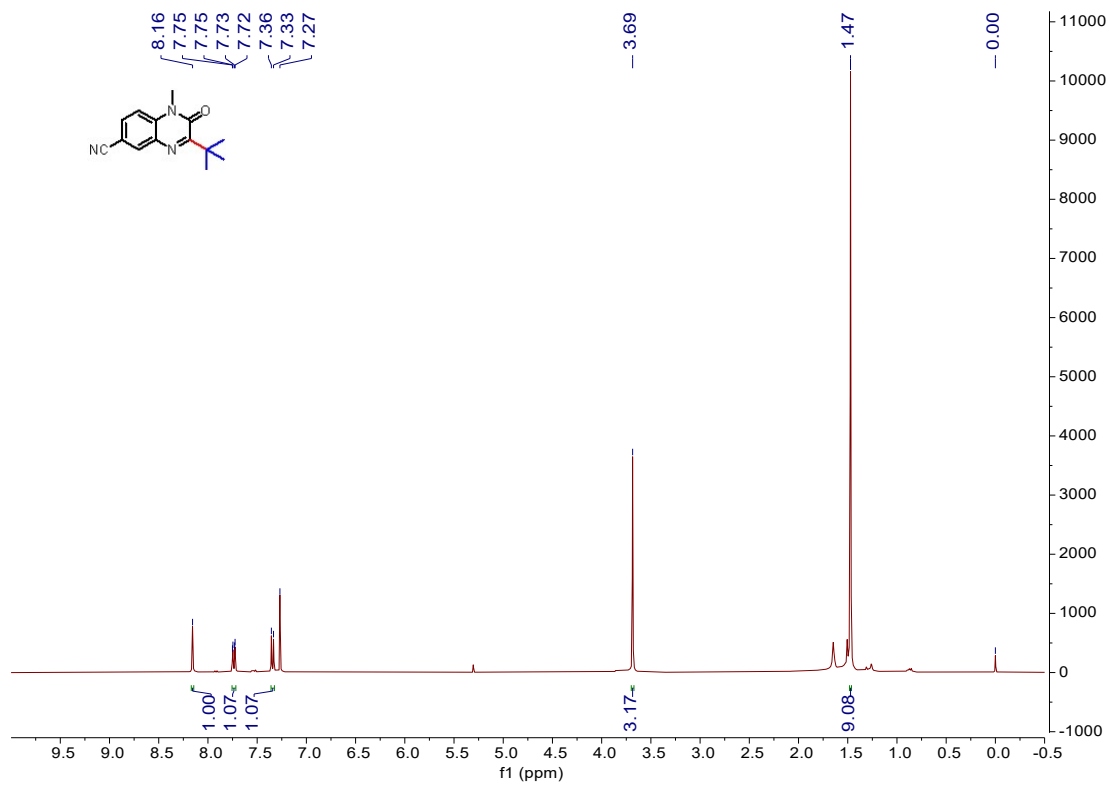
### <sup>1</sup>H NMR spectrum of compound **4ag**



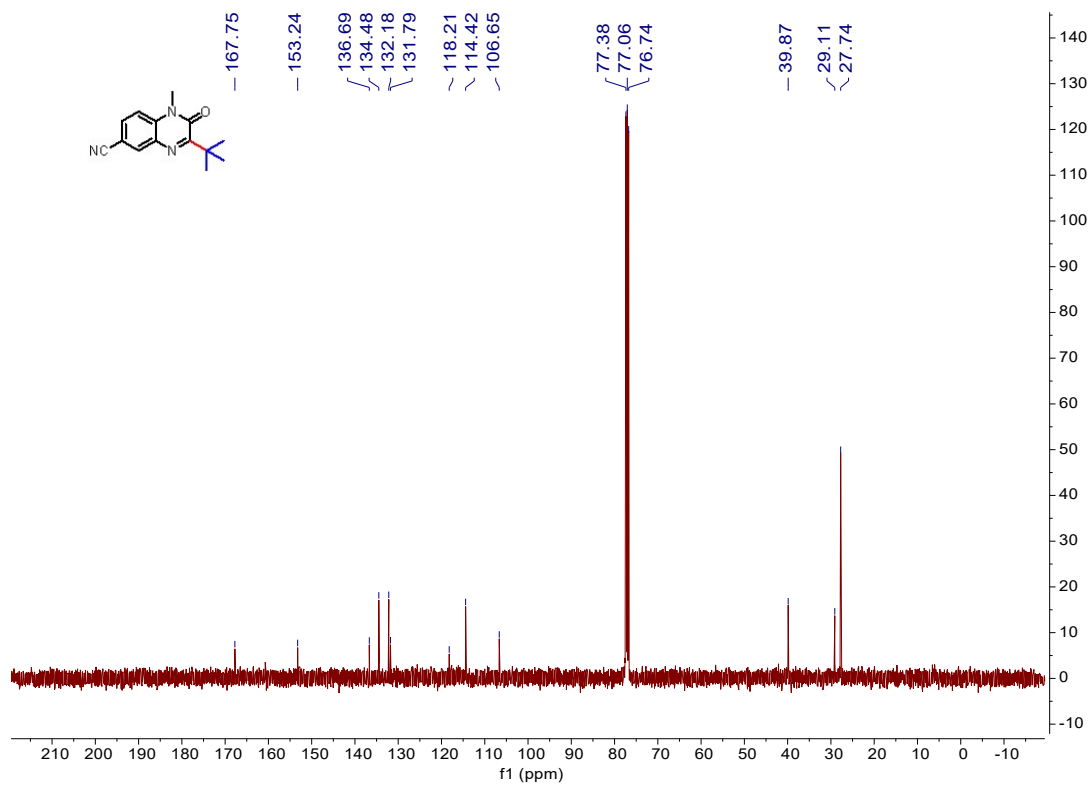
**<sup>1</sup>H NMR spectrum of compound 4ah**



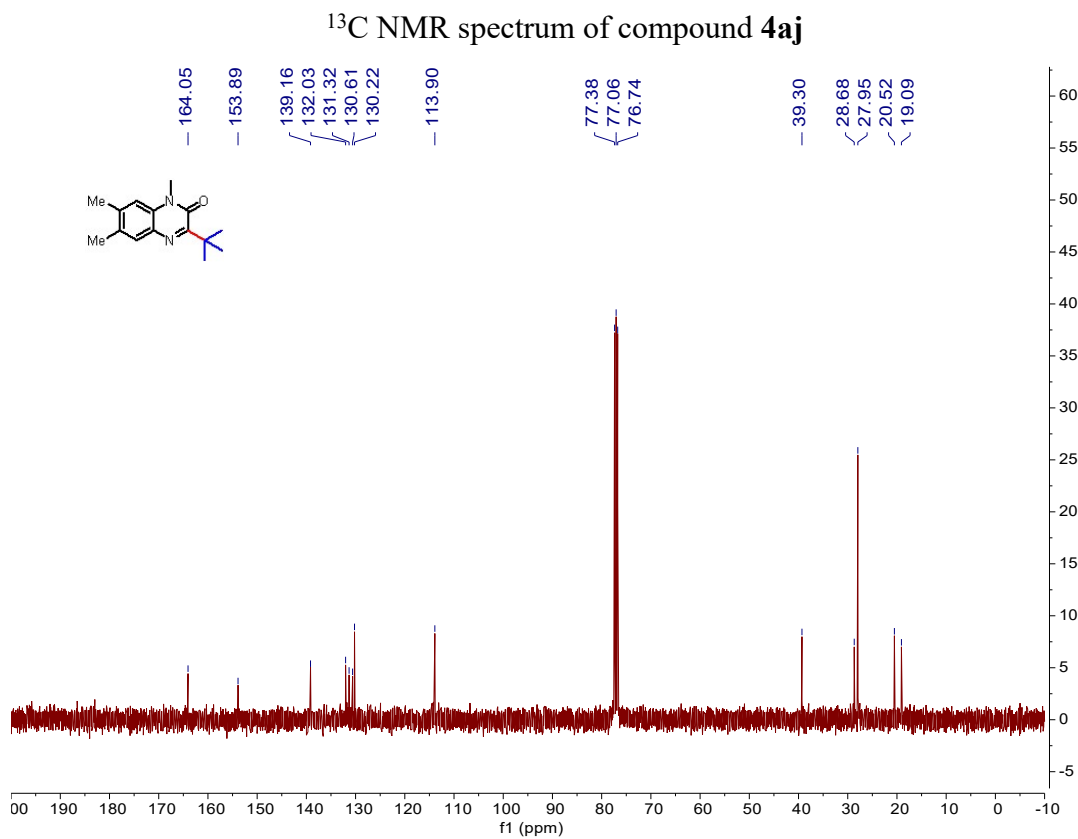
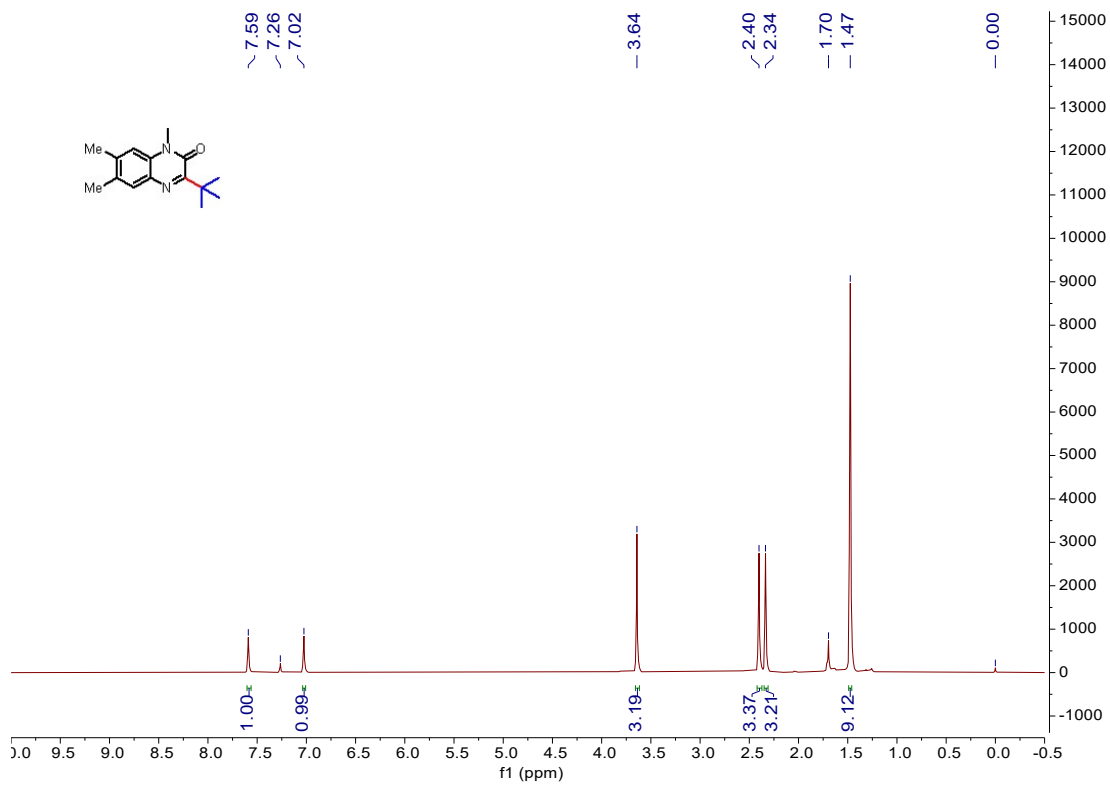
<sup>1</sup>H NMR spectrum of compound **4ai**



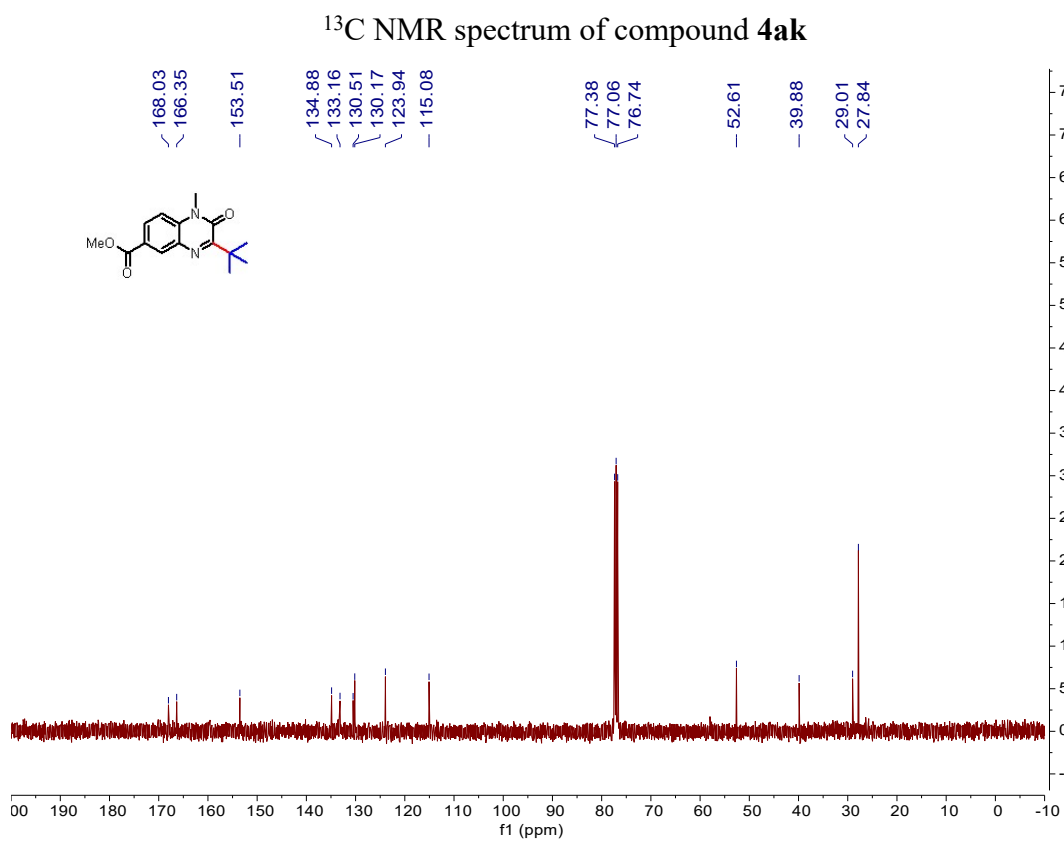
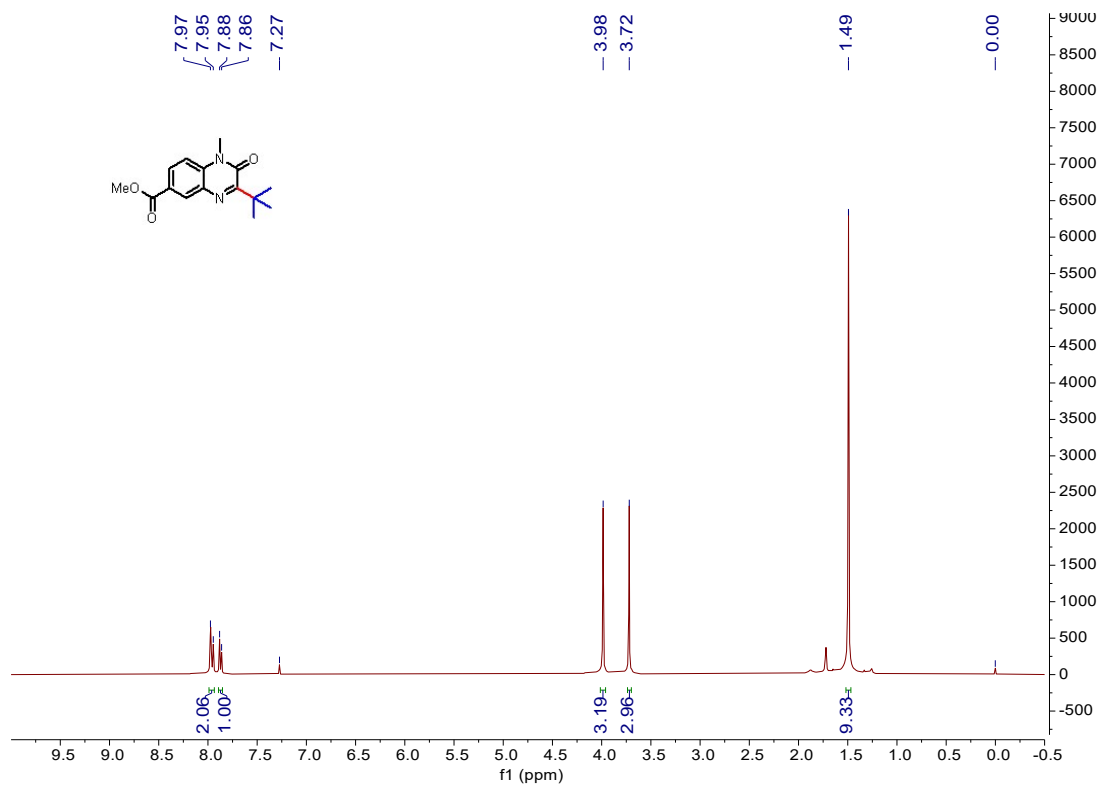
**<sup>13</sup>C NMR spectrum of compound 4ai**



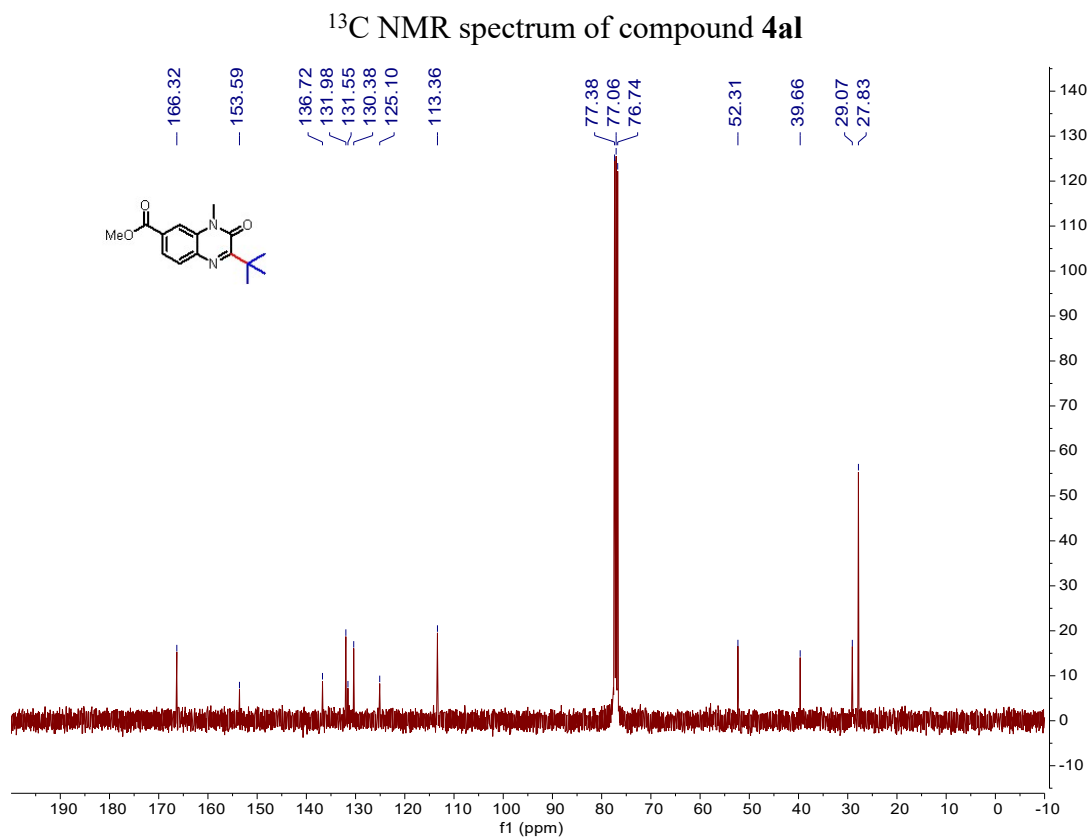
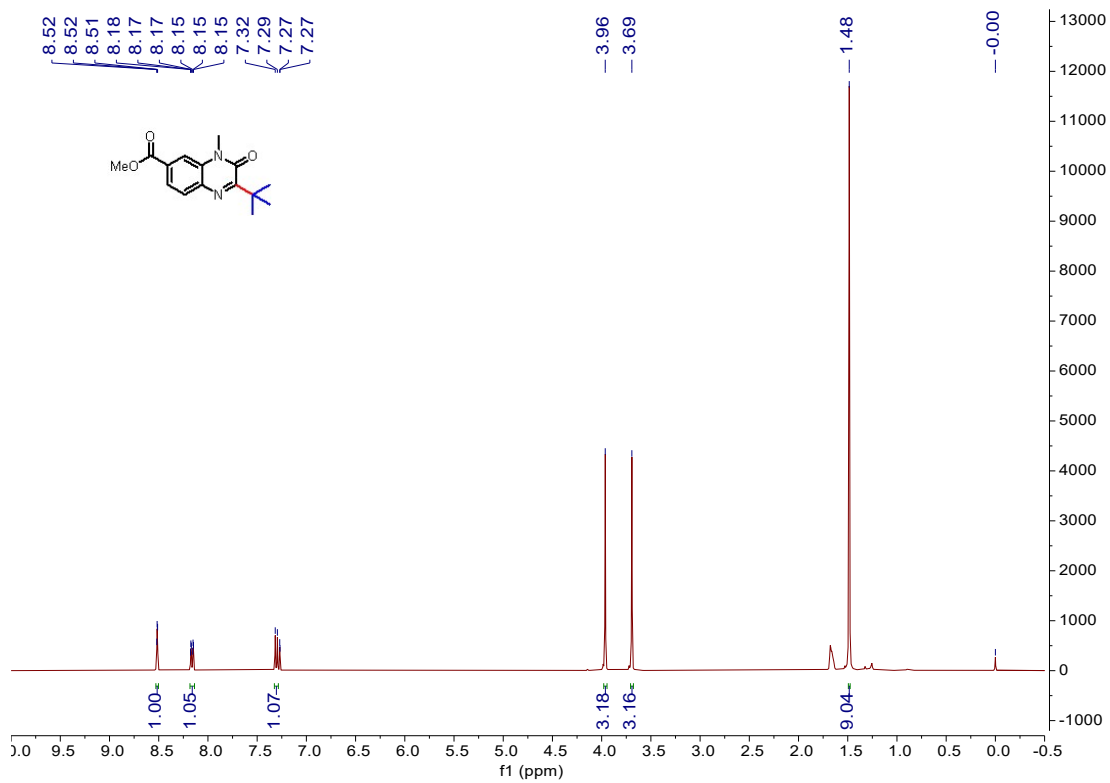
**<sup>1</sup>H NMR spectrum of compound 4aj**



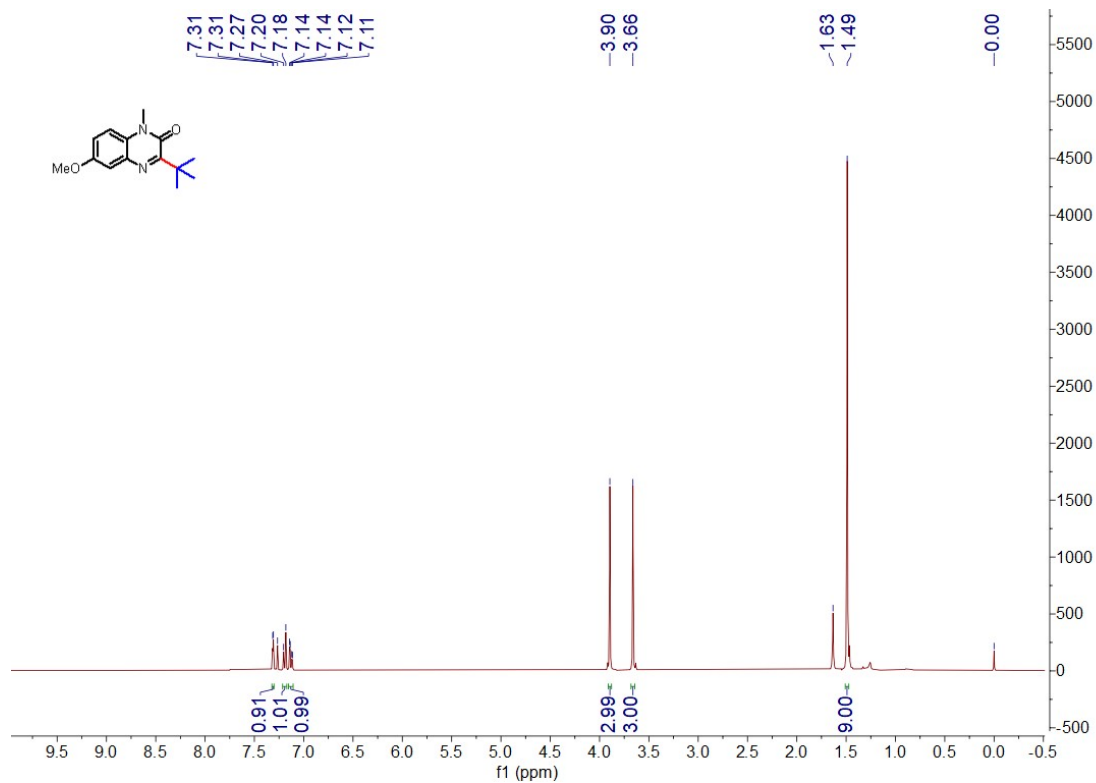
**<sup>1</sup>H NMR spectrum of compound 4ak**



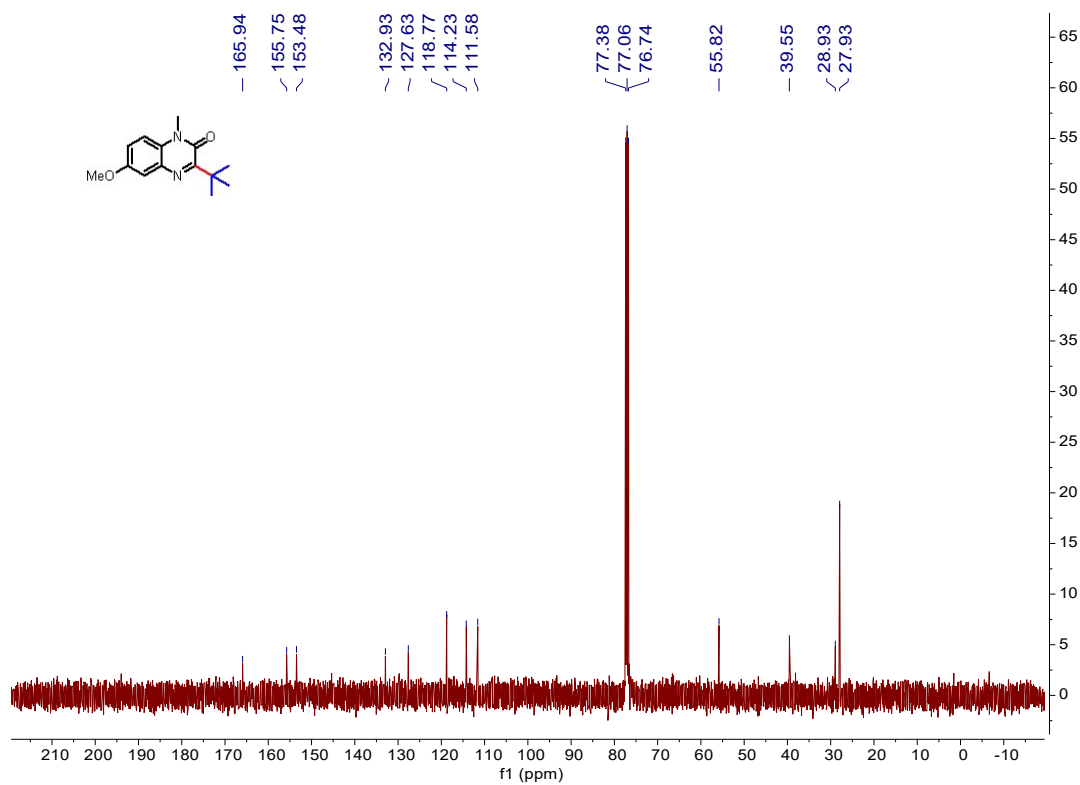
<sup>1</sup>H NMR spectrum of compound **4al**



**<sup>1</sup>H NMR spectrum of compound 4aI**

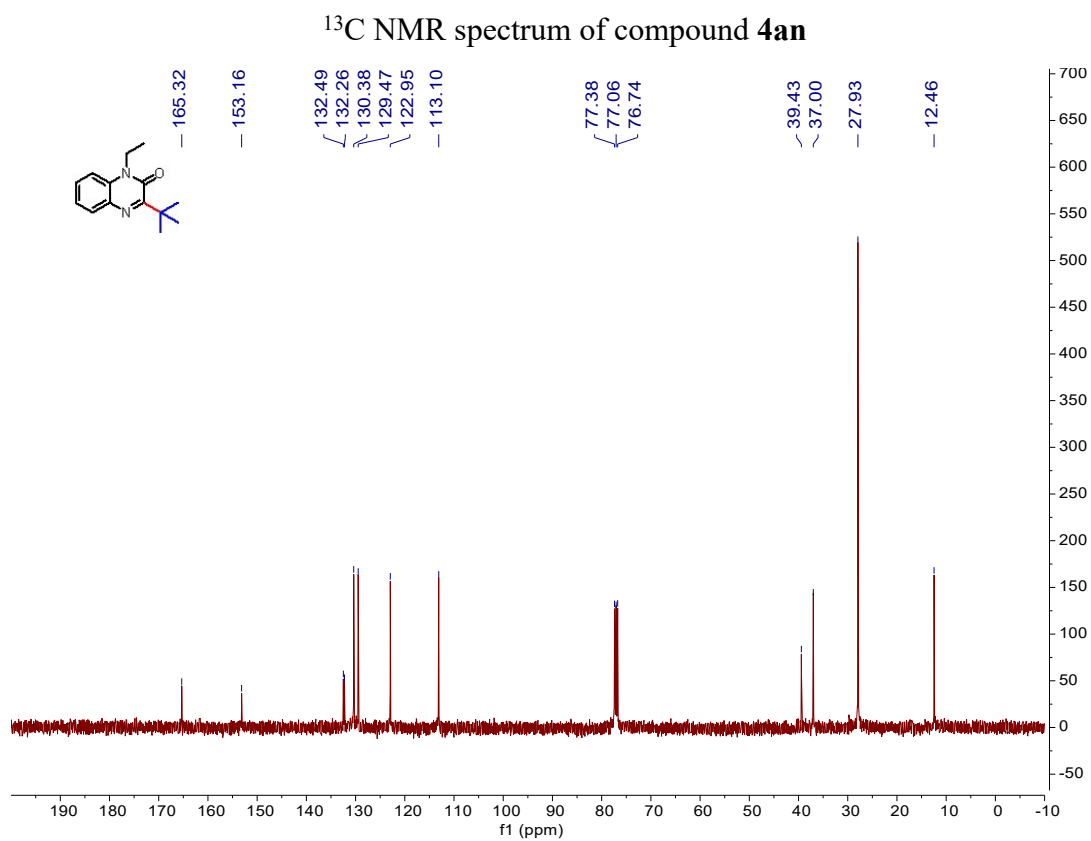
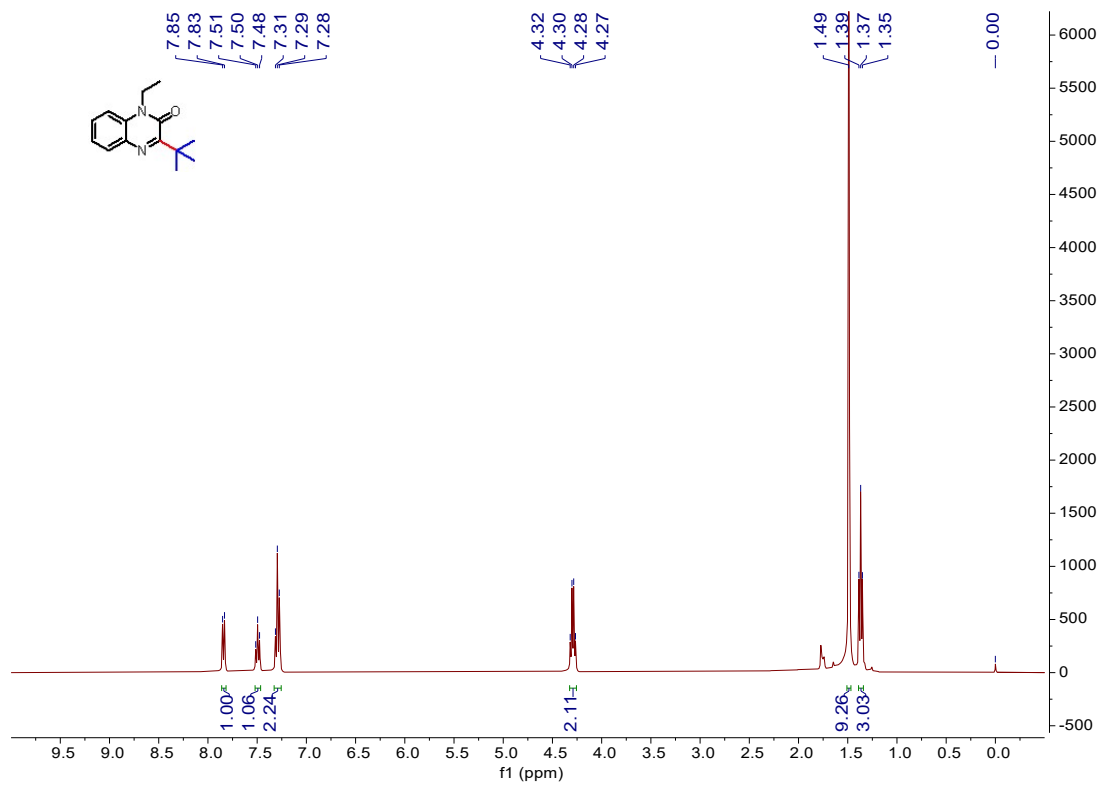


**<sup>13</sup>C NMR spectrum of compound 4am**

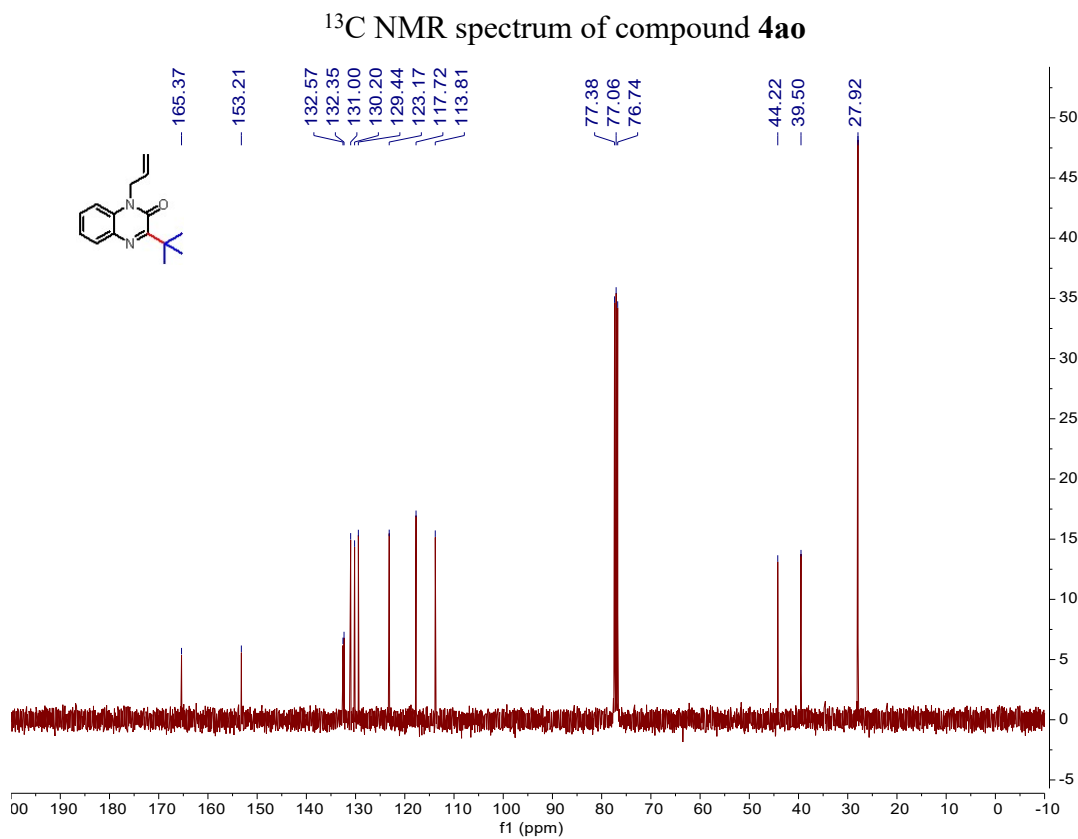
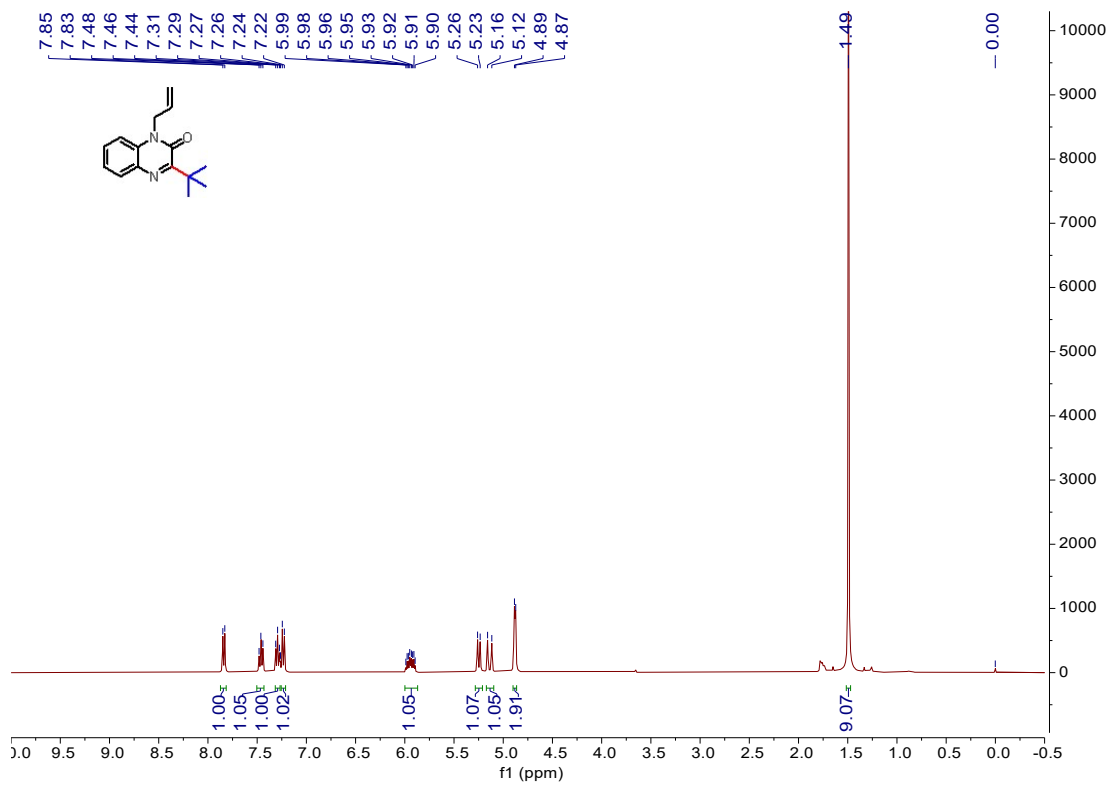


**<sup>1</sup>H NMR spectrum of compound 4an**

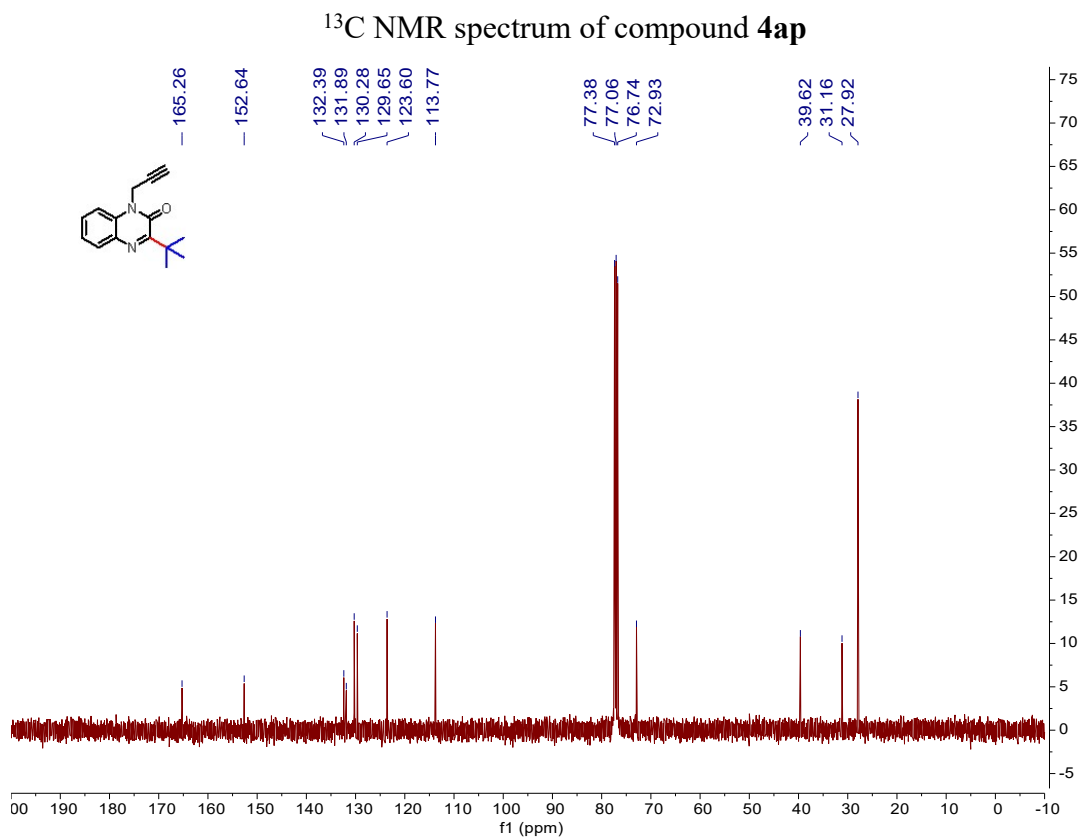
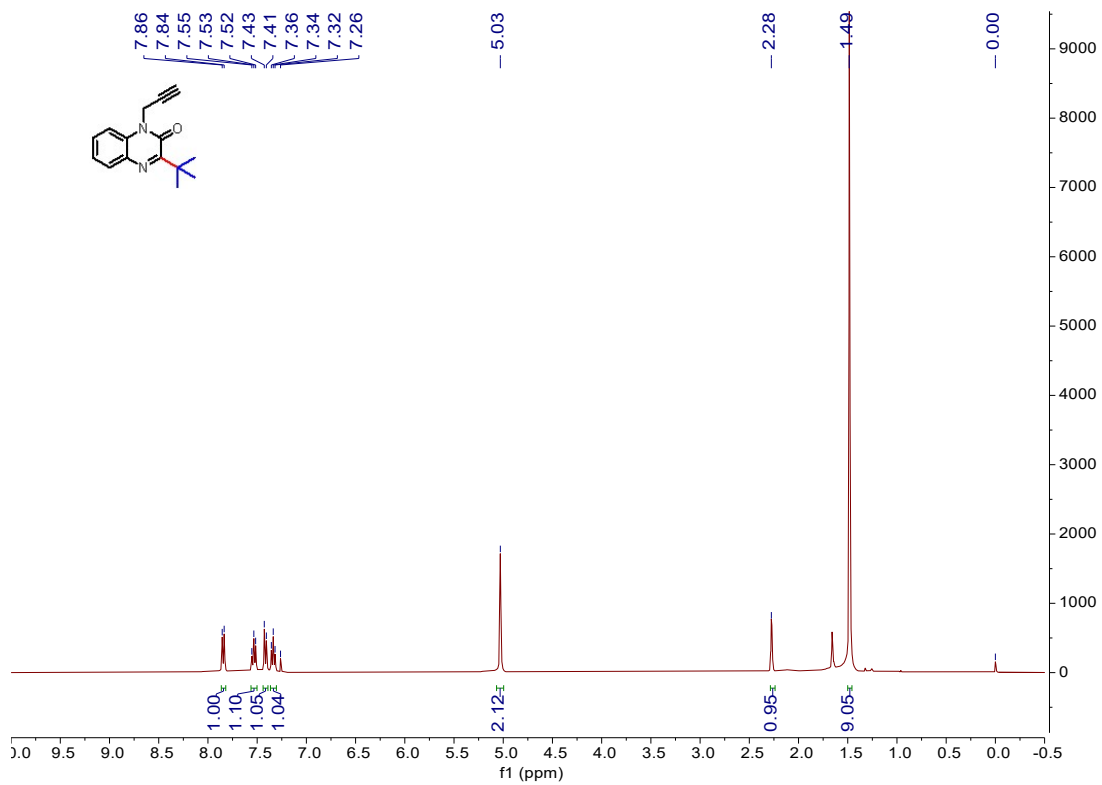




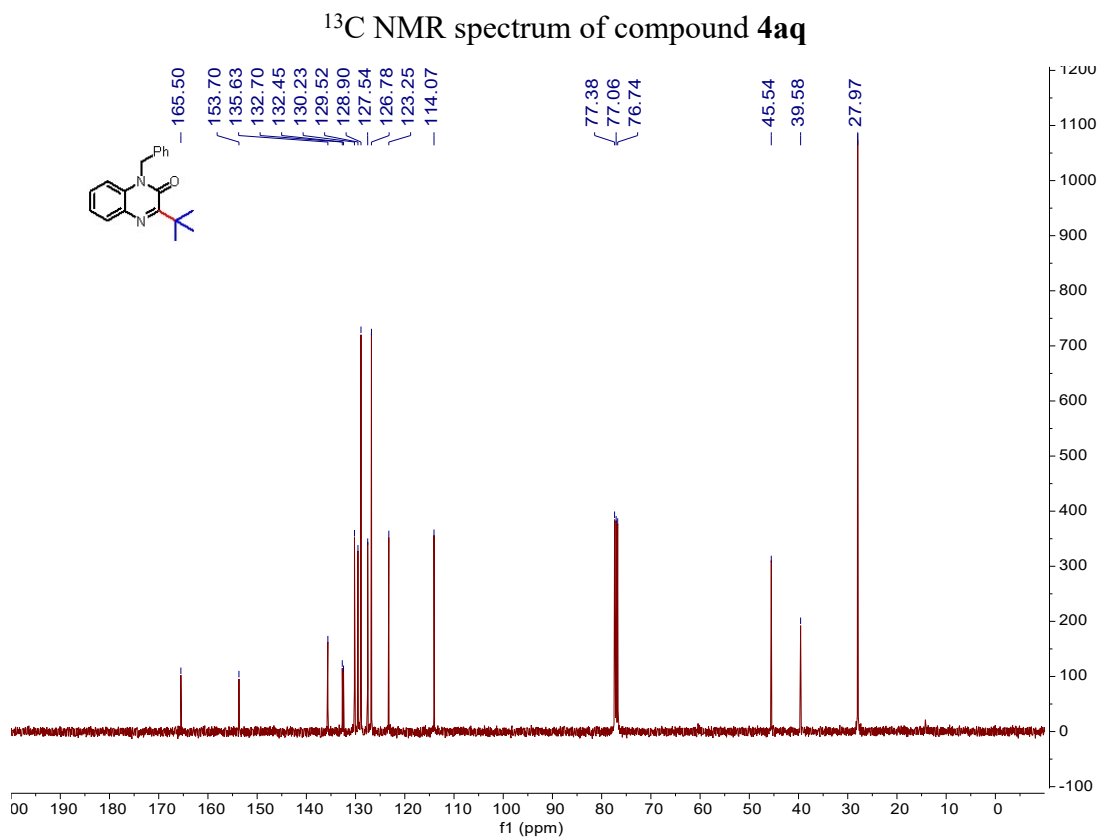
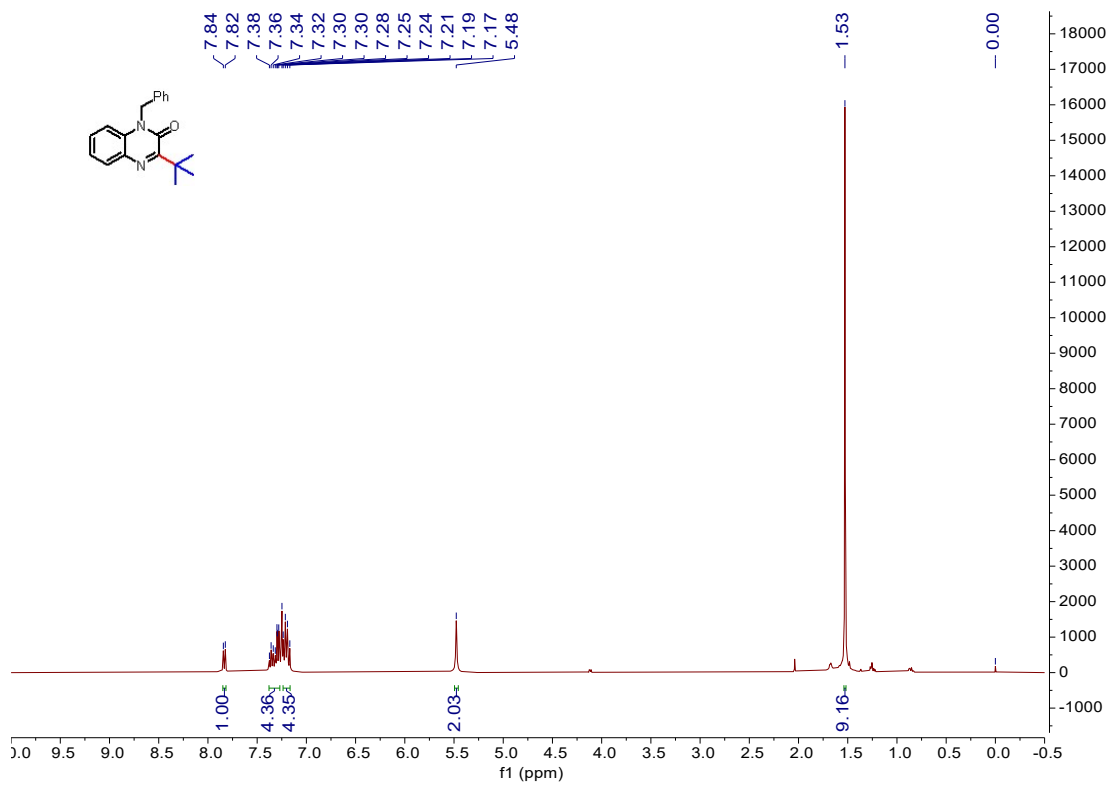
**<sup>1</sup>H NMR spectrum of compound 4ao**



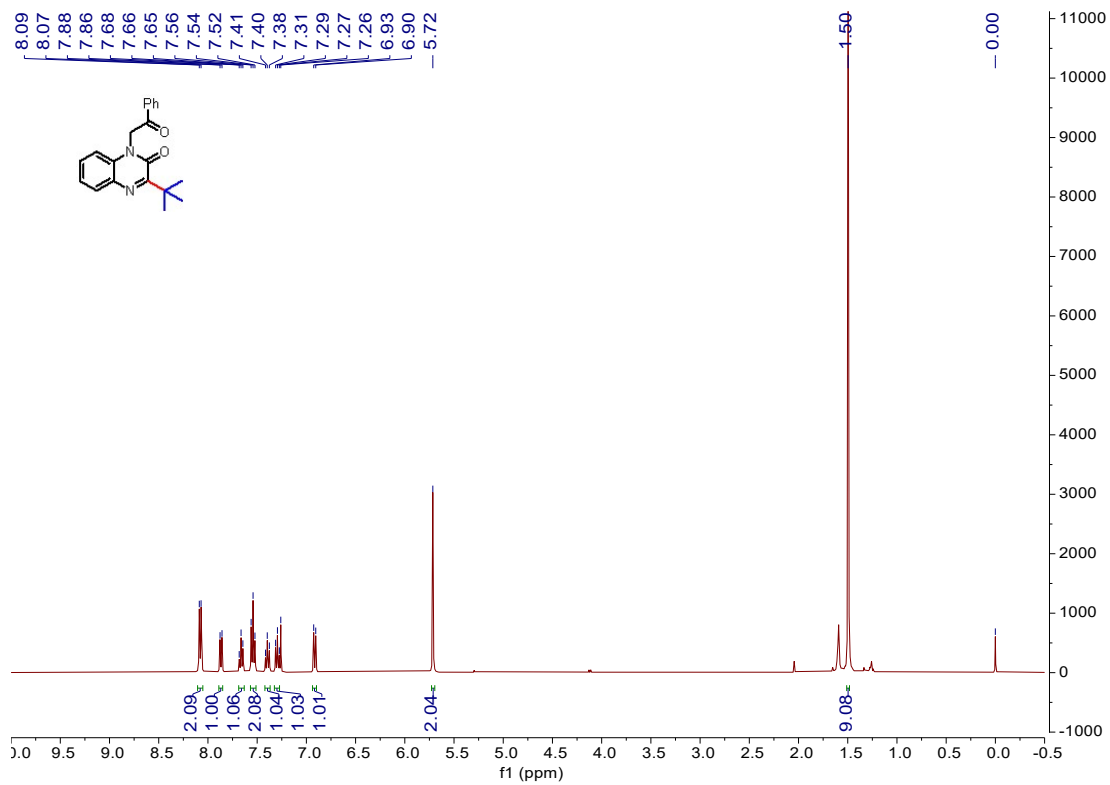
**<sup>1</sup>H NMR spectrum of compound 4ap**



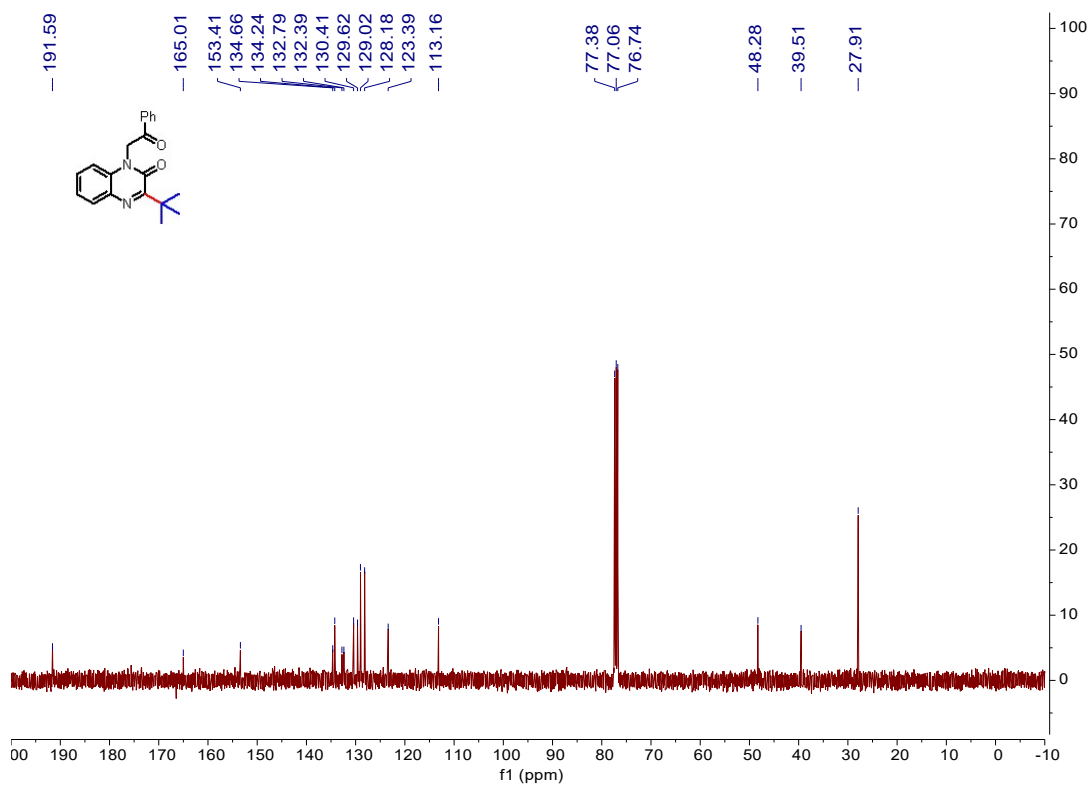
**<sup>1</sup>H NMR spectrum of compound 4aq**



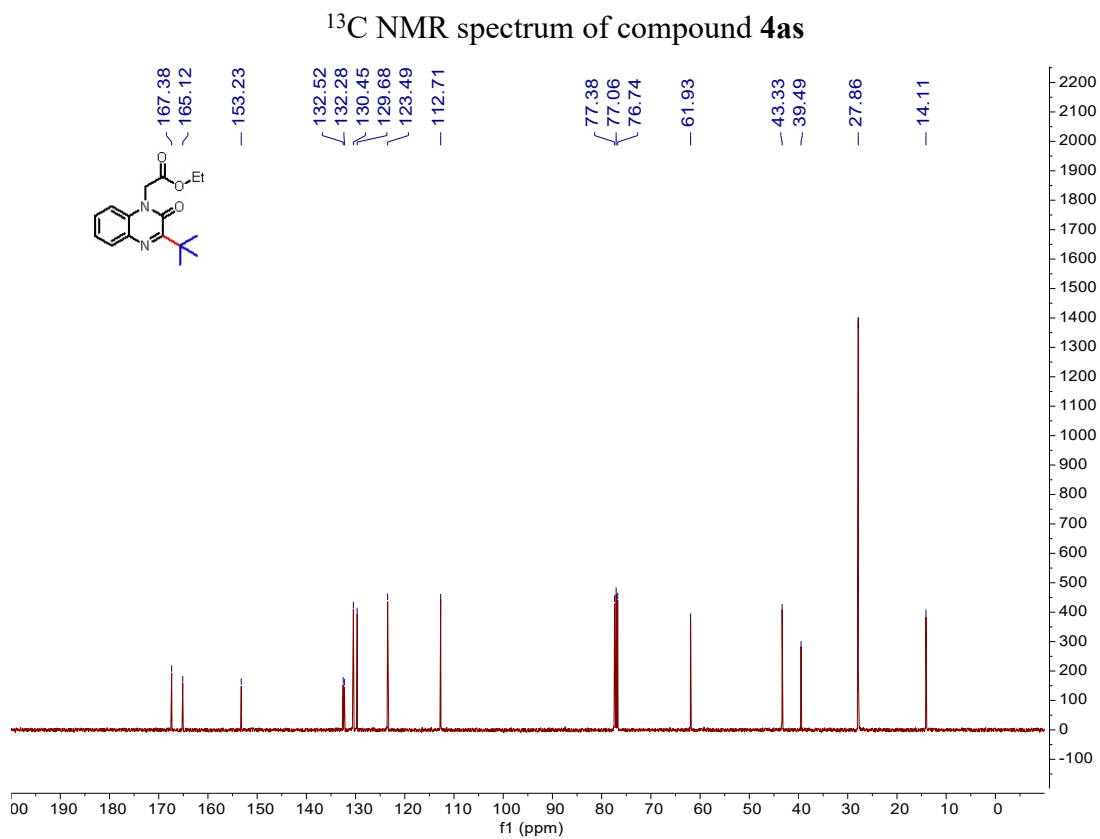
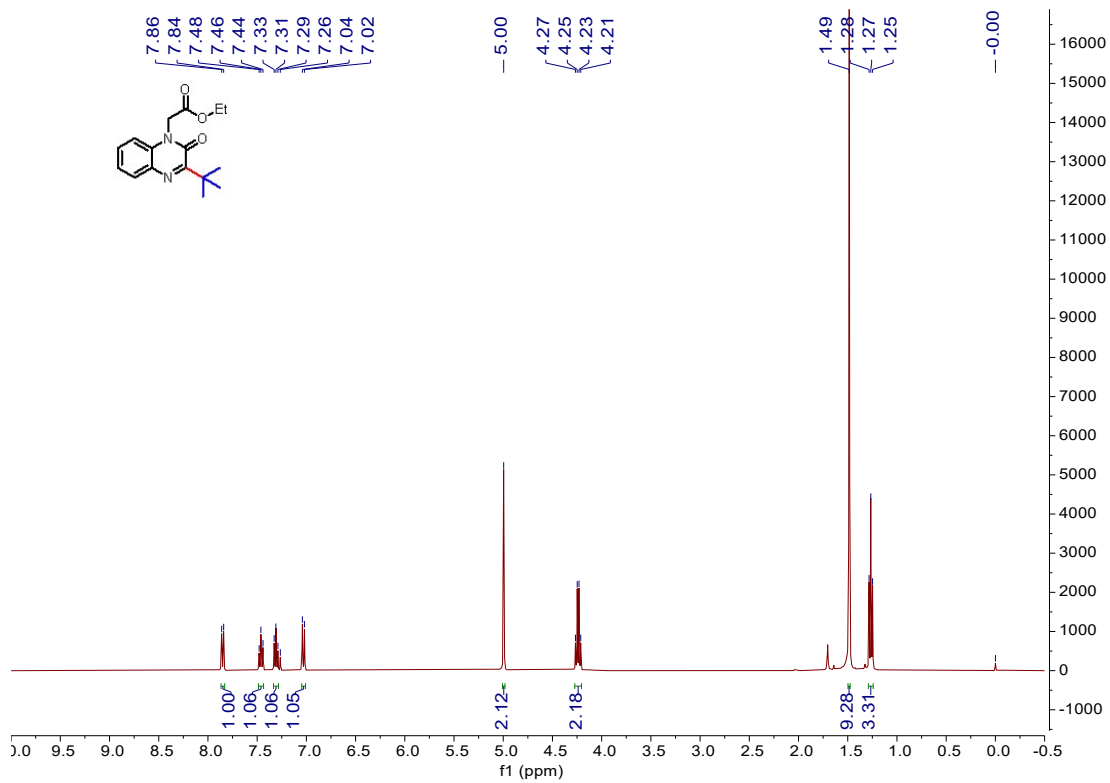
**<sup>1</sup>H NMR spectrum of compound 4ar**



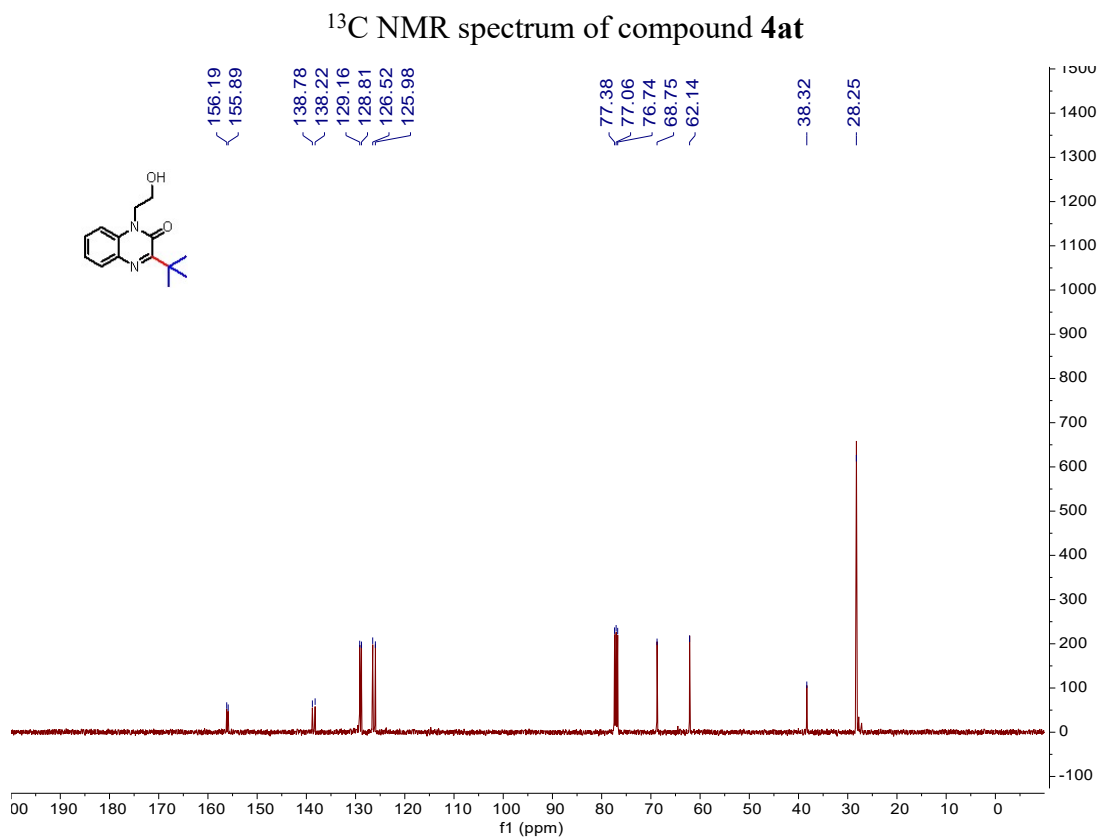
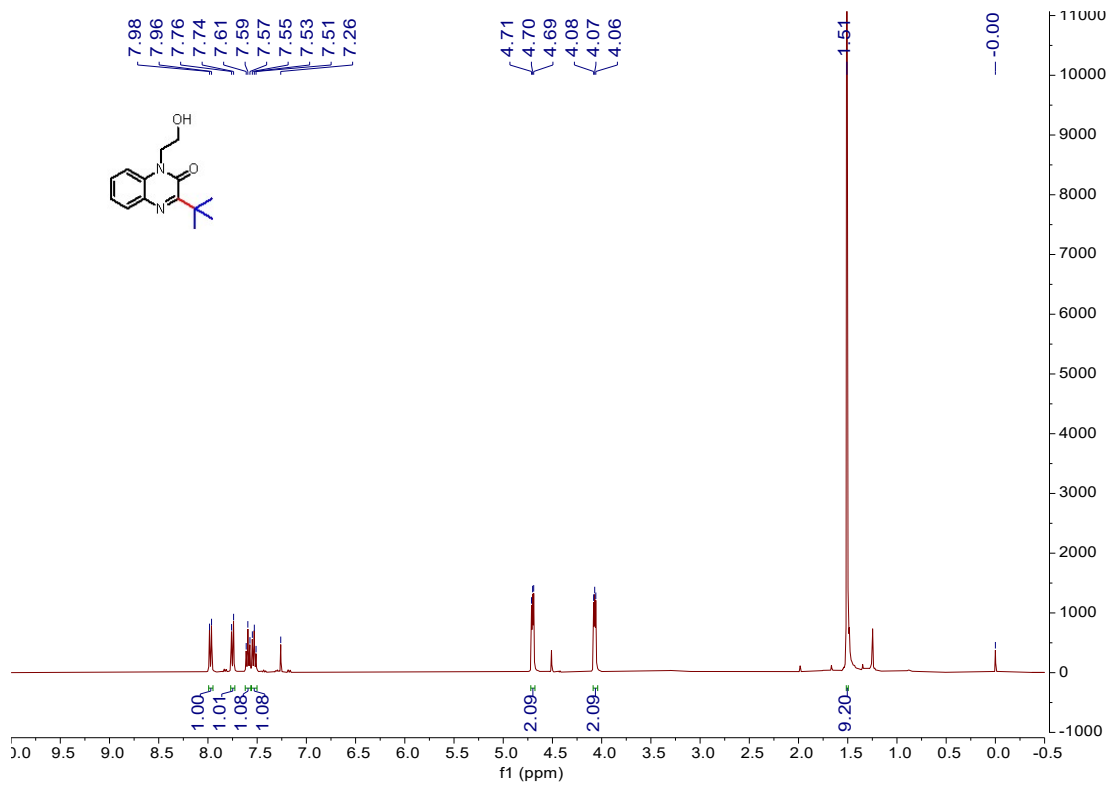
**<sup>13</sup>C NMR spectrum of compound 4ar**



**<sup>1</sup>H NMR spectrum of compound 4as**

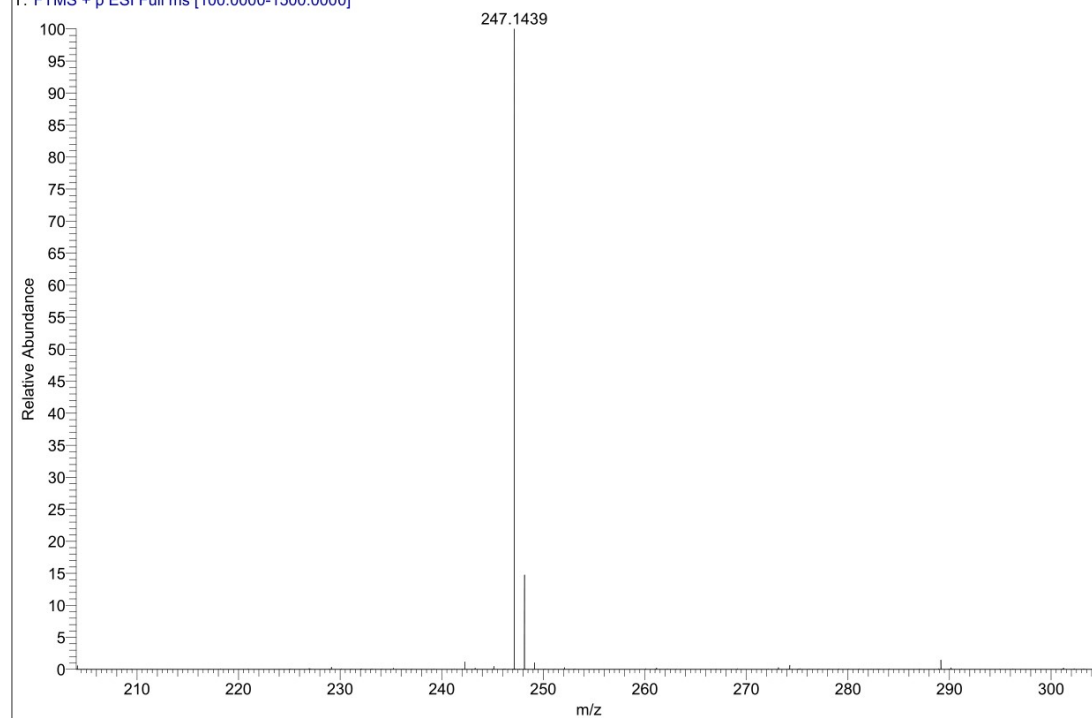


**<sup>1</sup>H NMR spectrum of compound 4at**

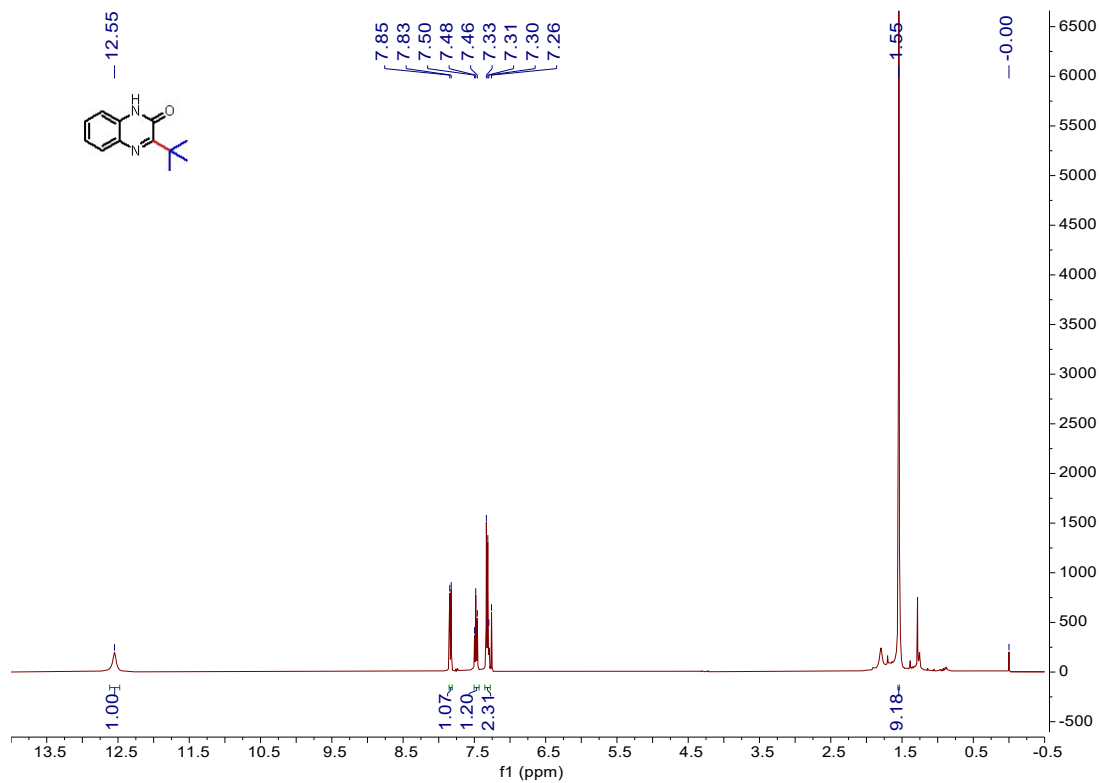


HRMS of compound 4at

SXD-04-57 #30-36 RT: 0.13-0.16 AV: 7 NL: 3.89E9  
T: FTMS + p ESI Full ms [100.0000-1500.0000]

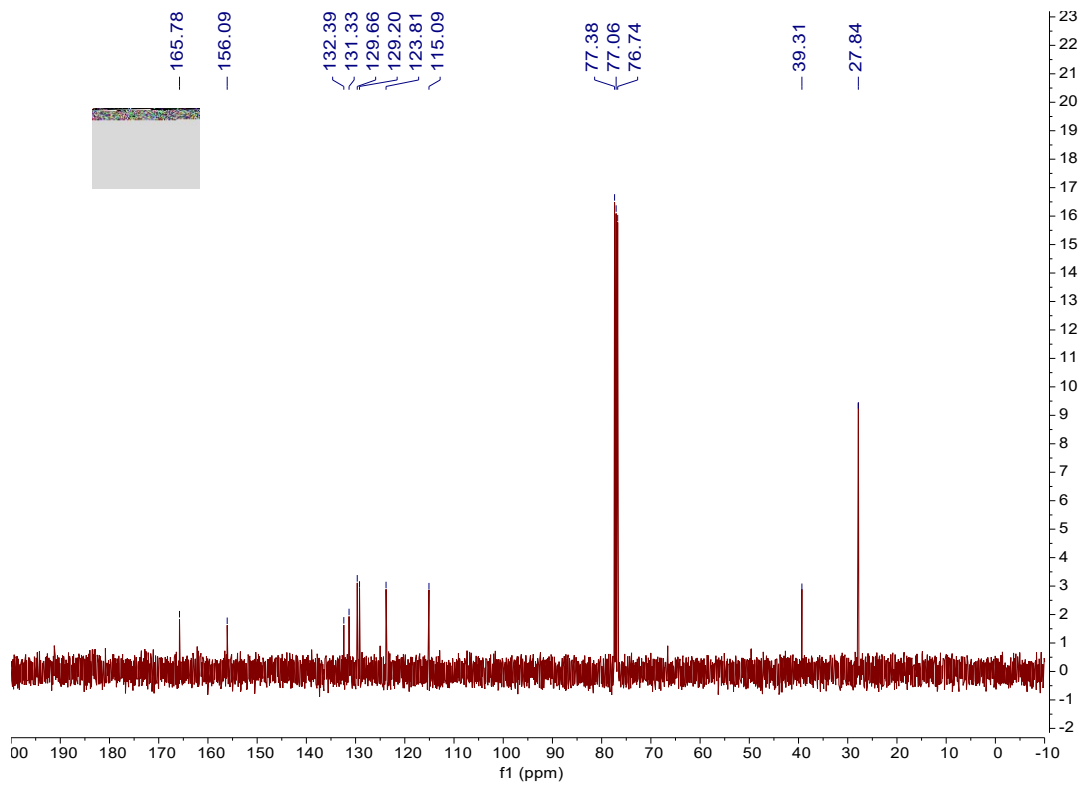


### <sup>1</sup>H NMR spectrum of compound **4au**

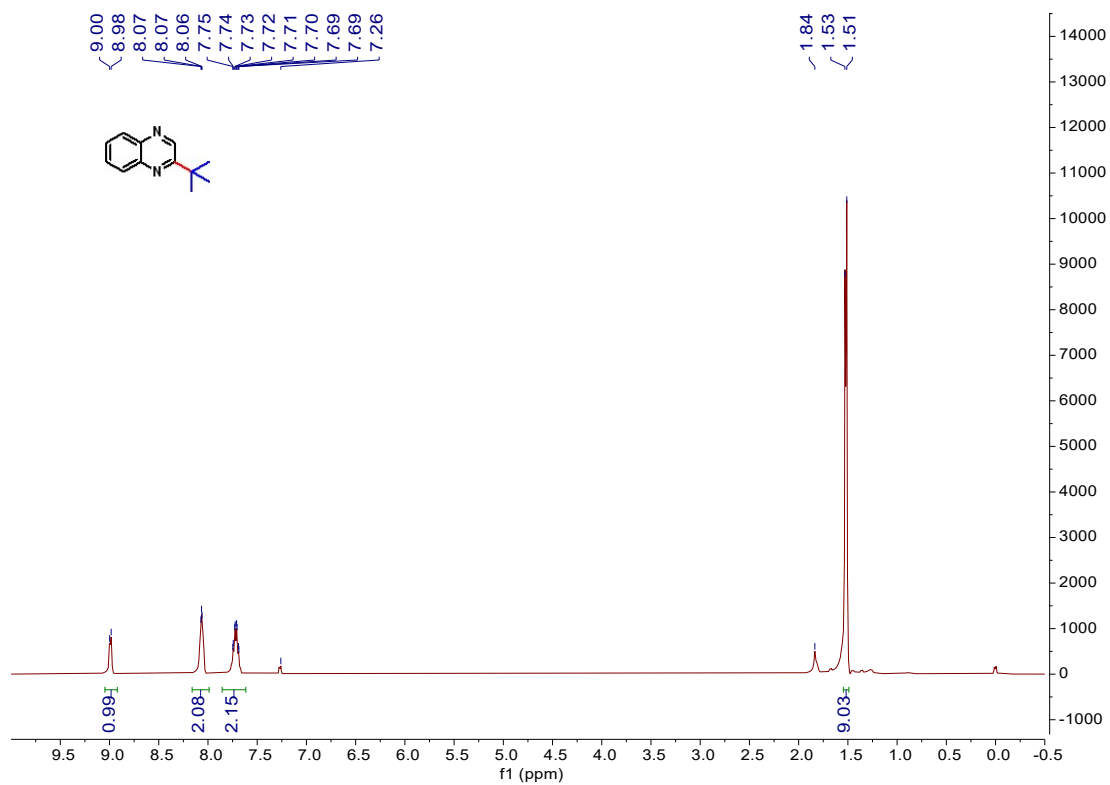


### <sup>13</sup>C NMR spectrum of compound **4au**

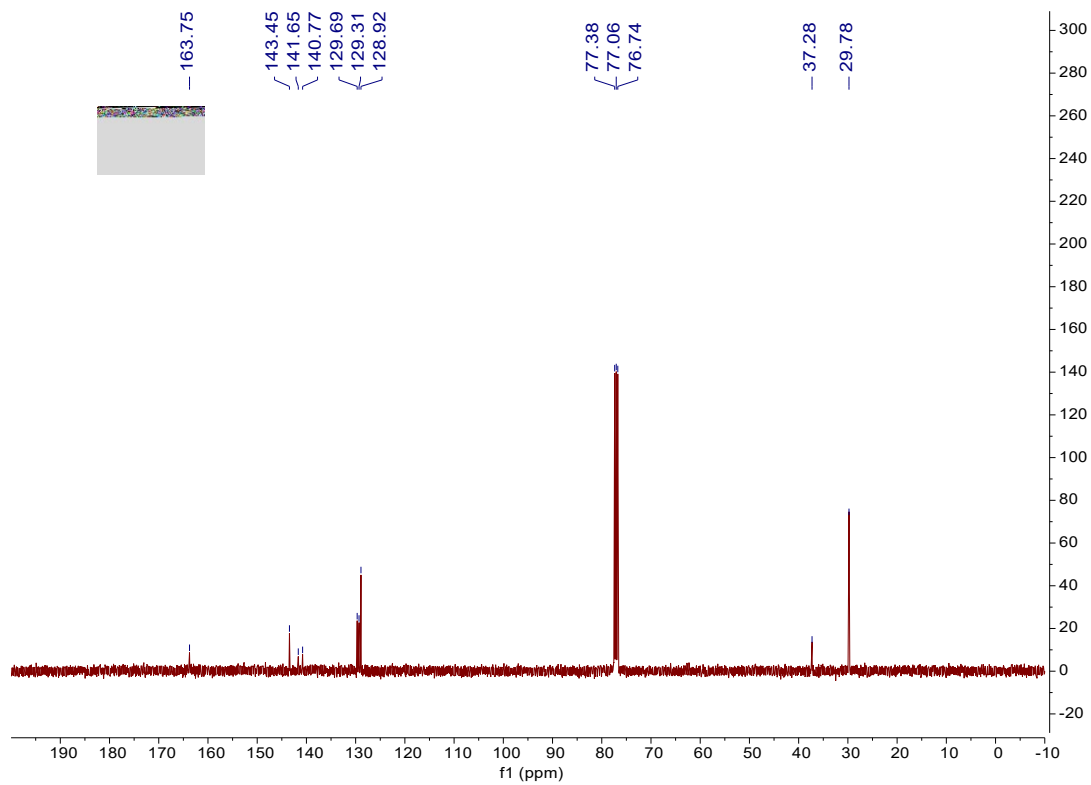




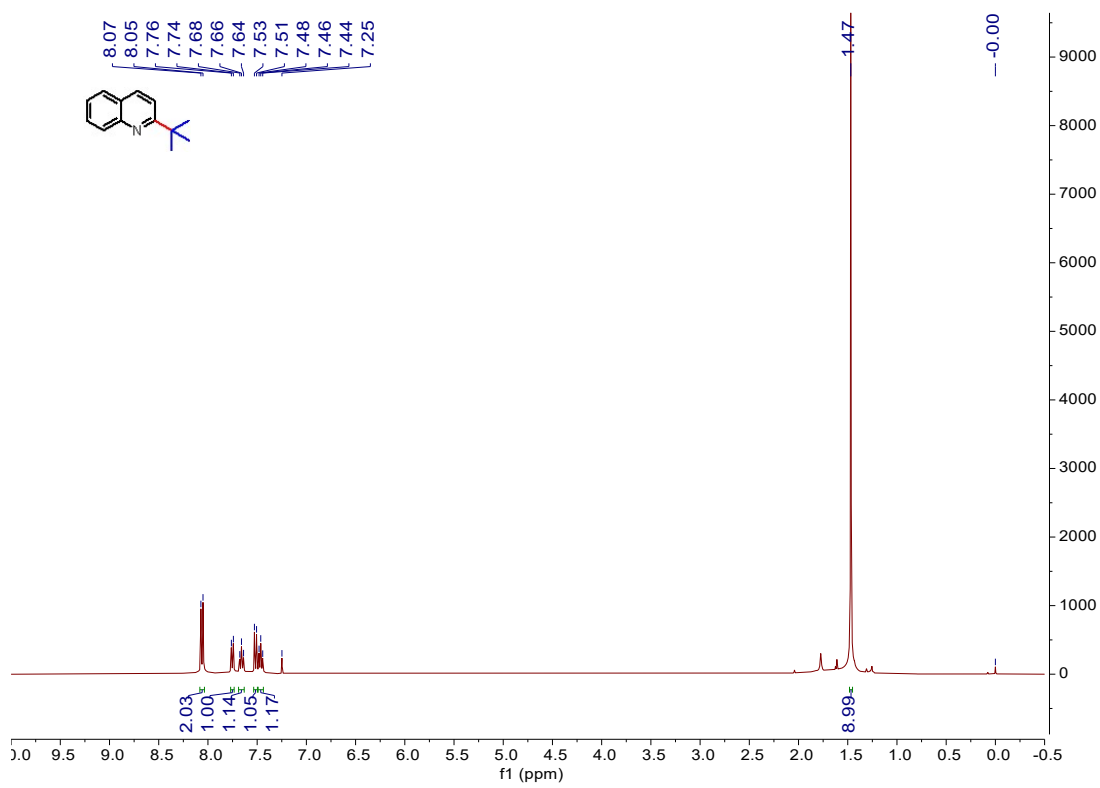
<sup>1</sup>H NMR spectrum of compound 4av



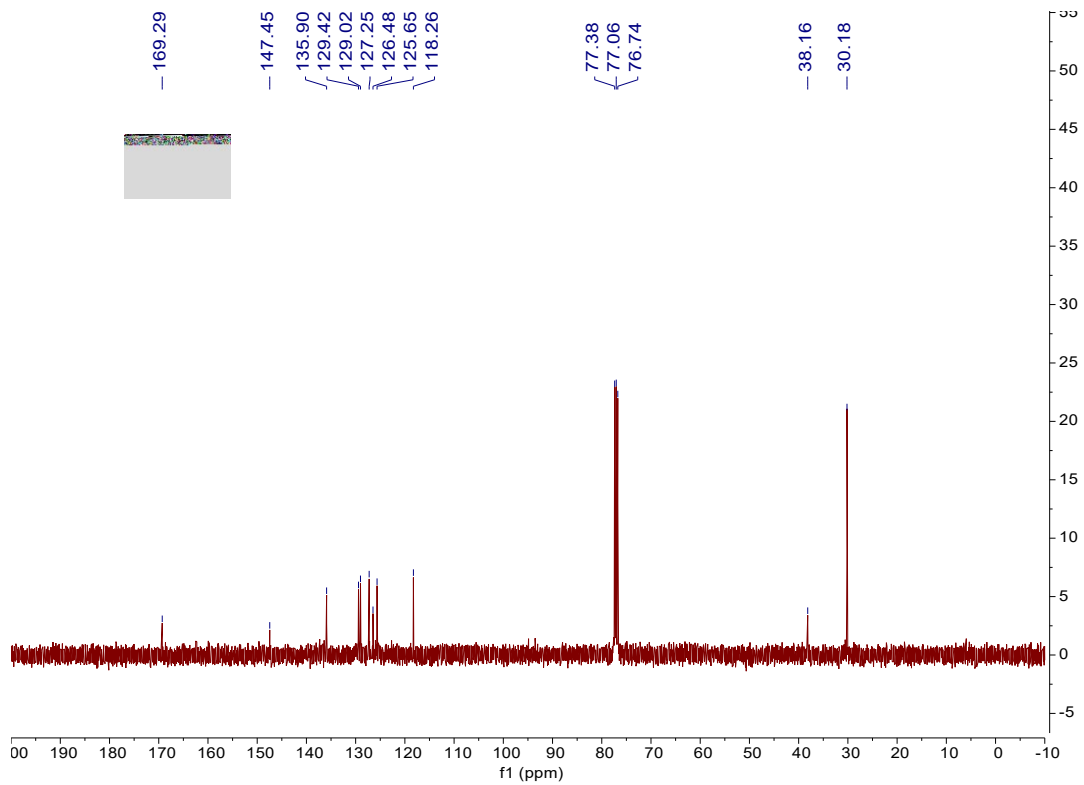
<sup>13</sup>C NMR spectrum of compound 4av



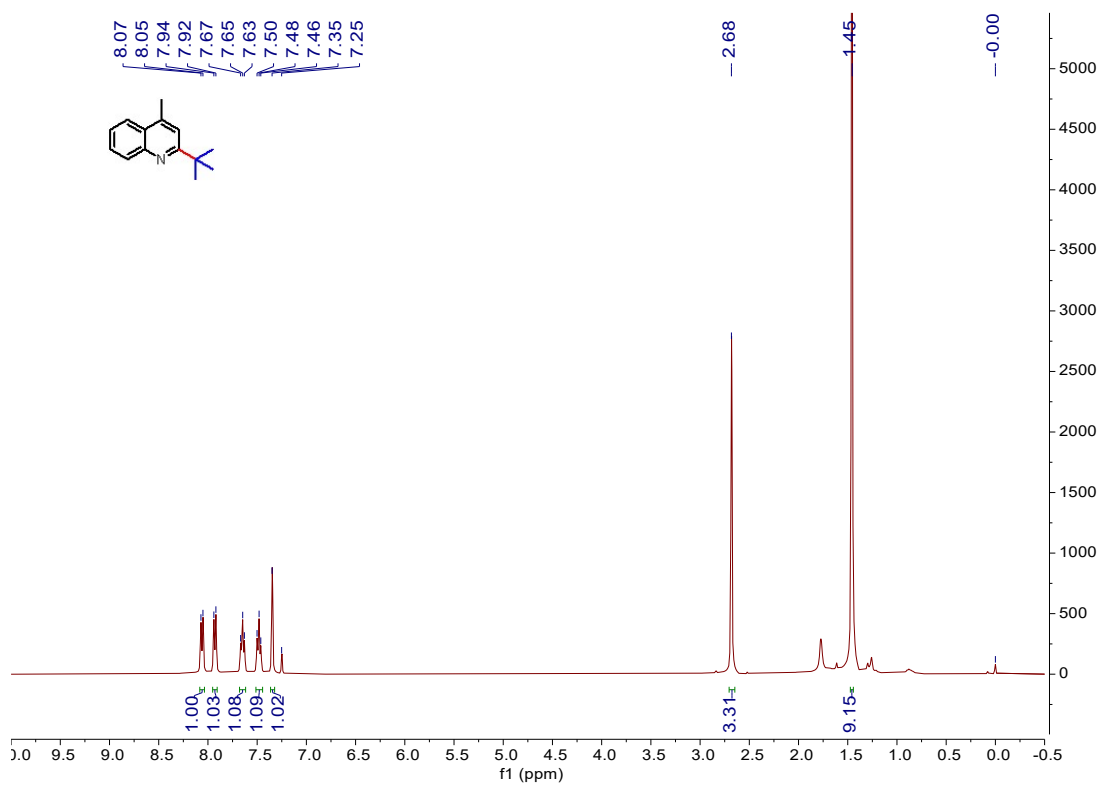
<sup>1</sup>H NMR spectrum of compound **4aw**



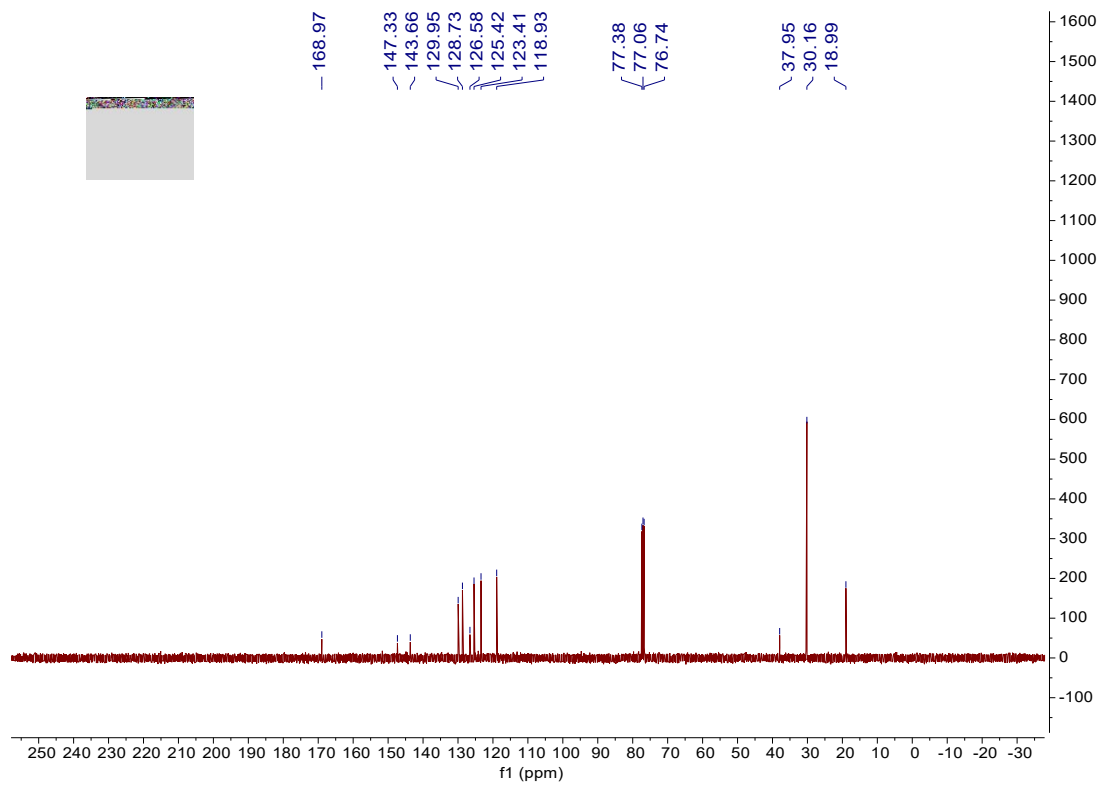
<sup>13</sup>C NMR spectrum of compound **4aw**



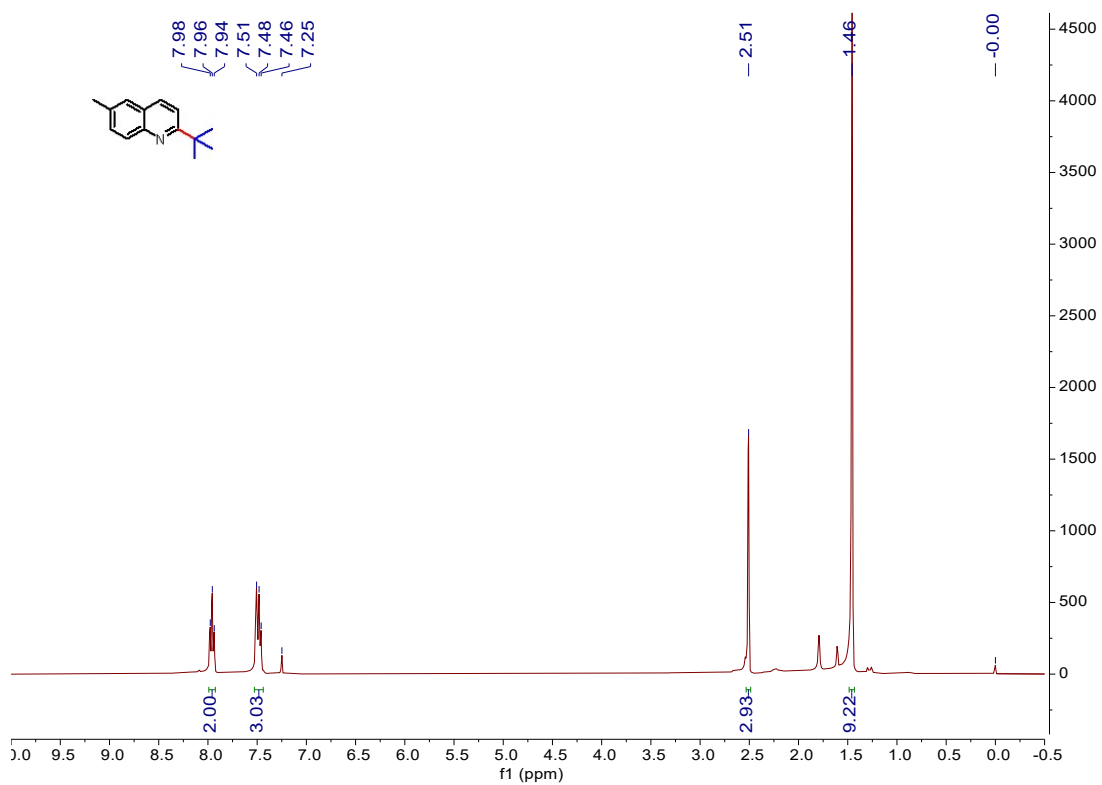
<sup>1</sup>H NMR spectrum of compound 4ax



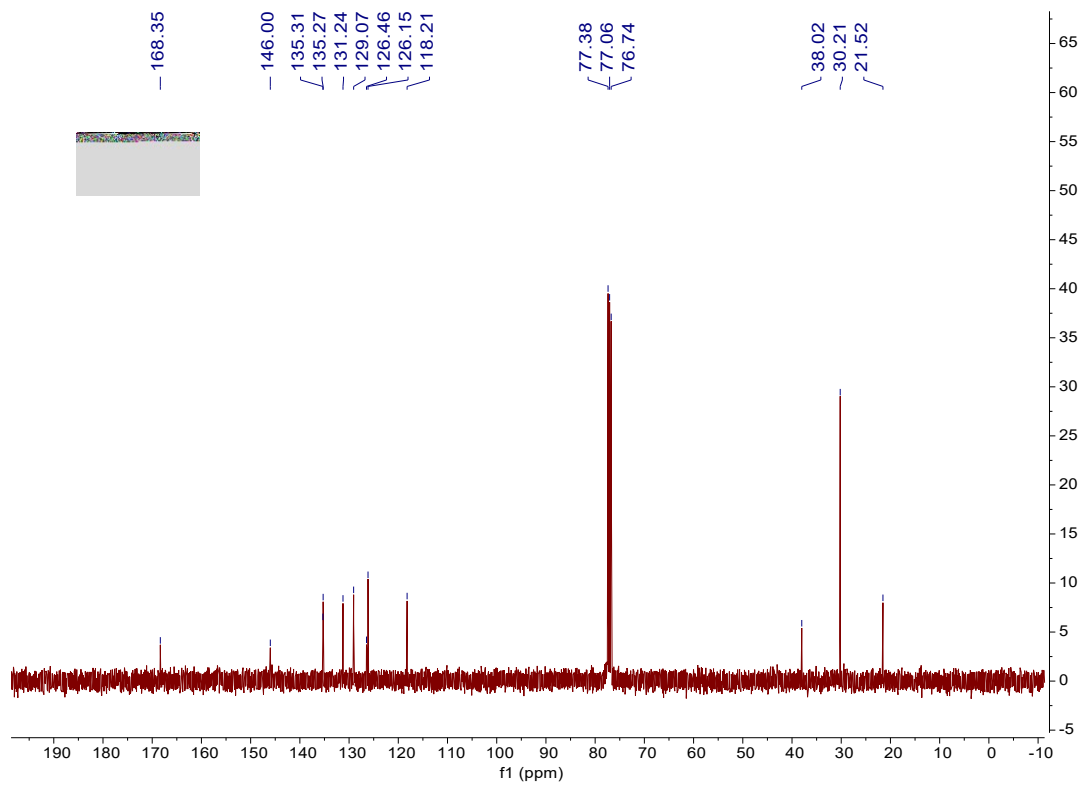
<sup>13</sup>C NMR spectrum of compound 4ax



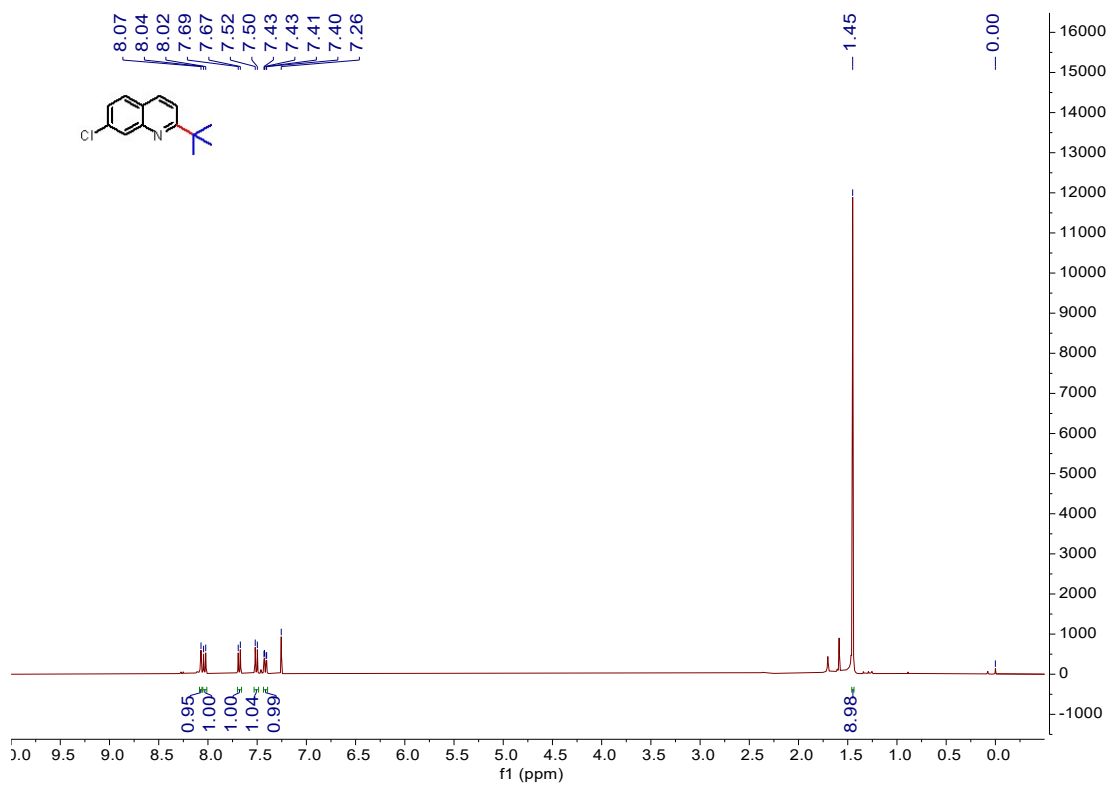
<sup>1</sup>H NMR spectrum of compound 4ay



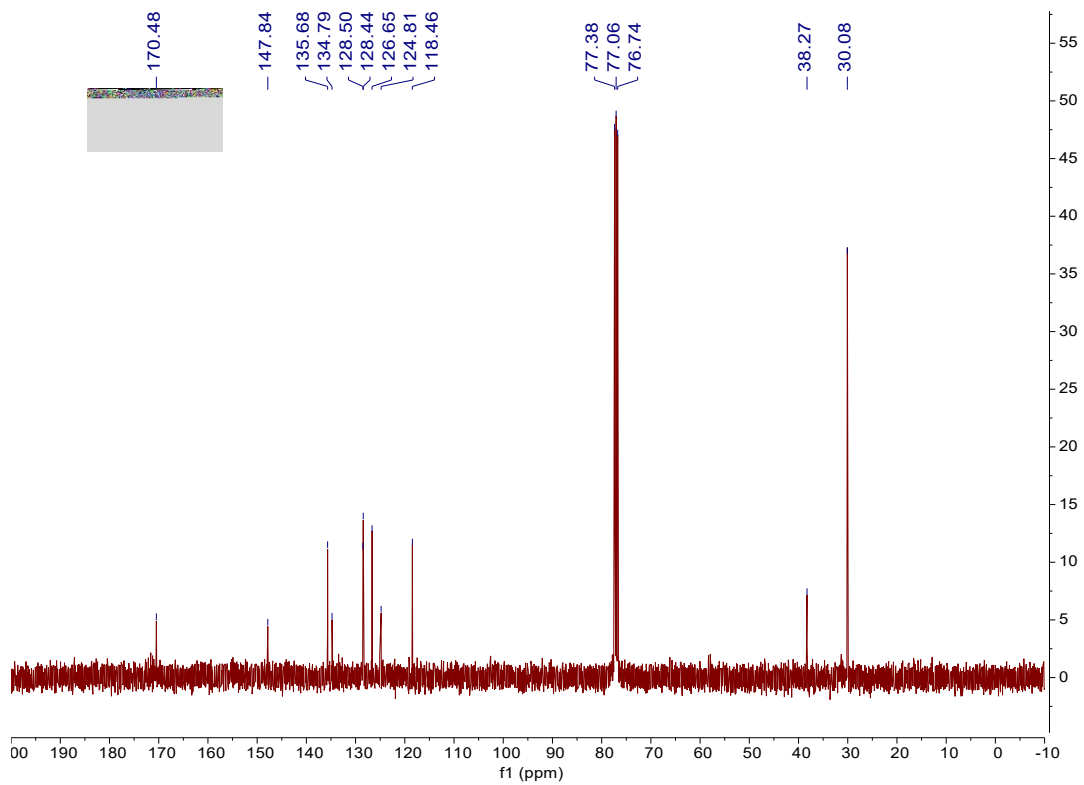
<sup>13</sup>C NMR spectrum of compound 4ay



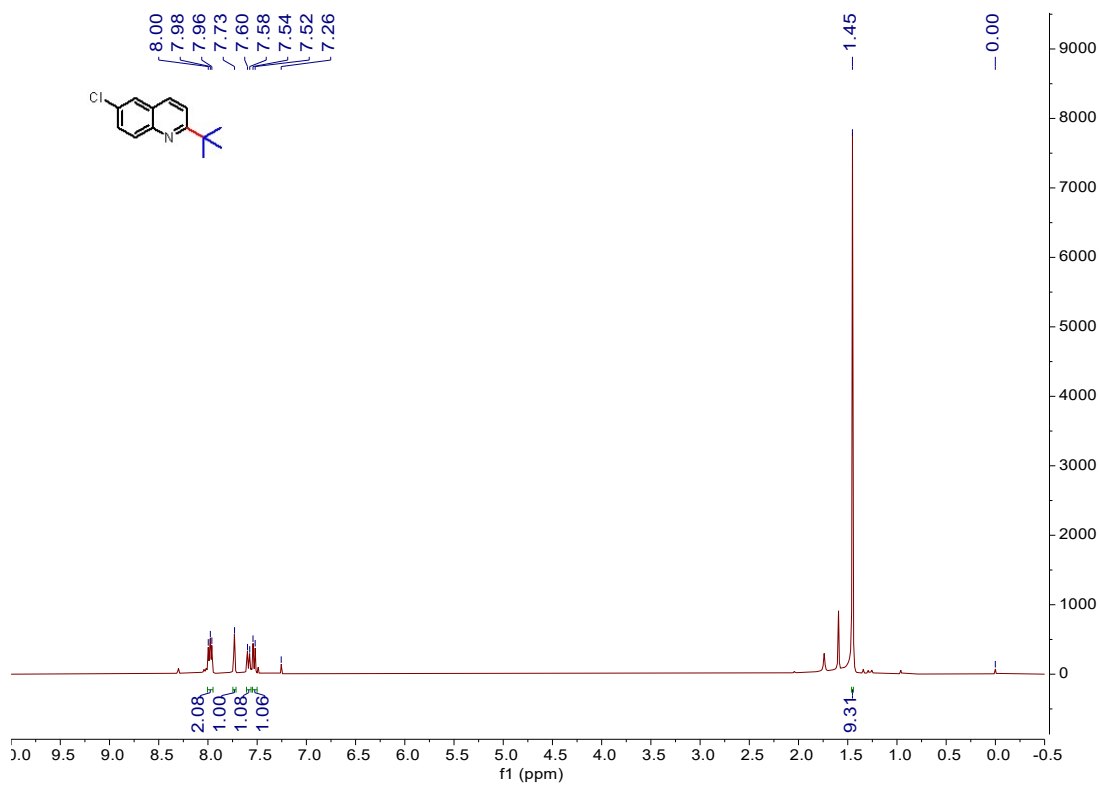
<sup>1</sup>H NMR spectrum of compound **4az**



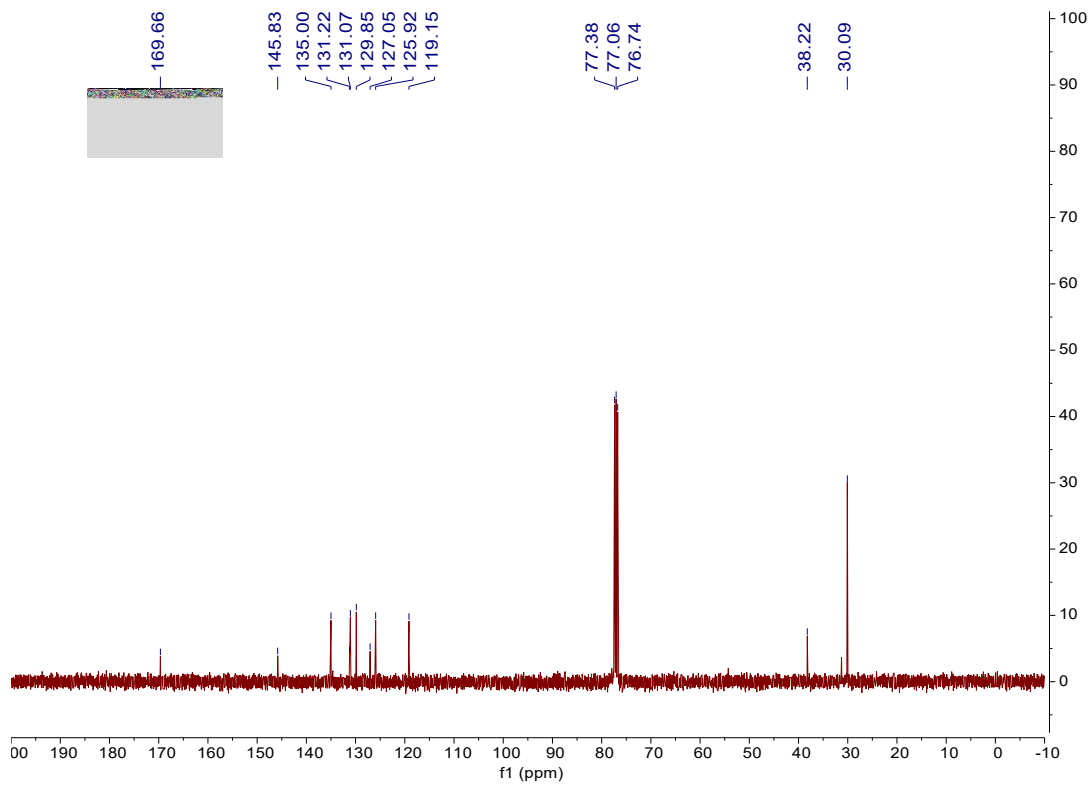
<sup>13</sup>C NMR spectrum of compound **4az**



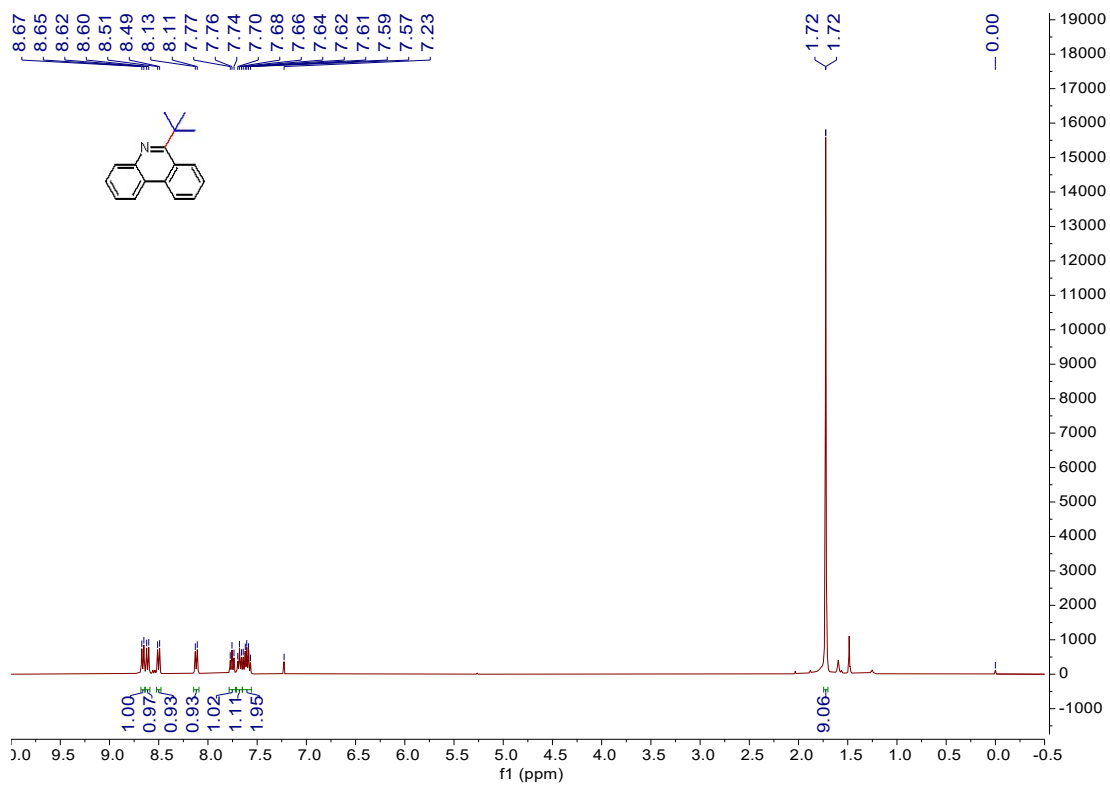
<sup>1</sup>H NMR spectrum of compound **4ba**



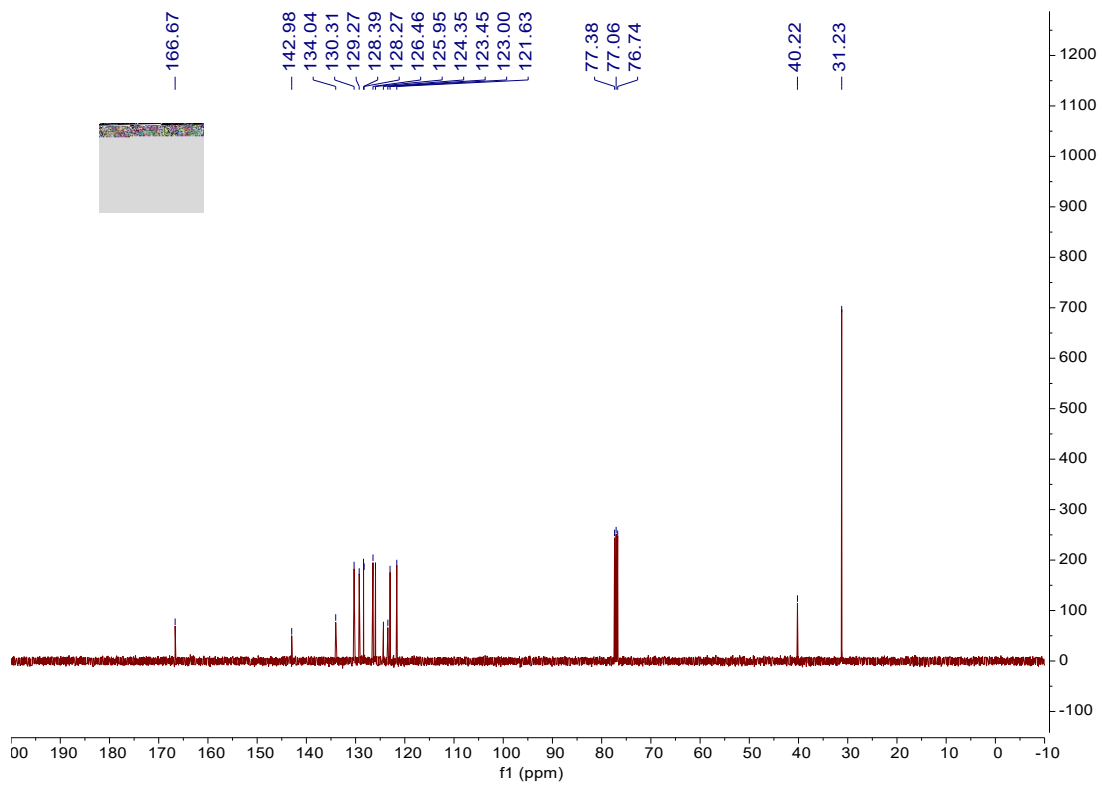
<sup>13</sup>C NMR spectrum of compound **4ba**



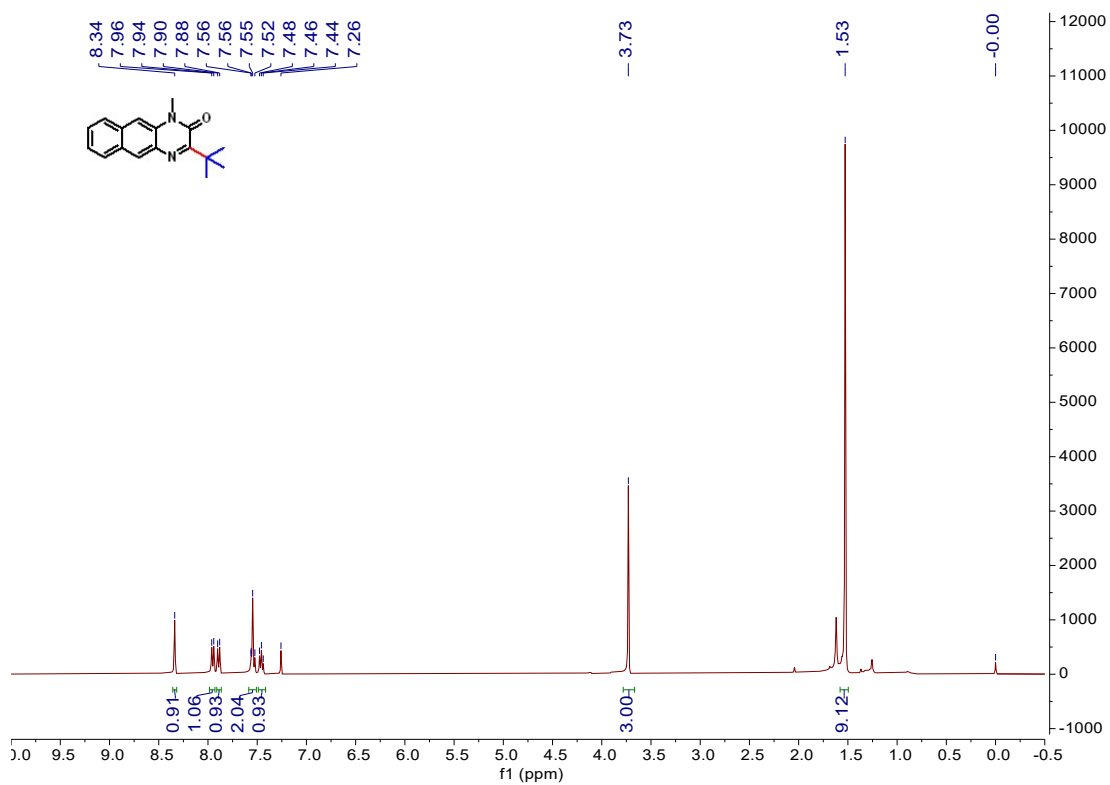
<sup>1</sup>H NMR spectrum of compound **4bb**



<sup>13</sup>C NMR spectrum of compound **4bb**

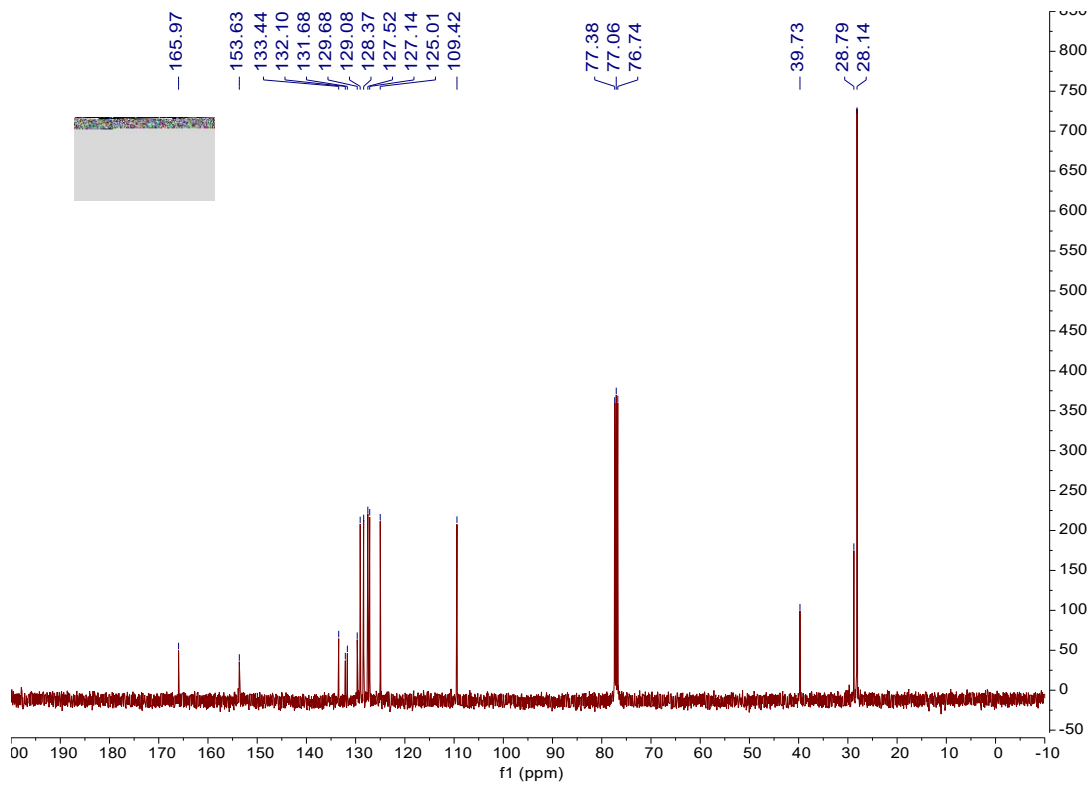


<sup>1</sup>H NMR spectrum of compound **4bc**

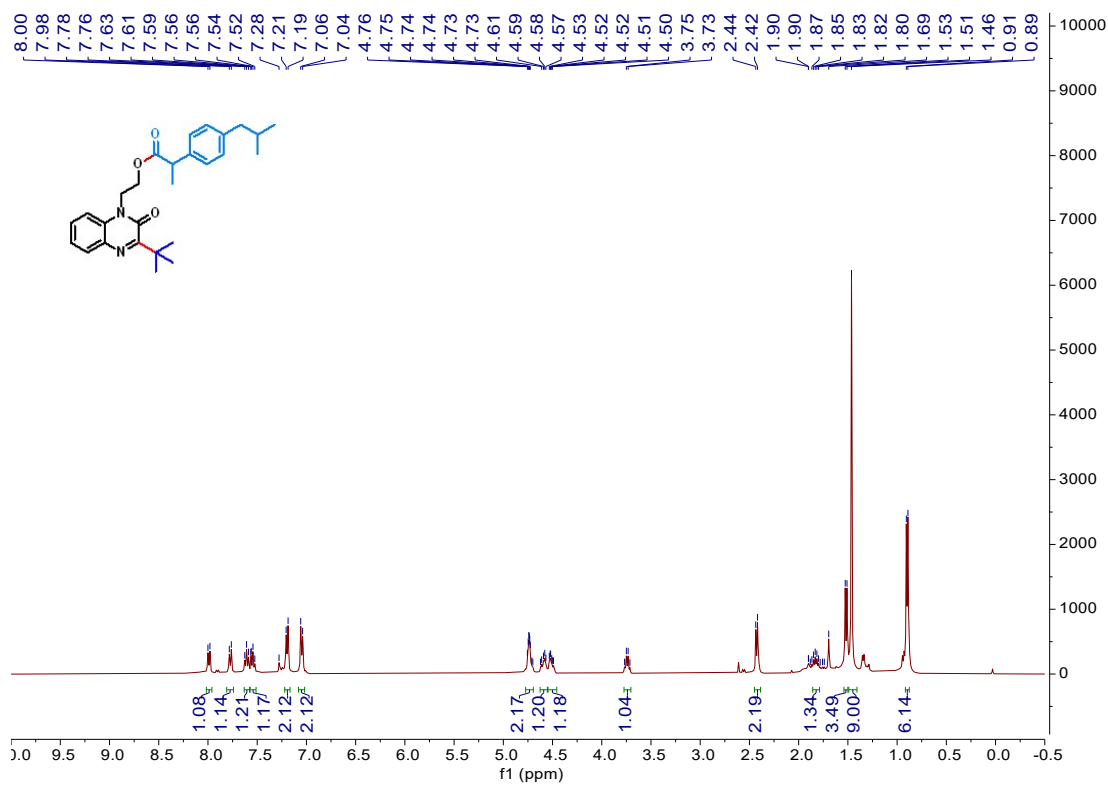


<sup>13</sup>C NMR spectrum of compound **4bc**

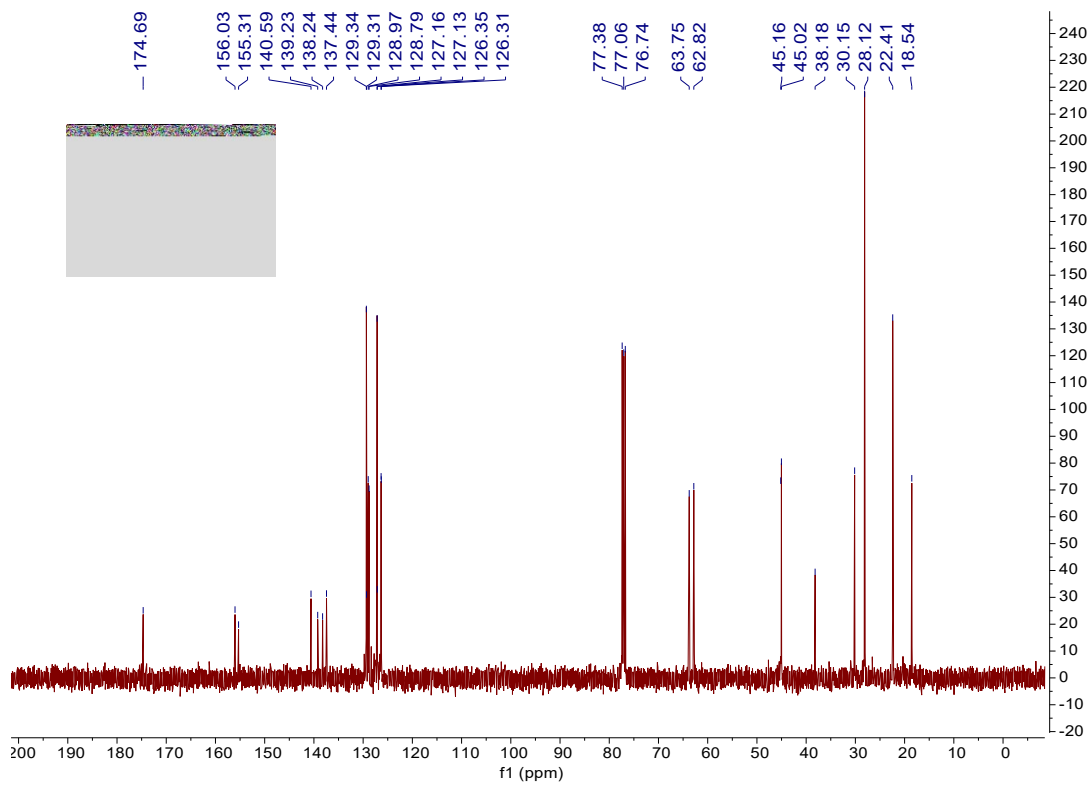




<sup>1</sup>H NMR spectrum of compound 4bd

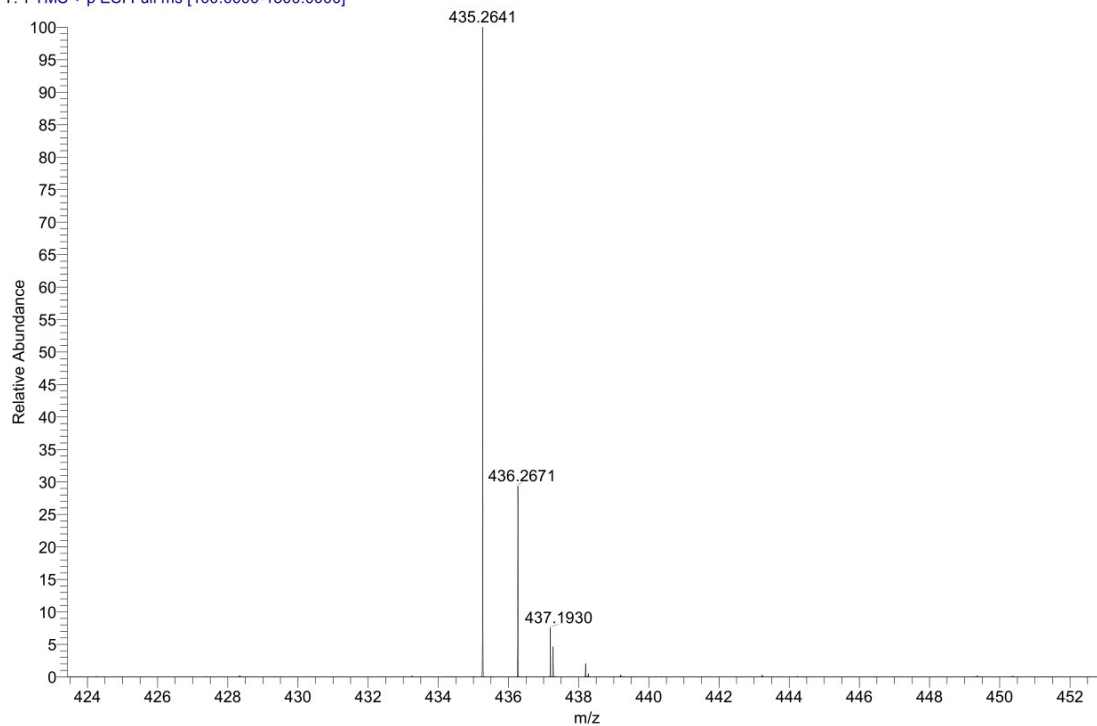


<sup>13</sup>C NMR spectrum of compound 4bd

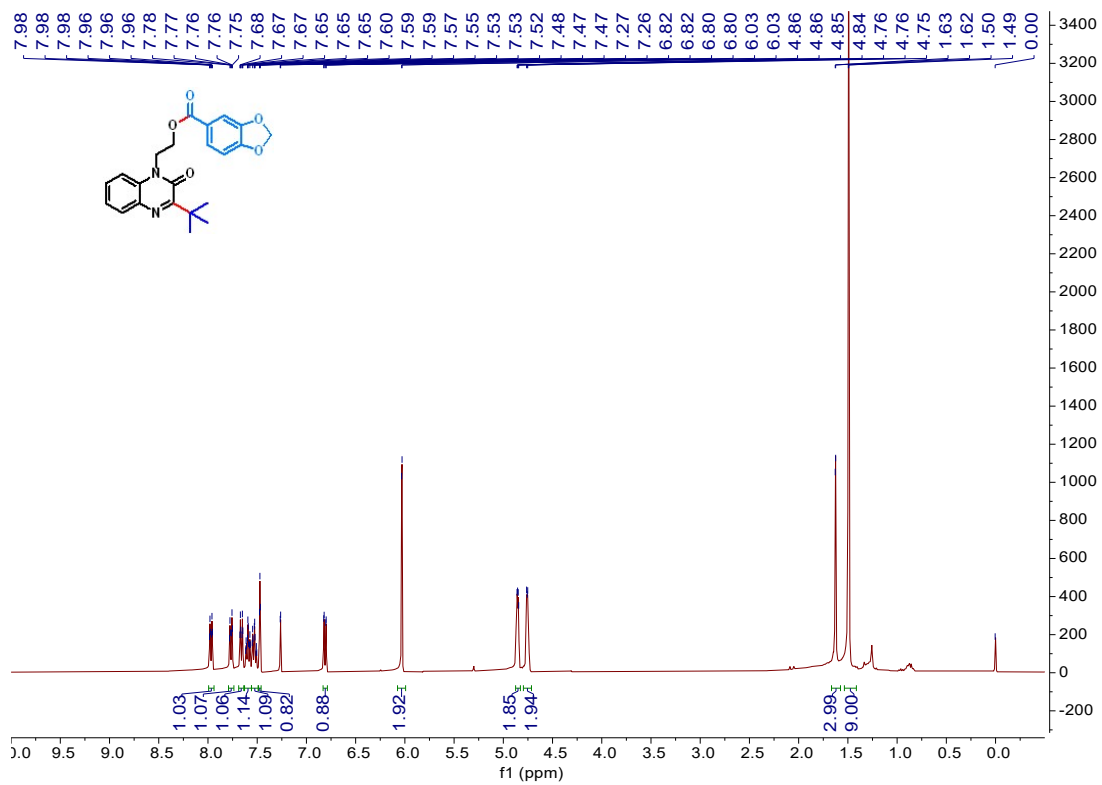


### HRMS of compound 4bd

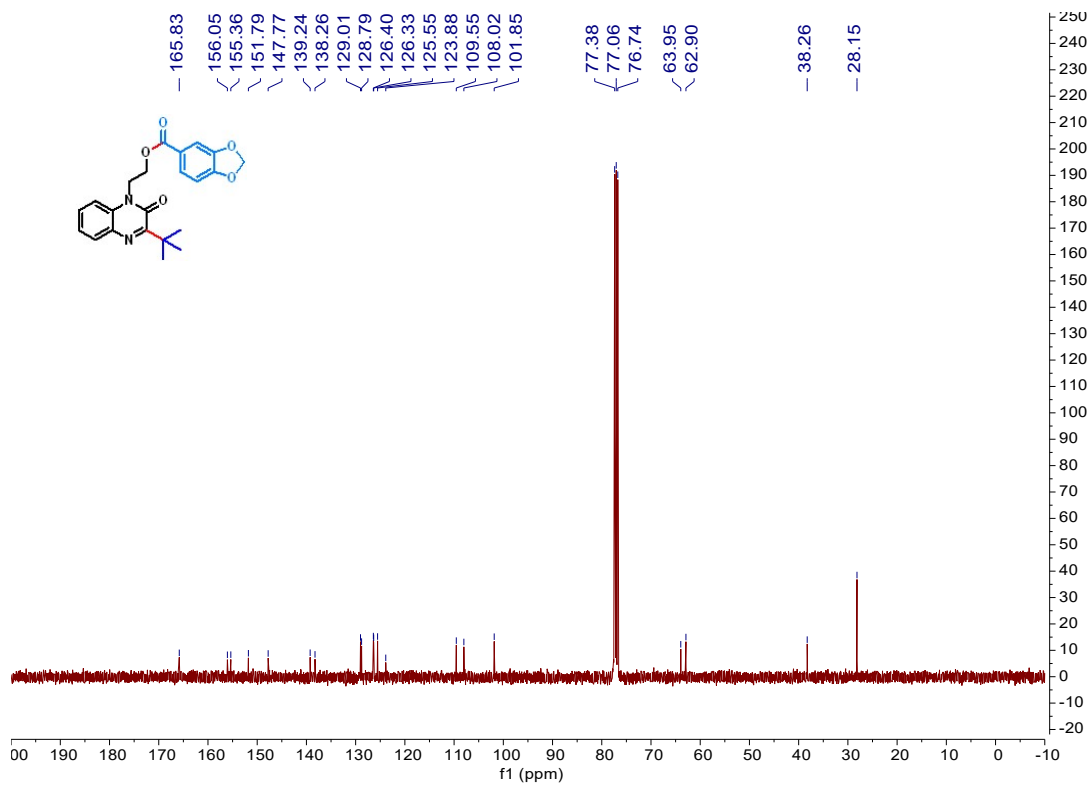
SXD-04-109 #24-31 RT: 0.10-0.14 AV: 8 SB: 26 0.59-0.70 NL: 2.97E9  
 T: FTMS + p ESI Full ms [100.0000-1500.0000]



### <sup>1</sup>H NMR spectrum of compound 4be

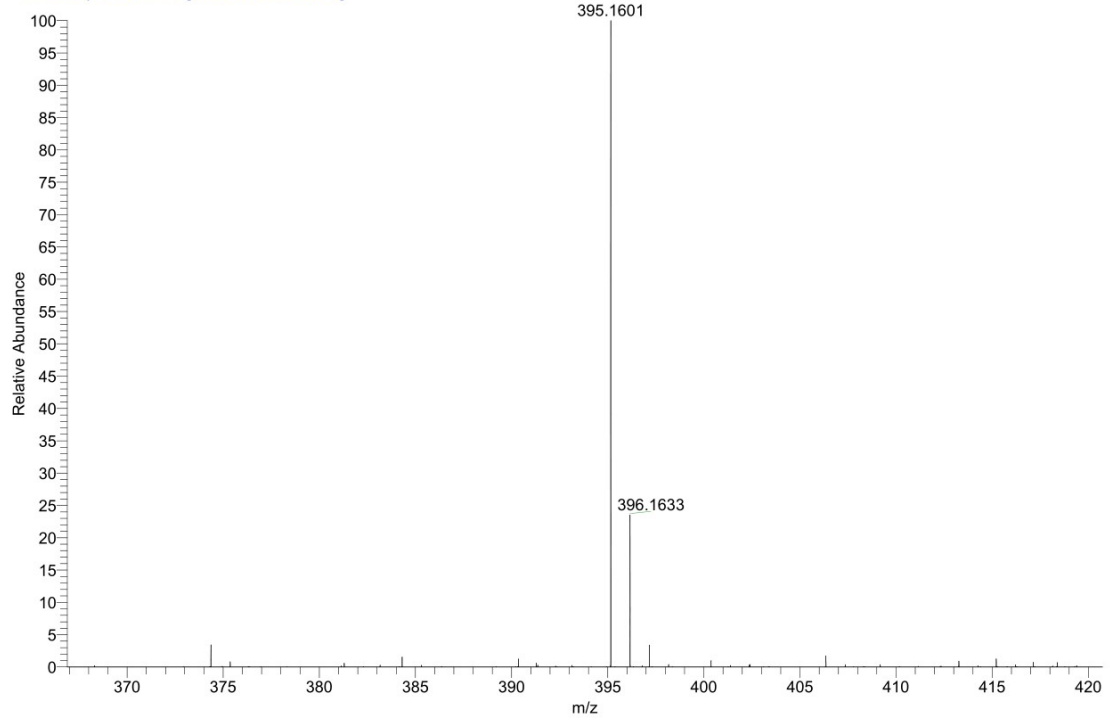


**<sup>13</sup>C NMR spectrum of compound 4be**

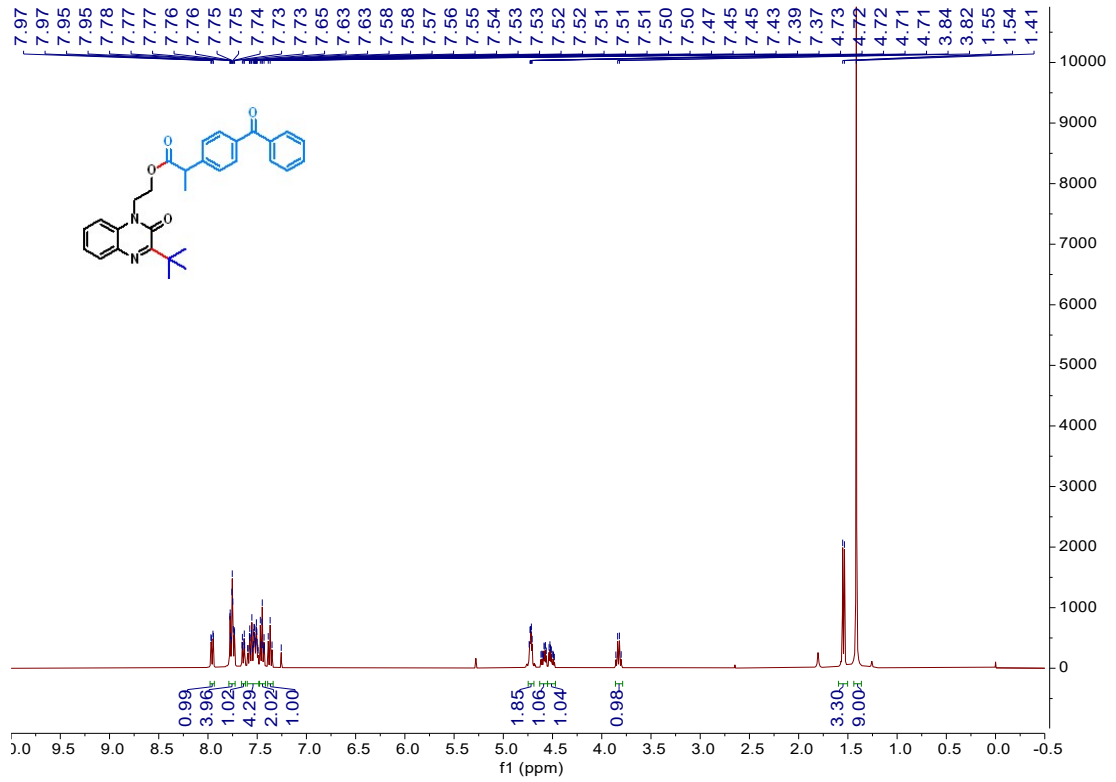


# HRMS of compound 4be

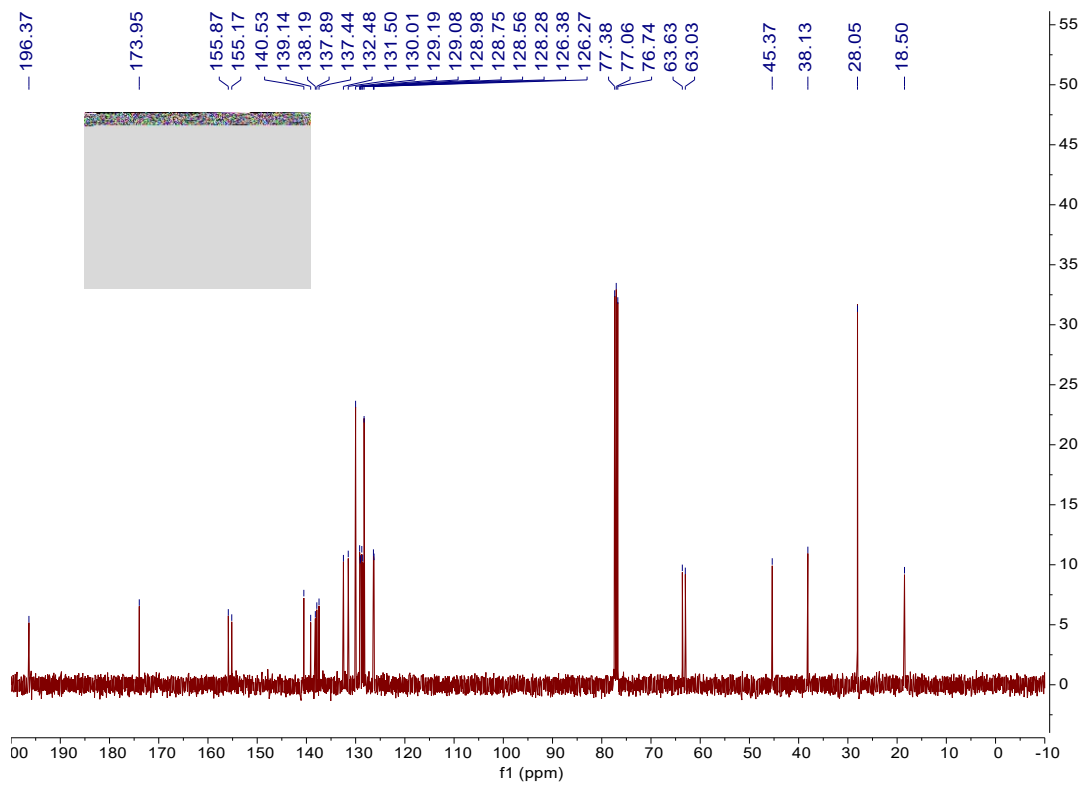
SXD-04-107 #21-24 RT: 0.09-0.10 AV: 4 NL: 1.50E9  
T: FTMS + p ESI Full ms [100.0000-1500.0000]



# <sup>1</sup>H NMR spectrum of compound 4bf

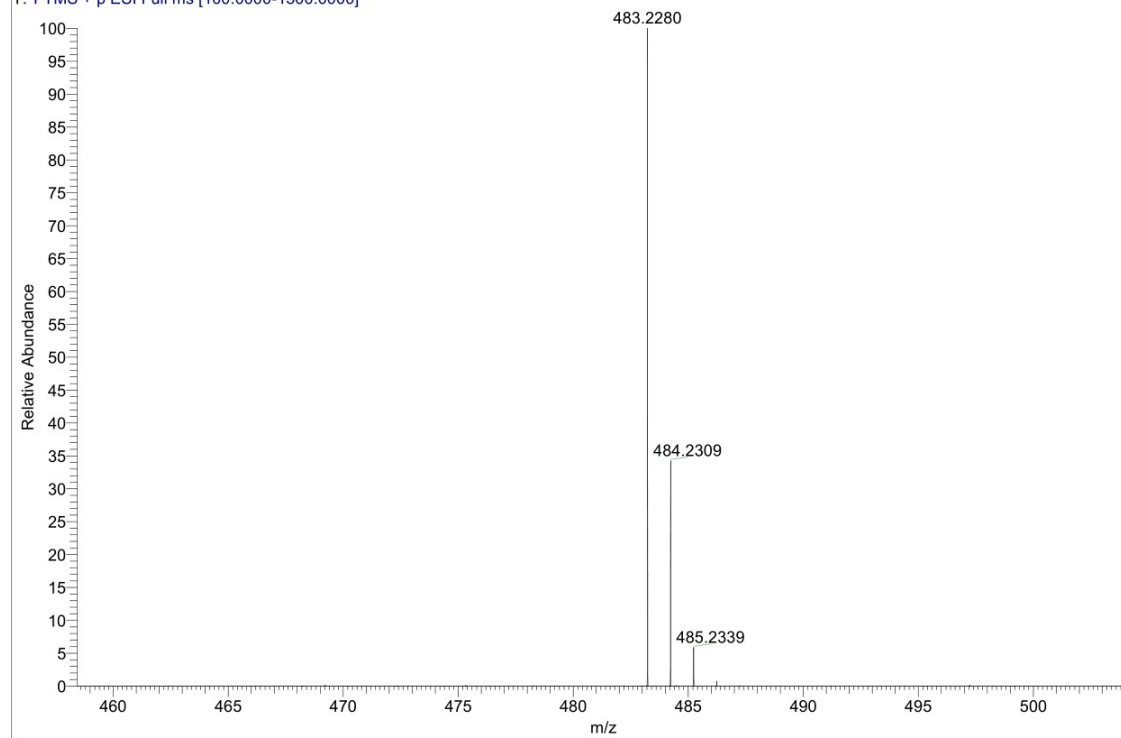


# <sup>13</sup>C NMR spectrum of compound 4bf

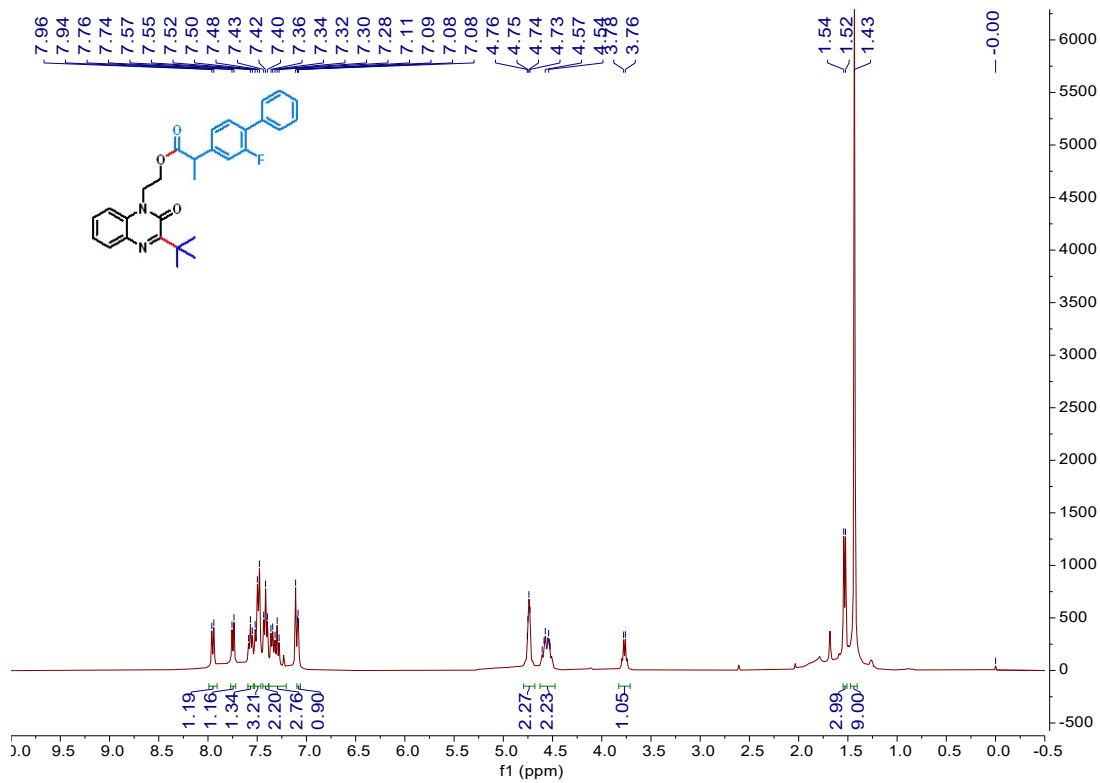


### HRMS of compound 4bf

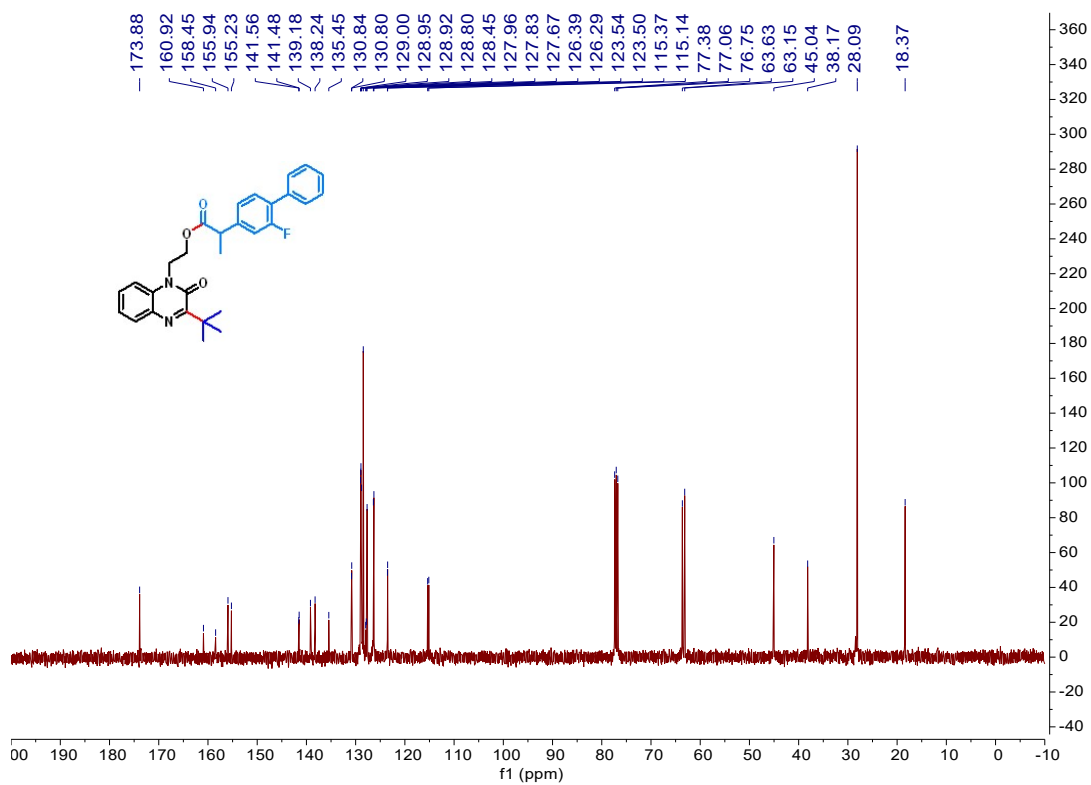
SXD-04-105 #25-31 RT: 0.11-0.14 AV: 7 SB: 24 0.74-0.84 NL: 2.62E9  
 T: FTMS + p ESI Full ms [100.0000-1500.0000]



### <sup>1</sup>H NMR spectrum of compound 4bg

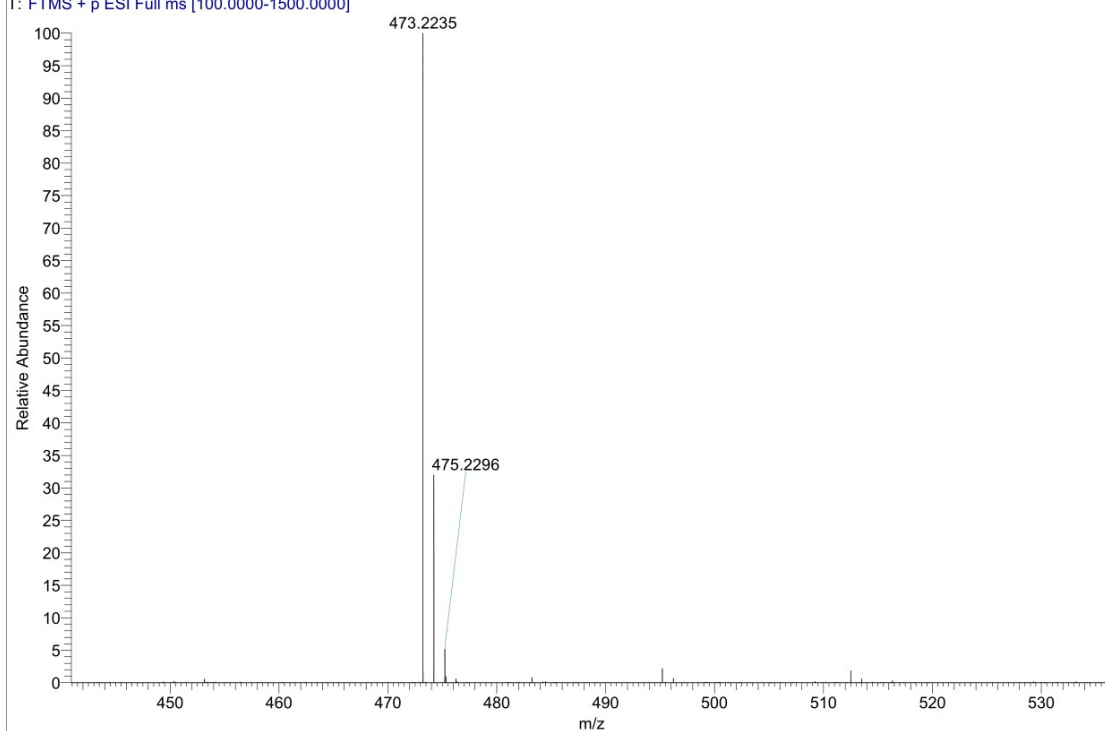


**<sup>13</sup>C NMR spectrum of compound 4bg**

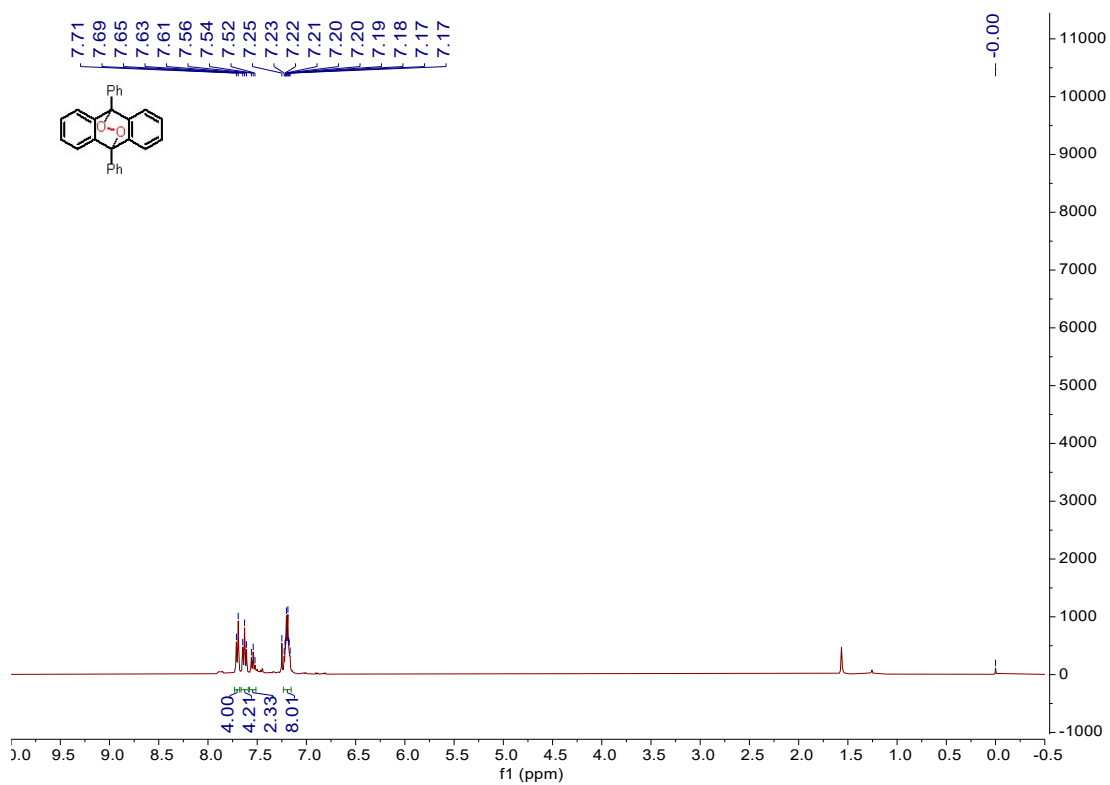


**HRMS of compound 4bg**

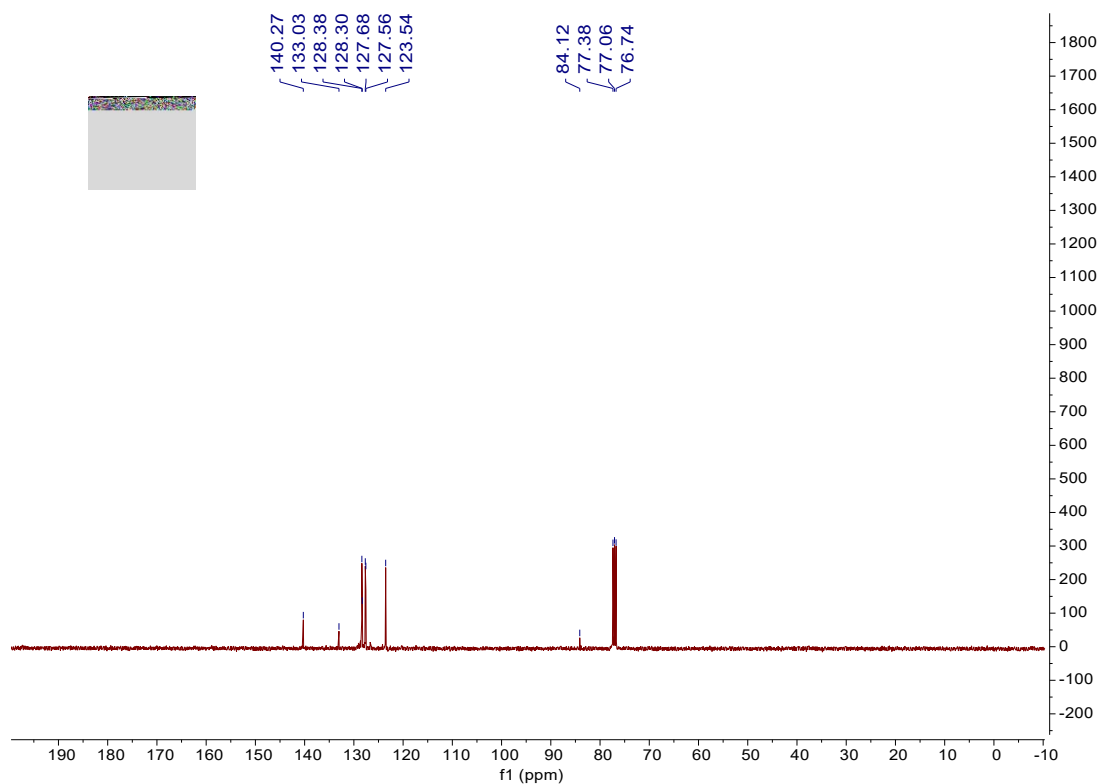
SXD-04-108 #23-30 RT: 0.10-0.13 AV: 8 SB: 18 0.67-0.75 NL: 1.22E9  
T: FTMS + p ESI Full ms [100.0000-1500.0000]



$^1\text{H}$  NMR spectrum of compound **5a**



$^{13}\text{C}$  NMR spectrum of compound **5a**



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