

*Electronic Supplementary Information (ESI)*

## **Metal-Free Oxidative Isocyanides Insertion with Aromatic Aldehydes to Aroylated *N*-heterocycles**

Yimiao He,<sup>\*a</sup> Xuelian Wang,<sup>a</sup> Jun-An Xiao,<sup>a</sup> Jinying Pang,<sup>a</sup> Chunfang Gan,<sup>a</sup> Yanmin Huang,<sup>a</sup> Chusheng Huang<sup>\*a</sup>

*College of Chemistry and Materials Science, Guangxi Teachers Education University, Nanning 530001, P. R. China. E-mail: heyimiao@gxtc.edu.cn; huangcs@gxtc.edu.cn.*

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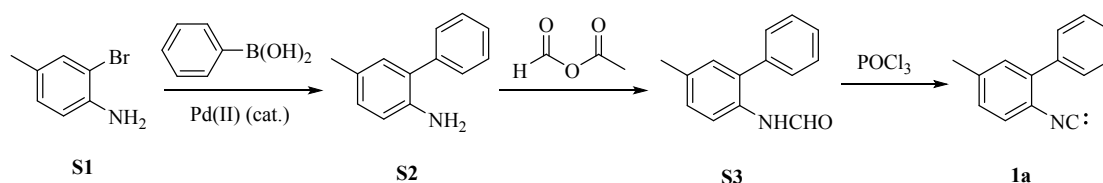
## 1. General information

Reactions were monitored by using thin-layer chromatography (TLC) on commercial silica gel plates (GF254). Visualization of the developed plates was performed under UV lights (254 nm). Flash column chromatography was performed on silica gel (200-300 mesh).  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were recorded on Bruker AV300, 400 and 500 MHz spectrometers. Chemical shifts ( $\delta$ ) were reported in ppm referenced to the  $\text{CDCl}_3$  residual peak ( $\delta$  7.26) or the  $\text{DMSO-d}_6$  residual peak ( $\delta$  2.50) for  $^1\text{H}$  NMR. Chemical shifts of  $^{13}\text{C}$  NMR were reported relative to  $\text{CDCl}_3$  ( $\delta$  77.0) or  $\text{D}_6\text{-DMSO}$  ( $\delta$  39.5). The following abbreviations were used to describe peak splitting patterns when appropriate: br s = broad singlet, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. Coupling constant,  $J$ , was reported in Hertz unit (Hz). Melting points (mp) were taken on a MEL-TEMP® apparatus and were uncorrected. High resolution mass spectra (HRMS) were obtained on an ESI-LC-MS/MS spectrometer.

## 2. Synthesis of isocyanides

### 2.1 Synthesis of phenyl isocyanides 1a-1w

The isocyanide **1a** was prepared according to the reported method.<sup>1</sup>



**4-Methyl-2-(phenyl)phenyl isocyanide (1a).** To an oven-dried three necked flask, 2-bromo-4-methylaniline **S1** (925 mg, 5 mmol), phenylboronic acid (732 mg, 6 mmol), aqueous solution of  $\text{K}_2\text{CO}_3$  (2M, 11 mL) and DME (10 mL) were added under a gentle stream of Ar, and the mixture was stirred for 30 min at room temperature under Ar atmosphere.  $\text{PdCl}_2(\text{PPh}_3)_2$  (70 mg, 0.10 mmol) was subsequently added at room temperature, and the mixture was stirred overnight at 80 °C under Ar. The reaction mixture was cooled to room temperature and diluted with EtOAc. The organic layer was washed with water and dried over  $\text{Na}_2\text{SO}_4$ . After removing the volatiles in vacuo, the residue was subjected to column chromatography on silica gel (petroleum ether/EtOAc = 4 : 1) to afford 4-methyl-2-phenylaniline **S2** (788 mg, 86% yield).

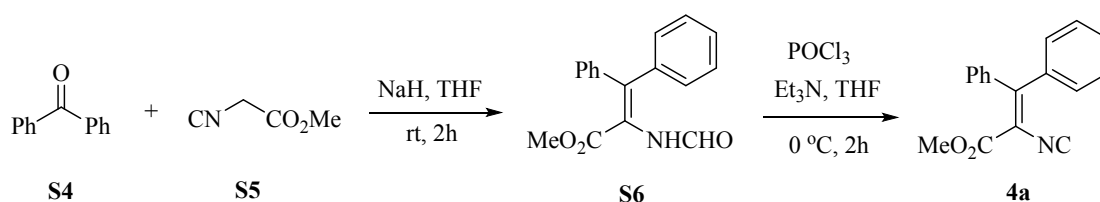
Acetic formic anhydride (0.89 mL) was added dropwise to a stirred solution of **S2** (788 mg, 4.30 mmol) in THF (8 mL) at 0 °C, and then the mixture was stirred for 2 h

at room temperature. the mixture was quenched by sat. aqueous solution of NaHCO<sub>3</sub> and extracted with EtOAc three times. The extract was dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated under reduced pressure to give formamide **S3** as a pale yellow oil. This material was used for the subsequent dehydration without further purification.

To an oven-dried three necked flask equipped with a dropping funnel, THF (8 mL), NEt<sub>3</sub> (4.3 mL) and the whole amount of **S3** obtained above were added under Ar atmosphere and cooled to 0 °C. POCl<sub>3</sub> (0.7 mL) in 2 mL of THF was added dropwise, and the mixture was stirred for 2 h at 0 °C until the addition was complete. The mixture was slowly quenched by sat. aqueous solution of NaHCO<sub>3</sub> at 0 °C and stirred for 1 h at room temperature. The mixture was extracted with EtOAc three times, dried over Na<sub>2</sub>SO<sub>4</sub> and evaporated under reduced pressure. The residues were purified by column chromatography (petroleum ether/EtOAc = 30 : 1) to give **1a** as a pale yellow solid (811 mg, 84% yield).

**1b-1w** were prepared according to the procedure described for **1a**.

## 2.2 Synthesis of vinyl isocyanides **4a-4e**<sup>2</sup>



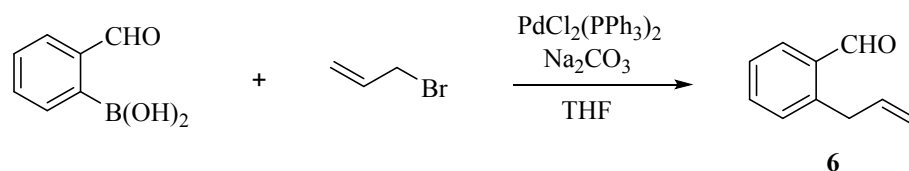
To a mixture of benzophenone **S4** (0.91 g, 5 mmol) and methyl isocyanoacetate **S5** (0.5 g, 5 mmol) in THF (5 mL), a suspension of NaH (60% in oil) (0.24 g, 6 mmol) in THF (5 mL) was added dropwise at room temperature for 2h. After the reaction was completed (as judged by TLC analysis), the solvent was removed under reduced pressure and the residue was extracted with CH<sub>2</sub>Cl<sub>2</sub> three times and the extract was washed with H<sub>2</sub>O, dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated under reduced pressure. The material **S6** was used for the subsequent dehydration without further purification.

To an oven-dried three necked flask equipped with a dropping funnel, THF (10 mL), NEt<sub>3</sub> (5 mL) and the whole amount of **S6** obtained above were added under Ar atmosphere and cooled to 0 °C. POCl<sub>3</sub> (0.8 mL) in 2 mL of THF was added dropwise, and the mixture was stirred for 2 h at 0 °C until the addition was complete. the mixture was slowly quenched by sat. aqueous solution of NaHCO<sub>3</sub> at 0 °C and stirred for 1 h at room temperature. The mixture was extracted with EtOAc three times, dried over Na<sub>2</sub>SO<sub>4</sub> and evaporated under reduced pressure. The residues were purified by column chromatography (petroleum ether/EtOAc = 30 : 1) to give **4a** as a white solid

(1.2 g, 89% yield).

**4b-4e** were prepared according to the procedure described for **4a**.

### 3. Synthesis of material **6**<sup>3</sup>

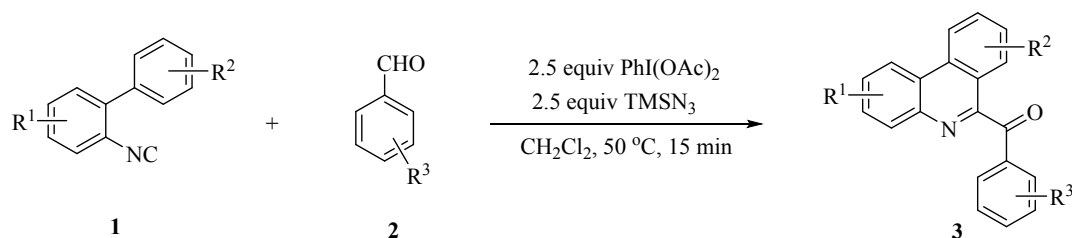


a 100 mL round-bottom flask was equipped with a rubber septum and magnetic stir bar and was charged with a solution of 2-formylphenylboronic acid (950 mg, 5 mmol) and allyl bromide (0.5 mL, 6 mmol) in THF (25 mL). Then PdCl<sub>2</sub>(PPh<sub>3</sub>)<sub>2</sub> (88 mg, 0.125 mmol) and aq. Na<sub>2</sub>CO<sub>3</sub> (1M, 10 mmol) solution was added. The reaction mixture was heated at reflux for 3-4 h. The reaction mixture was quenched with H<sub>2</sub>O and extracted with CH<sub>2</sub>Cl<sub>2</sub> three times. The combined organic layers were washed with H<sub>2</sub>O, dried over MgSO<sub>4</sub>, and concentrated in vacuo. The residues were purified by column chromatography on silica gel (petroleum ether/EtOAc = 30:1) to afford the desired product **6** (82% yield) as a pale yellow oil.

## 4. General procedure and product characterization

### 4.1 General procedure

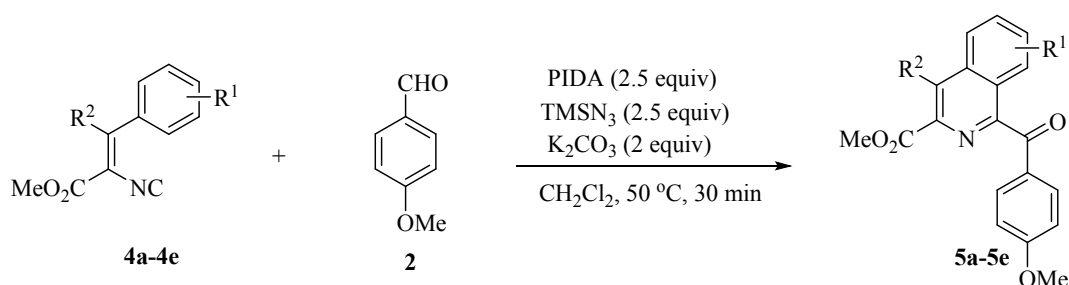
Typical procedure for PIDA-mediated oxidative phenyl isocyanides insertion with aromatic aldehydes to 6-arylated phenanthridines **3a-3w**.



A oven-dried sealed tube was equipped with a magnetic stir bar and was charged with a mixture of 2-isocyanobiphenyl (0.2 mmol), aldehyde (1.2 mmol) and TMSN<sub>3</sub> (0.5 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (1.0 mL). Then (diacetoxyiodo)benzene (PIDA) (0.5 mmol) was dissolved in CH<sub>2</sub>Cl<sub>2</sub> (1.0 mL) and added dropwise to the reaction mixture for 10

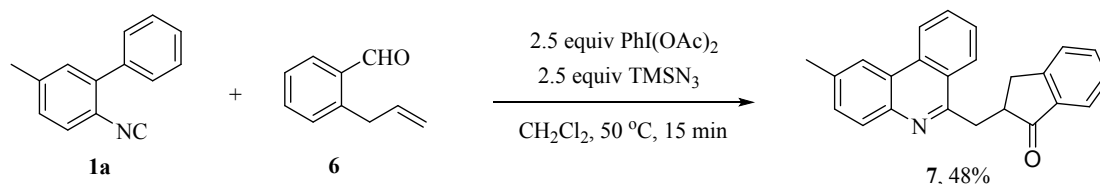
minutes at 50 °C. After the reaction was complete (as determined by TLC analysis), the reaction was cooled to room temperature and CH<sub>2</sub>Cl<sub>2</sub> (20 mL) was added to the solution and washed with water (20 ml x 3), dried over Na<sub>2</sub>SO<sub>4</sub>, concentrated under reduced pressure. The crude product was purified by column chromatography on silica gel (petroleum ether/EtOAc = 30:1) to afford the targeted product **3a-3w**.

Typical procedure for PIDA-mediated oxidative vinyl isocyanides insertion with aromatic aldehydes to 1-arylated isoquinolines **5a-5e**.



A oven-dried sealed tube was equipped with a magnetic stir bar and was charged with a mixture of vinyl isocyanide (0.2 mmol), aldehyde (1.2 mmol), K<sub>2</sub>CO<sub>3</sub> (0.4 mmol) and TMSN<sub>3</sub> (0.5 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (1.0 mL). Then (diacetoxyiodo)benzene (PIDA) (0.5 mmol) was dissolved in CH<sub>2</sub>Cl<sub>2</sub> (1.0 mL) and added dropwise to the reaction mixture for 10 minutes at 50 °C. After the reaction was complete (as determined by TLC analysis), the reaction was cooled to room temperature and CH<sub>2</sub>Cl<sub>2</sub> (20 mL) was added to the solution and washed with water (20 ml x 3), dried over Na<sub>2</sub>SO<sub>4</sub>, concentrated under reduced pressure. The crude product was purified by column chromatography on silica gel (petroleum ether/EtOAc = 30:1) to afford the targeted product **5a-5e**.

Typical procedure for PIDA-mediated oxidative phenyl isocyanide insertion with aromatic aldehyde bearing *ortho* terminal alkene **6** to phenanthridine derivative **7**.

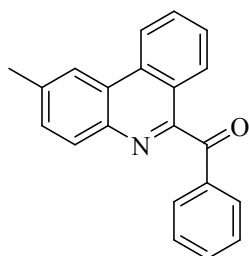


A oven-dried sealed tube was equipped with a magnetic stir bar and was charged with a mixture of 2-isocyanobiphenyl **1a** (0.2 mmol), aldehyde **6** (1.2 mmol) and TMSN<sub>3</sub> (0.5 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (1.0 mL). Then (diacetoxyiodo)benzene (PIDA) (0.5 mmol)

was dissolved in CH<sub>2</sub>Cl<sub>2</sub> (1.0 mL) and added dropwise to the reaction mixture for 10 minutes at 50 °C. After the reaction was complete (as determined by TLC analysis), the reaction was cooled to room temperature and CH<sub>2</sub>Cl<sub>2</sub> (20 mL) was added to the solution and washed with water (20 ml x 3), dried over Na<sub>2</sub>SO<sub>4</sub>, concentrated under reduced pressure. The crude product was purified by column chromatography on silica gel (petroleum ether/EtOAc = 30:1) to afford the targeted product **7**.

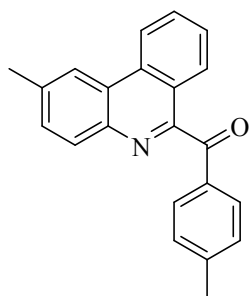
## 4.2 Product characterization

### (2-methylphenanthridin-6-yl)(phenyl)methanone (**3a**)<sup>4</sup>



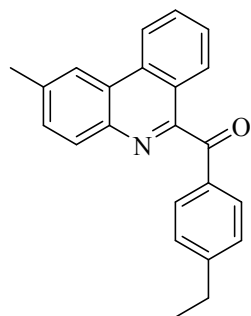
Yield: 74%. Mp 171-173 °C. Pale yellow solid. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 8.70 (d, *J* = 8.4 Hz, 1H), 8.44 (s, 1H), 8.16-8.09 (m, 2H), 8.04 (d, *J* = 8.0 Hz, 2H), 7.88 (t, *J* = 7.2 Hz, 1H), 7.67-7.60 (m, 3H), 7.48 (t, *J* = 7.2 Hz, 2H), 2.68 (s, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>): δ 194.9, 156.4, 140.9, 138.3, 136.3, 133.8, 133.0, 131.0, 130.8, 130.4, 128.5, 127.6, 127.2, 124.3, 124.0, 122.2, 121.7, 22.1; HRMS (ESI): Exact mass calcd for C<sub>21</sub>H<sub>15</sub>NO [M+H]<sup>+</sup>, 298.1193; Found: 298.1190.

### (2-methylphenanthridin-6-yl)(p-tolyl)methanone (**3b**)<sup>4a</sup>



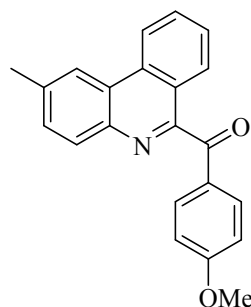
Yield: 66%. Mp 170-172 °C. Yellow solid. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 8.70 (d, *J* = 8.4 Hz, 1H), 8.43 (s, 1H), 8.11 (t, *J* = 7.2 Hz, 2H), 7.93 (d, *J* = 8.0 Hz, 2H), 7.87 (t, *J* = 7.6 Hz, 1H), 7.66-7.60 (m, 2H), 7.27 (d, *J* = 7.6 Hz, 2H), 2.67 (s, 3H), 2.43 (s, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>): δ 194.6, 156.8, 144.9, 141.0, 138.2, 133.9, 133.0, 130.9, 129.3, 127.6, 127.3, 124.3, 123.9, 122.2, 121.7, 22.1, 21.8; HRMS (ESI): Exact mass calcd for C<sub>22</sub>H<sub>17</sub>NO [M+H]<sup>+</sup>, 312.1383; Found: 312.1385.

**(4-ethylphenyl)(2-methylphenanthridin-6-yl)methanone (3c)**



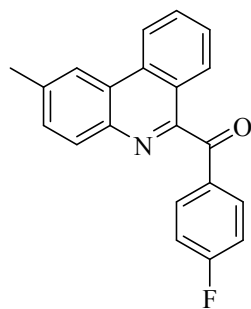
Yield: 63%. Mp 129-131 °C. Yellow solid. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 8.70 (d, *J* = 8.0 Hz, 1H), 8.43 (s, 1H), 8.12 (t, *J* = 8.0 Hz, 2H), 7.96 (d, *J* = 7.6 Hz, 2H), 7.87 (t, *J* = 7.6 Hz, 1H), 7.66-7.60 (m, 2H), 7.28 (d, *J* = 7.6 Hz, 2H), 2.75-2.69 (m, 2H), 2.67 (s, 3H), 1.26 (t, *J* = 7.2 Hz, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>): δ 197.5, 157.2, 141.3, 140.2, 138.3, 137.0, 133.2, 132.4, 132.0, 131.8, 130.7, 130.6, 127.6, 127.4, 125.4, 124.5, 124.1, 122.2, 121.6, 29.1, 22.1, 15.1; HRMS (ESI): Exact mass calcd for C<sub>23</sub>H<sub>19</sub>NO [M+H]<sup>+</sup>, 326.1539; Found: 326.1539.

**(4-methoxyphenyl)(2-methylphenanthridin-6-yl)methanone (3d) <sup>4a</sup>**



Yield: 76%. Mp 142-144 °C. White solid. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 8.70 (d, *J* = 8.0 Hz, 1H), 8.43 (s, 1H), 8.12 (t, *J* = 5.6 Hz, 2H), 8.01 (t, *J* = 7.6 Hz, 2H), 7.87 (t, *J* = 7.6 Hz, 1H), 7.65-7.60 (m, 2H), 6.94 (d, *J* = 7.6 Hz, 2H), 3.87 (s, 3H), 2.67 (s, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>): δ 193.6, 164.4, 157.1, 141.1, 138.2, 133.3, 133.1, 131.0, 130.9, 130.4, 129.5, 127.6, 127.5, 124.4, 124.0, 122.3, 121.8, 113.9, 55.6, 22.2; HRMS (ESI): Exact mass calcd for C<sub>22</sub>H<sub>17</sub>NO<sub>2</sub> [M+H]<sup>+</sup>, 328.1332; Found: 328.1331.

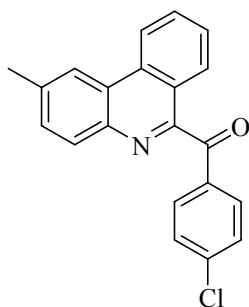
**(4-fluorophenyl)(2-methylphenanthridin-6-yl)methanone (3e) <sup>4a</sup>**



Yield: 49%. Mp 188-190 °C. White solid. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ 8.71 (d, *J* =

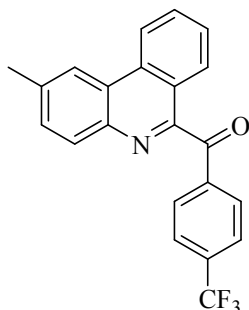
8.1 Hz, 1H), 8.44 (s, 1H), 8.18 (d,  $J = 8.4$  Hz, 1H), 8.14-8.09 (m, 3H), 7.92-7.87 (m, 1H), 7.70-7.62 (m, 2H), 7.17 (t,  $J = 8.7$  Hz, 2H), 2.69 (s, 3H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  193.2, 168.0, 164.6, 155.9, 140.9, 138.6, 133.7, 133.6, 133.1, 132.8, 132.7, 131.1, 130.9, 130.4, 127.7, 127.2, 124.4, 123.9, 122.3, 121.8, 115.9, 115.6, 22.2; HRMS (ESI): Exact mass calcd for  $\text{C}_{21}\text{H}_{14}\text{FNO}$   $[\text{M}+\text{H}]^+$ , 316.1132; Found: 316.1129.

**(4-chlorophenyl)(2-methylphenanthridin-6-yl)methanone (3f)** <sup>4a</sup>



Yield: 45%. Mp 216-218 °C. White solid.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.72 (d,  $J = 8.4$  Hz, 1H), 8.45 (s, 1H), 8.20 (d,  $J = 8.1$  Hz, 1H), 8.11 (d,  $J = 8.4$  Hz, 1H), 8.04-8.00 (m, 2H), 7.93-7.88 (m, 1H), 7.71-7.62 (m, 2H), 7.49-7.45 (m, 2H), 2.69 (s, 3H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  193.5, 155.6, 140.8, 140.4, 138.7, 134.7, 133.1, 132.3, 131.1, 131.0, 130.4, 128.9, 127.8, 127.2, 124.5, 123.9, 122.3, 121.8, 22.2.

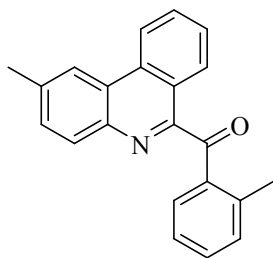
**(2-methylphenanthridin-6-yl)(4-(trifluoromethyl)phenyl)methanone (3g)**



Yield: 13%. Pale yellow oil.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.75 (d,  $J = 8.4$  Hz, 1H), 8.47 (s, 1H), 8.28 (d,  $J = 8.4$  Hz, 1H), 8.20 (d,  $J = 8.1$  Hz, 2H), 8.10 (d,  $J = 8.4$  Hz, 1H), 7.96-7.91 (m, 1H), 7.79-7.63 (m, 4H), 2.71 (s, 3H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  193.5, 154.9, 140.8, 139.3, 139.0, 133.2, 131.2, 131.0, 130.5, 129.2, 127.9, 127.1, 125.5, 125.4, 124.6, 123.9, 122.4, 121.8, 22.2; HRMS (ESI): Exact mass calcd for  $\text{C}_{22}\text{H}_{14}\text{F}_3\text{NO}$   $[\text{M}+\text{H}]^+$ , 366.1025; Found: 366.1029.

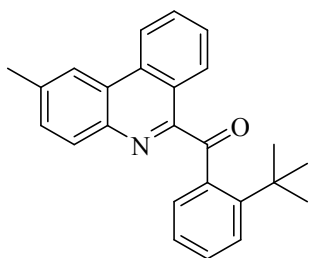
**(2-methylphenanthridin-6-yl)(o-tolyl)methanone (3h)** <sup>4a</sup>





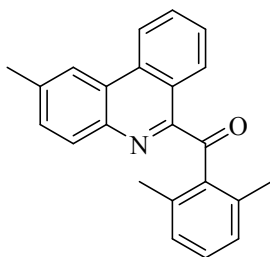
Yield: 50%. Mp 126-128 °C. White solid.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.70 (d,  $J = 6.8$  Hz, 1H), 8.42 (s, 1H), 8.25 (d,  $J = 8.0$  Hz, 1H), 8.05 (d,  $J = 8.4$  Hz, 1H), 7.88 (t,  $J = 7.2$  Hz, 1H), 7.67 (t,  $J = 8.0$  Hz, 1H), 7.58 (d,  $J = 8.4$  Hz, 1H), 7.52 (d,  $J = 7.6$  Hz, 1H), 7.45 (t,  $J = 7.6$  Hz, 1H), 7.35 (d,  $J = 7.2$  Hz, 1H), 7.18 (t,  $J = 7.6$  Hz, 1H), 2.66 (s, 6H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  197.5, 157.2, 141.3, 140.2, 138.3, 137.0, 133.2, 132.4, 132.0, 131.8, 130.7, 130.6, 127.6, 127.4, 125.4, 124.5, 124.1, 122.2, 121.6, 21.9, 21.4; HRMS (ESI): Exact mass calcd for  $\text{C}_{22}\text{H}_{17}\text{NO}$   $[\text{M}+\text{H}]^+$ , 312.1385; Found: 312.1384.

**(2-(*tert*-butyl)phenyl)(2-methylphenanthridin-6-yl)methanone (3i)**



Yield: 35%. Pale yellow oil.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  9.04 (d,  $J = 8.4$  Hz, 1H), 8.72 (d,  $J = 8.4$  Hz, 1H), 8.41 (s, 1H), 7.99-7.77 (m, 3H), 7.66 (d,  $J = 8.1$  Hz, 1H), 7.56-7.53 (m, 1H), 7.47-7.42 (m, 1H), 7.20 (d,  $J = 4.5$  Hz, 2H), 2.66 (s, 3H), 1.50 (s, 9H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  201.5, 154.3, 149.1, 140.9, 140.8, 139.3, 133.2, 131.2, 130.6, 129.4, 128.9, 128.1, 127.6, 127.5, 124.9, 124.6, 124.5, 122.1, 121.6, 36.4, 32.3, 22.2; HRMS (ESI): Exact mass calcd for  $\text{C}_{25}\text{H}_{23}\text{NO}$   $[\text{M}+\text{H}]^+$ , 354.1852; Found: 354.1859.

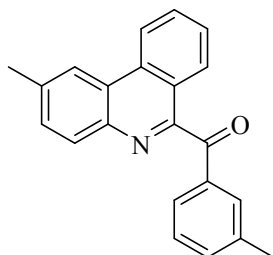
**(2,6-dimethylphenyl)(2-methylphenanthridin-6-yl)methanone (3j)**



Yield: 51%. Mp 101-103 °C. White solid.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  9.05 (d,  $J = 8.4$  Hz, 1H), 8.68 (d,  $J = 8.4$  Hz, 1H), 8.36 (s, 1H), 7.93 (d,  $J = 8.4$  Hz, 1H), 7.87 (t,  $J = 7.2$  Hz, 1H), 7.67 (t,  $J = 8.0$  Hz, 1H), 7.58 (d,  $J = 8.4$  Hz, 1H), 7.52 (d,  $J = 7.6$  Hz, 1H), 7.45 (t,  $J = 7.6$  Hz, 1H), 7.35 (d,  $J = 7.2$  Hz, 1H), 7.18 (t,  $J = 7.6$  Hz, 1H), 2.66 (s, 6H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  197.5, 157.2, 141.3, 140.2, 138.3, 137.0, 133.2, 132.4, 132.0, 131.8, 130.7, 130.6, 127.6, 127.4, 125.4, 124.5, 124.1, 122.2, 121.6, 21.9, 21.4; HRMS (ESI): Exact mass calcd for  $\text{C}_{24}\text{H}_{22}\text{NO}$   $[\text{M}+\text{H}]^+$ , 338.1684; Found: 338.1684.

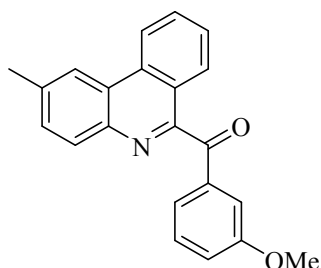
= 7.6 Hz, 1H), 7.77 (t,  $J = 7.6$  Hz, 1H), 7.50 (d,  $J = 8.4$  Hz, 1H), 7.23 (d,  $J = 7.2$  Hz, 1H), 7.07 (d,  $J = 7.6$  Hz, 2H), 2.62 (s, 3H), 2.22 (s, 6H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  202.4, 153.8, 142.2, 141.5, 139.6, 135.2, 133.7, 131.6, 130.7, 128.9, 128.3, 127.9, 127.7, 125.4, 124.0, 122.4, 121.8, 22.3, 20.0; HRMS (ESI): Exact mass calcd for  $\text{C}_{23}\text{H}_{19}\text{NO}$   $[\text{M}+\text{H}]^+$ , 326.1539; Found: 326.1540.

**(2-methylphenanthridin-6-yl)(m-tolyl)methanone (3k) <sup>4a</sup>**



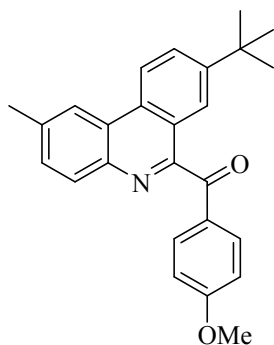
Yield: 42%. Mp 124-126 °C. Yellow solid.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.71 (d,  $J = 8.4$  Hz, 1H), 8.44 (s, 1H), 8.12 (d,  $J = 8.4$  Hz, 2H), 7.87 (t,  $J = 7.6$  Hz, 2H), 7.80 (t,  $J = 7.6$  Hz, 1H), 7.66-7.64 (m, 2H), 7.43 (d,  $J = 7.2$  Hz, 1H), 7.35 (t,  $J = 7.6$  Hz, 1H), 2.68 (s, 3H), 2.38 (s, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  195.2, 156.8, 141.0, 138.4, 138.3, 136.4, 134.8, 133.0, 131.1, 131.0, 130.8, 130.4, 128.5, 127.6, 127.3, 124.3, 124.0, 122.3, 121.8, 22.1, 21.3; HRMS (ESI): Exact mass calcd for  $\text{C}_{22}\text{H}_{17}\text{NO}$   $[\text{M}+\text{H}]^+$ , 312.1383; Found: 312.1384.

**(3-methoxyphenyl)(2-methylphenanthridin-6-yl)methanone (3l)**



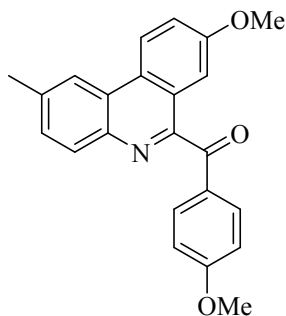
Yield: 52%. Mp 148-150 °C. White solid.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.70 (d,  $J = 8.0$  Hz, 1H), 8.43 (s, 1H), 8.12 (t,  $J = 8.0$  Hz, 2H), 7.87 (t,  $J = 7.2$  Hz, 1H), 7.67-7.60 (m, 3H), 7.50 (d,  $J = 7.6$  Hz, 1H), 7.34 (t,  $J = 8.0$  Hz, 1H), 7.17 (d,  $J = 8.0$  Hz, 1H), 3.85 (s, 3H), 2.67 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  194.7, 160.2, 156.8, 141.5, 138.5, 138.3, 133.4, 131.1, 130.8, 129.7, 127.8, 127.6, 124.7, 124.3, 124.2, 122.5, 120.8, 114.9, 55.7, 22.2; HRMS (ESI): Exact mass calcd for  $\text{C}_{22}\text{H}_{17}\text{NO}_2$   $[\text{M}+\text{H}]^+$ , 328.1332; Found: 328.1332.

**(8-(tert-butyl)-2-methylphenanthridin-6-yl)(4-methoxyphenyl)methanone (3m)**



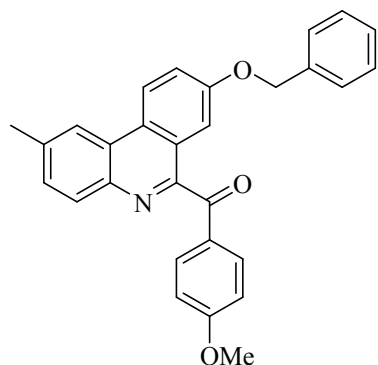
Yield: 47%. Mp 128-130 °C. Yellow solid.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.62 (d,  $J$  = 8.4 Hz, 1H), 8.39 (s, 1H), 8.08 (d,  $J$  = 8.8 Hz, 2H), 8.03 (d,  $J$  = 7.6 Hz, 2H), 7.94 (d,  $J$  = 8.4 Hz, 1H), 7.57 (d,  $J$  = 8.4 Hz, 1H), 6.95 (d,  $J$  = 8.0 Hz, 2H), 3.87 (s, 3H), 2.66 (s, 3H), 1.38 (s, 9H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  193.8, 164.2, 156.9, 150.7, 140.8, 138.0, 133.2, 130.9, 130.4, 130.2, 129.6, 129.4, 124.3, 124.0, 122.8, 122.0, 121.6, 113.8, 55.5, 35.0, 31.1, 22.1; HRMS (ESI): Exact mass calcd for  $\text{C}_{26}\text{H}_{25}\text{NO}_2$   $[\text{M}+\text{H}]^+$ , 384.1958; Found: 384.1960.

**(8-methoxy-2-methylphenanthridin-6-yl)(4-methoxyphenyl)methanone (3n)**



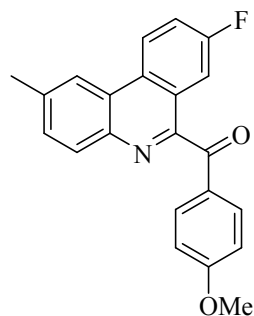
Yield: 62%. Mp 185-187 °C. Yellow solid.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.59 (d,  $J$  = 8.8 Hz, 1H), 8.33 (s, 1H), 8.08-8.01 (m, 3H), 7.55-7.48 (m, 3H), 6.95 (d,  $J$  = 8.0 Hz, 2H), 3.88 (s, 6H), 2.65 (s, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  193.6, 164.2, 158.8, 155.6, 140.2, 138.3, 133.3, 132.2, 130.2, 129.8, 129.4, 127.5, 125.3, 124.5, 123.8, 122.2, 121.2, 113.8, 106.7, 55.5, 22.1; HRMS (ESI): Exact mass calcd for  $\text{C}_{23}\text{H}_{19}\text{NO}_3$   $[\text{M}+\text{H}]^+$ , 358.1438; Found: 358.1436.

**(8-(benzyloxy)-2-methylphenanthridin-6-yl)(4-methoxyphenyl)methanone (3o)**



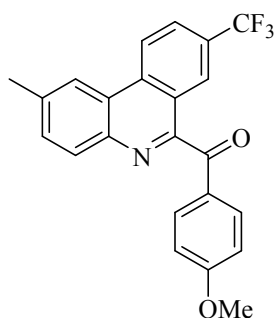
Yield: 76%. Mp 189-191 °C. White solid.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.61 (d,  $J = 8.8$  Hz, 1H), 8.34 (s, 1H), 8.07 (d,  $J = 8.4$  Hz, 1H), 8.01 (d,  $J = 7.6$  Hz, 2H), 7.61 (s, 1H), 7.55 (t,  $J = 8.4$  Hz, 2H), 7.43 (d,  $J = 7.2$  Hz, 2H), 7.38-7.31 (m, 3H), 6.95 (d,  $J = 7.6$  Hz, 2H), 5.12 (s, 2H), 3.89 (s, 3H), 2.65 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  193.7, 164.5, 158.3, 156.1, 140.8, 138.4, 136.7, 133.4, 130.7, 130.1, 130.0, 128.8, 128.3, 128.0, 127.9, 125.6, 124.8, 124.1, 122.6, 121.4, 114.1, 108.9, 70.8, 55.7, 22.2; HRMS (ESI): Exact mass calcd for  $\text{C}_{29}\text{H}_{23}\text{NO}_3$   $[\text{M}+\text{H}]^+$ , 434.1751; Found: 434.1752.

**(8-fluoro-2-methylphenanthridin-6-yl)(4-methoxyphenyl)methanone (3p)**



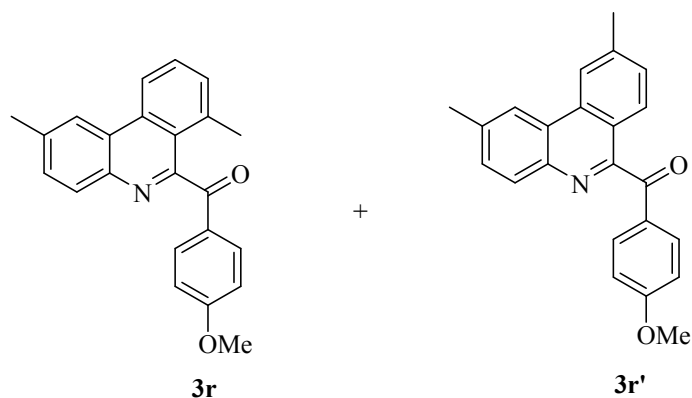
Yield: 87%. Mp 176-178 °C. Yellow solid.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.70-8.67 (m, 1H), 8.36 (s, 1H), 8.10 (d,  $J = 8.4$  Hz, 1H), 8.03 (d,  $J = 8.0$  Hz, 2H), 7.83 (d,  $J = 9.6$  Hz, 1H), 7.61 (t,  $J = 8.0$  Hz, 2H), 6.96 (d,  $J = 7.6$  Hz, 2H), 3.89 (s, 3H), 2.67 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  192.6, 164.4, 162.8, 160.3, 155.7, 140.8, 138.7, 133.2, 130.6, 130.5, 129.8, 129.6, 125.2, 124.7, 124.6, 124.1, 121.4, 120.2, 119.9, 113.9, 112.1, 111.9, 55.4, 21.9; HRMS (ESI): Exact mass calcd for  $\text{C}_{22}\text{H}_{16}\text{FNO}_2$   $[\text{M}+\text{H}]^+$ , 346.1238; Found: 346.1240.

**(4-methoxyphenyl)(2-methyl-8-(trifluoromethyl)phenanthridin-6-yl)methanone (3q)**



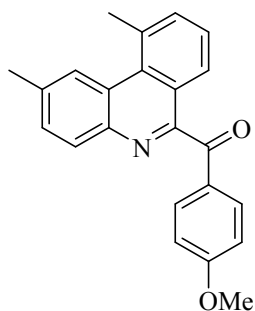
Yield: 62%. Mp 201-203 °C. White solid.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.81 (d,  $J = 8.8$  Hz, 1H), 8.49 (s, 1H), 8.44 (s, 1H), 8.14 (d,  $J = 8.4$  Hz, 1H), 8.05 (d,  $J = 8.4$  Hz, 3H), 7.69 (t,  $J = 8.4$  Hz, 1H), 6.97 (t,  $J = 7.6$  Hz, 2H), 3.89 (s, 3H), 2.69 (s, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  192.6, 164.6, 156.3, 141.6, 139.2, 133.5, 132.1, 130.7, 129.2, 126.8, 126.7, 125.1, 125.0, 123.5, 123.4, 122.2, 114.0, 55.7, 22.2; HRMS (ESI): Exact mass calcd for  $\text{C}_{23}\text{H}_{16}\text{F}_3\text{NO}_2$   $[\text{M}+\text{H}]^+$ , 396.1206; Found: 396.1206.

**(2,7-dimethylphenanthridin-6-yl)(4-methoxyphenyl)methanone (3r) and (2,9-dimethylphenanthridin-6-yl)(4-methoxyphenyl)methanone (3r')**



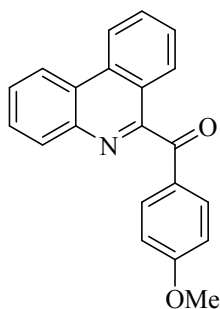
Yield: **3r** = 36%, **3r'** = 20%. Yellow solid.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.61 (d,  $J = 8.4$  Hz, 0.64H), 8.45 (s, 0.36H), 8.42 (s, 1H), 8.09-7.94 (m, 3H), 7.75 (t,  $J = 7.2$  Hz, 0.64H), 7.58 (d,  $J = 6.0$  Hz, 1H), 7.46 (d,  $J = 7.2$  Hz, 1H), 6.94 (t,  $J = 7.2$  Hz, 2H), 3.87 (s, 3H), 2.65 (s, 4H), 2.54 (s, 1.8H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  195.2, 164.8, 164.7, 157.7, 141.3, 138.2, 136.7, 134.7, 133.6, 133.3, 131.1, 131.0, 130.9, 130.5, 130.3, 129.7, 127.8, 124.9, 122.4, 122.3, 122.1, 120.9, 114.5, 114.4, 55.9, 32.0, 24.1, 23.0, 22.4, 22.3.

**(4-methoxyphenyl)(10-methylphenanthridin-6-yl)methanone (3s)**



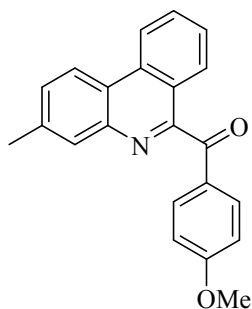
Yield: 44%. Mp 143-145 °C. Yellow solid.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.68 (s, 1H), 8.14 (d,  $J = 8.4$  Hz, 1H), 7.95 (t,  $J = 7.2$  Hz, 3H), 7.69 (d,  $J = 7.2$  Hz, 1H), 7.61 (d,  $J = 8.4$  Hz, 1H), 7.51 (t,  $J = 7.6$  Hz, 1H), 6.92 (d,  $J = 7.6$  Hz, 2H), 3.86 (s, 3H), 3.18 (s, 3H), 2.67 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  193.9, 164.3, 158.1, 142.6, 136.9, 135.5, 135.0, 133.0, 130.7, 129.8, 129.7, 126.9, 126.4, 126.0, 125.8, 125.5, 113.9, 55.4, 26.7, 22.3; HRMS (ESI): Exact mass calcd for  $\text{C}_{22}\text{H}_{17}\text{NO}_2$   $[\text{M}+\text{H}]^+$ , 342.1489; Found: 342.1487.

**(4-methoxyphenyl)(phenanthridin-6-yl)methanone (3t)**



Yield: 66%. Mp 162-164 °C. Yellow solid.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.72 (d,  $J = 8.0$  Hz, 1H), 8.65 (d,  $J = 7.6$  Hz, 1H), 8.22 (t,  $J = 7.6$  Hz, 1H), 8.12 (d,  $J = 8.0$  Hz, 1H), 8.02 (d,  $J = 8.0$  Hz, 2H), 7.89 (t,  $J = 7.6$  Hz, 1H), 7.81-7.74 (m, 2H), 7.66 (t,  $J = 7.2$  Hz, 1H), 6.94 (d,  $J = 8.4$  Hz, 2H), 3.87 (s, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  193.4, 164.3, 158.0, 142.7, 133.3, 133.2, 131.2, 130.6, 129.2, 129.0, 128.0, 127.7, 127.4, 124.4, 123.8, 122.2, 122.1, 113.9, 55.5; HRMS (ESI): Exact mass calcd for  $\text{C}_{21}\text{H}_{15}\text{NO}_2$   $[\text{M}+\text{H}]^+$ , 314.1176; Found: 314.1179.

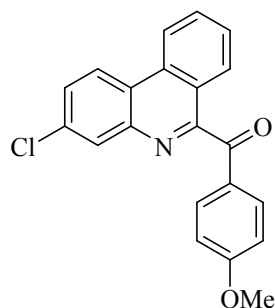
**(4-methoxyphenyl)(3-methylphenanthridin-6-yl)methanone (3u)**



Yield: 62%. Mp 143-145 °C. Yellow solid.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.66 (d,  $J$

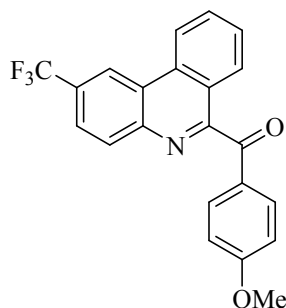
= 8.4 Hz, 1H), 8.53 (d,  $J = 8.4$  Hz, 1H), 8.10 (d,  $J = 8.4$  Hz, 1H), 8.01 (d,  $J = 7.6$  Hz, 3H), 7.86 (t,  $J = 7.6$  Hz, 1H), 7.63-7.57 (m, 2H), 6.94 (d,  $J = 8.0$  Hz, 2H), 3.87 (s, 3H), 2.60 (s, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  193.5, 164.3, 157.9, 142.8, 139.3, 133.3, 133.2, 131.1, 130.1, 129.7, 129.3, 127.4, 127.2, 123.5, 122.1, 122.0, 121.9, 113.8, 55.5, 21.5; HRMS (ESI): Exact mass calcd for  $\text{C}_{22}\text{H}_{17}\text{NO}_2$   $[\text{M}+\text{H}]^+$ , 328.1332; Found: 328.1334.

**(3-chlorophenanthridin-6-yl)(4-methoxyphenyl)methanone (3v)**



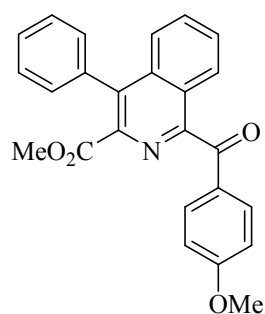
Yield: 75%. Mp 162-164 °C. Yellow solid.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.65 (d,  $J = 8.4$  Hz, 1H), 8.56 (d,  $J = 8.8$  Hz, 1H), 8.22 (s, 1H), 8.12 (d,  $J = 8.4$  Hz, 1H), 8.00 (d,  $J = 7.6$  Hz, 2H), 7.90 (t,  $J = 7.2$  Hz, 1H), 7.72-7.65 (m, 2H), 6.95 (d,  $J = 7.6$  Hz, 2H), 3.88 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  193.0, 164.8, 159.4, 143.9, 135.1, 133.3, 131.7, 130.1, 129.7, 128.7, 128.1, 128.0, 124.2, 123.7, 123.2, 122.4, 114.2, 55.7; HRMS (ESI): Exact mass calcd for  $\text{C}_{21}\text{H}_{14}\text{ClNO}_2$   $[\text{M}+\text{H}]^+$ , 348.0784; Found: 348.0786.

**(4-methoxyphenyl)(2-(trifluoromethyl)phenanthridin-6-yl)methanone (3w)**



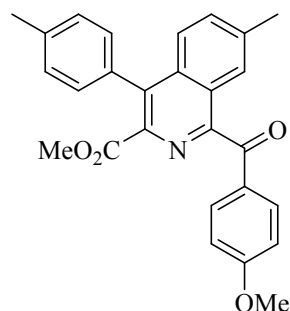
Yield: 81%. Mp 184-186 °C. White solid.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.92 (s, 1H), 8.75 (d,  $J = 8.4$  Hz, 1H), 8.33 (d,  $J = 8.4$  Hz, 1H), 8.15 (d,  $J = 8.0$  Hz, 1H), 8.00-7.95 (m, 4H), 7.73 (t,  $J = 7.2$  Hz, 1H), 6.96 (d,  $J = 8.0$  Hz, 2H), 3.88 (s, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  192.9, 164.6, 160.2, 144.2, 133.1, 133.0, 131.9, 131.5, 128.8, 127.8, 125.0, 124.0, 122.3, 120.0, 119.9, 114.0, 55.6; HRMS (ESI): Exact mass calcd for  $\text{C}_{22}\text{H}_{14}\text{F}_3\text{NO}_2$   $[\text{M}+\text{H}]^+$ , 382.1049; Found: 382.1050.

**methyl 1-(4-methoxybenzoyl)-4-phenylisoquinoline-3-carboxylate (5a)**



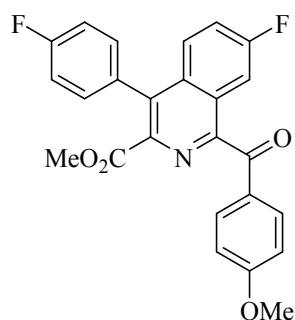
Yield: 48%. Mp 165-167 °C. White solid.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.24-8.20 (m, 1H), 8.10-8.04 (m, 2H), 7.76-7.66 (m, 3H), 7.55 (t,  $J = 5.7$  Hz, 3H), 7.46-7.41 (m, 2H), 7.02-6.97 (m, 2H), 3.90 (s, 3H), 3.69 (s, 3H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  192.5, 167.2, 164.4, 156.5, 140.6, 136.4, 135.6, 134.9, 133.4, 131.2, 129.7, 129.3, 129.2, 128.3, 126.8, 126.5, 126.4, 113.9, 55.6, 52.5; HRMS (ESI): Exact mass calcd for  $\text{C}_{25}\text{H}_{19}\text{NO}_4$   $[\text{M}+\text{H}]^+$ , 398.1392; Found: 398.1394.

**methyl 1-(4-methoxybenzoyl)-7-methyl-4-(p-tolyl)isoquinoline-3-carboxylate (5b)**



Yield: 46%. Mp 132-134 °C. Pale yellow Solid.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.07-8.03 (m, 2H), 7.97 (s, 1H), 7.66 (d,  $J = 8.7$  Hz, 1H), 7.51 (d,  $J = 8.7$  Hz, 1H), 7.36-7.28 (m, 4H), 6.98 (t,  $J = 8.7$  Hz, 2H), 3.88 (s, 3H), 3.71 (s, 3H), 2.49 (s, 6H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  192.7, 167.3, 164.3, 155.5, 139.8, 139.6, 137.9, 135.1, 134.8, 133.4, 132.7, 129.5, 129.3, 129.0, 126.8, 126.7, 125.1, 113.9, 55.6, 52.4, 21.9, 21.4; HRMS (ESI): Exact mass calcd for  $\text{C}_{27}\text{H}_{23}\text{NO}_4$   $[\text{M}+\text{H}]^+$ , 426.1705; Found: 426.1715.

**Methyl 7-fluoro-4-(4-fluorophenyl)-1-(4-methoxybenzoyl)isoquinoline-3-carboxylate (5c)**

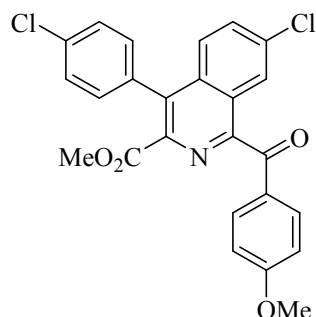


Yield: 42%. Mp 148-150 °C. White solid.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.09-8.05



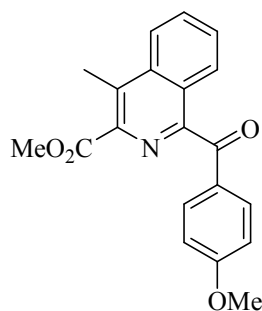
(m, 2H), 7.92 (d,  $J = 9.6$  Hz, 1H), 7.72 (t,  $J = 5.4$  Hz, 1H), 7.52-7.45 (m, 1H), 7.41-7.36 (m, 2H), 7.27-7.22 (m, 2H), 7.00 (d,  $J = 5.1$  Hz, 2H), 3.90 (s, 3H), 3.71 (s, 3H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  191.7, 166.8, 164.5, 163.8, 161.2, 160.5, 155.5, 140.2, 140.1, 134.2, 133.7, 133.5, 131.4, 131.3, 131.1, 129.7, 129.6, 128.9, 127.9, 127.7, 122.1, 121.7, 115.8, 115.5, 113.9, 110.5, 110.2, 55.6, 52.6; HRMS (ESI): Exact mass calcd for  $\text{C}_{25}\text{H}_{17}\text{F}_2\text{NO}_4$   $[\text{M}+\text{H}]^+$ , 434.1204; Found: 434.1205.

**methyl 7-chloro-4-(4-chlorophenyl)-1-(4-methoxybenzoyl)isoquinoline-3-carboxylate(5d)**



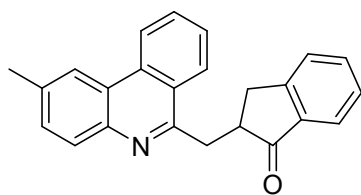
Yield: 61%. Mp 137-138 °C. Pale yellow solid.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.27 (s, 1H), 8.04 (t,  $J = 3.0$  Hz, 2H), 7.64 (t,  $J = 3.3$  Hz, 2H), 7.56-7.52 (m, 2H), 7.36-7.32 (m, 2H), 7.02-6.98 (m, 2H), 3.92 (s, 3H), 3.73 (s, 3H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  191.6, 166.6, 164.5, 155.6, 140.5, 135.8, 134.8, 134.7, 133.9, 133.5, 132.4, 130.9, 128.9, 128.8, 128.3, 127.2, 125.5, 114.0, 55.6, 52.7; HRMS (ESI): Exact mass calcd for  $\text{C}_{25}\text{H}_{17}\text{Cl}_2\text{NO}_4$   $[\text{M}+\text{H}]^+$ , 466.0613, Found: 466.0618;  $[\text{M}+2+\text{H}]^+$ , 468.0305, Found: 468.0309;  $[\text{M}+4+\text{H}]^+$ , 470.0125, Found: 470.0122.

**methyl 1-(4-methoxybenzoyl)-4-methylisoquinoline-3-carboxylate (5e)**



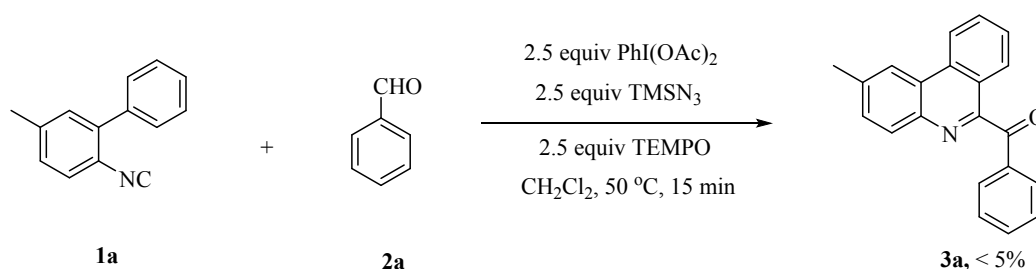
Yield: 41%. Mp 130-132 °C. Pale yellow solid.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.25-8.14 (m, 2H), 8.00-7.95 (m, 2H), 7.86-7.80 (m, 1H), 7.70-7.64 (m, 1H), 6.97-6.92 (m, 2H), 3.98 (s, 3H), 3.88 (s, 3H), 2.93 (s, 3H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  192.6, 167.7, 164.2, 155.0, 140.3, 136.6, 133.4, 131.1, 130.9, 129.4, 129.0, 126.9, 126.3, 124.5, 113.8, 55.5, 52.7, 14.6; HRMS (ESI): Exact mass calcd for  $\text{C}_{20}\text{H}_{17}\text{NO}_4$   $[\text{M}+\text{H}]^+$ , 336.1236; Found: 336.1233.

**2-((2-methylphenanthridin-6-yl)methyl)-2,3-dihydro-1H-inden-1-one (7)**



Yield: 48%. mp 172-174 °C. yellow solid. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 8.71 (d, *J* = 8.0 Hz, 1H), 8.42 (s, 1H), 8.29 (d, *J* = 8.4 Hz, 1H), 8.03 (d, *J* = 8.4 Hz, 1H), 7.88 (d, *J* = 7.2 Hz, 1H), 7.69 (t, *J* = 7.2 Hz, 1H), 7.58 (d, *J* = 8.4 Hz, 1H), 7.46 (t, *J* = 8.8 Hz, 2H), 7.39 (d, *J* = 7.6 Hz, 1H), 7.20 (t, *J* = 7.2 Hz, 1H), 3.34 (t, *J* = 6.8 Hz, 2H), 3.11 (t, *J* = 7.2 Hz, 2H), 2.66 (s, 3H), 2.10-2.02 (m, 1H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>): δ 197.6, 156.9, 143.3, 141.0, 138.5, 136.5, 133.0, 132.4, 131.3, 130.9, 130.8, 130.5, 127.8, 127.2, 125.9, 124.4, 123.9, 122.2, 121.7, 51.1, 31.5, 30.6, 22.1; HRMS (ESI): Exact mass calcd for C<sub>24</sub>H<sub>19</sub>NO [M+H]<sup>+</sup>, 338.1501; Found: 338.1502.

## 5. Free-Radical inhibition experiment

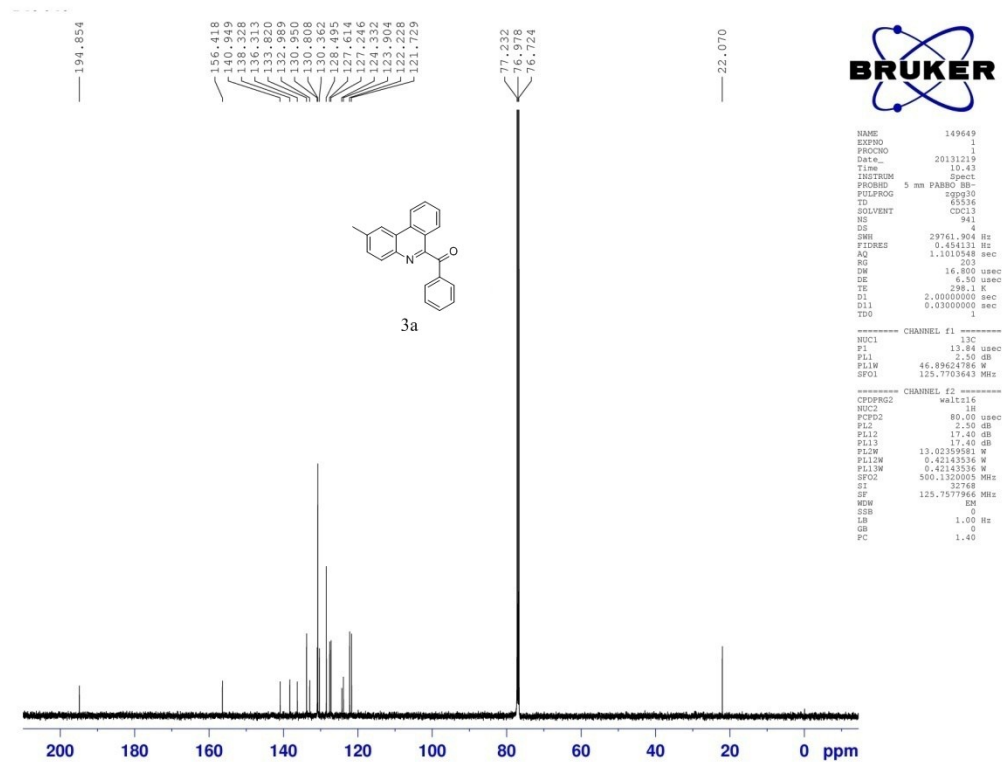
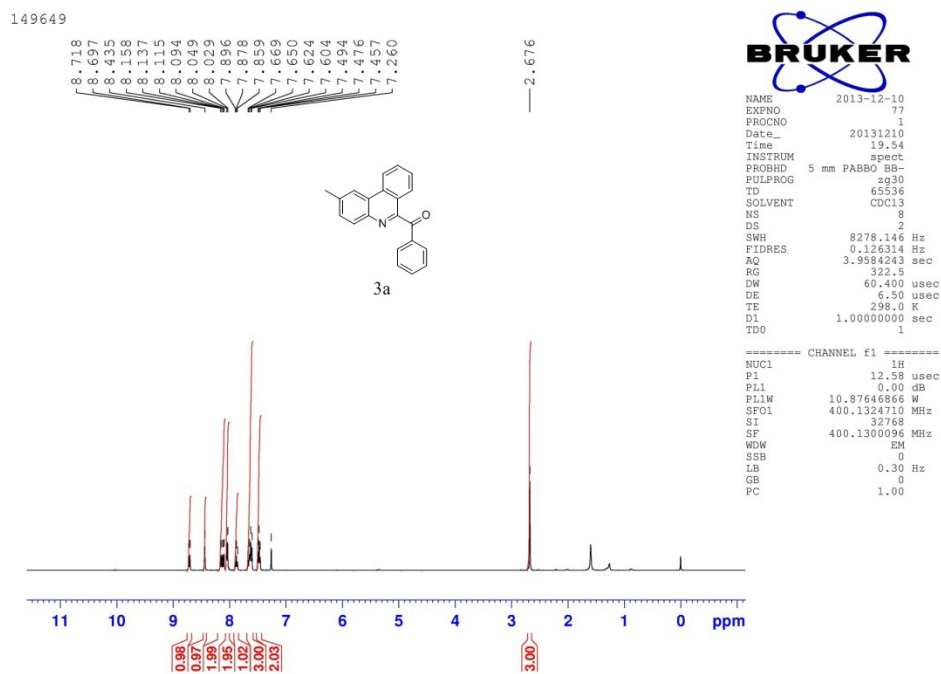


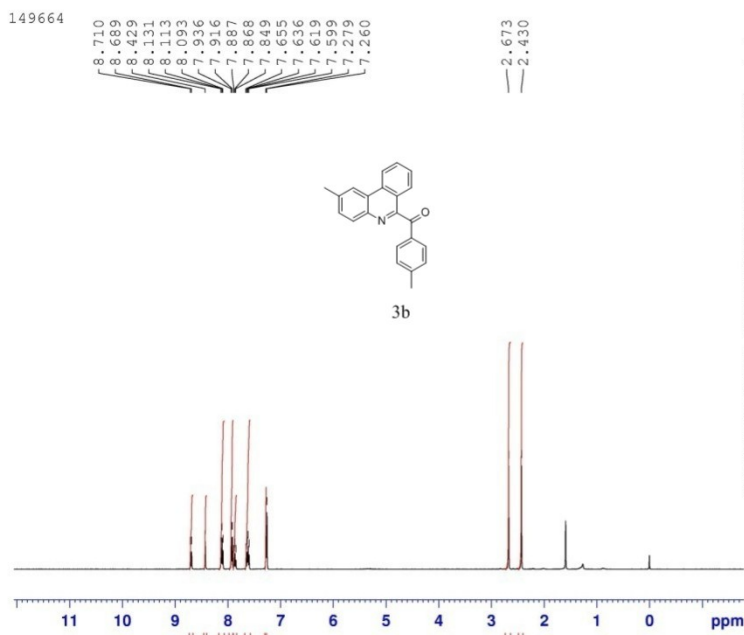
An oven-dried sealed tube was equipped with a magnetic stir bar and was charged with a mixture of 2-isocyanobiphenyl **1a** (0.2 mmol), benzaldehyde **2a** (1.2 mmol), TEMPO (0.5 mmol) and TMSN<sub>3</sub> (0.5 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (1.0 mL). Then (diacetoxyiodo)benzene (PIDA) (0.5 mmol) was dissolved in CH<sub>2</sub>Cl<sub>2</sub> (1.0 mL) and added dropwise to the reaction mixture for 10 minutes at 50 °C. After stirring for 5 minutes, the reaction was monitored by TLC analyst and almost no the targeted product **3a** was observed along with a great material **1a** remained.

## 6. References

- (1) (a) M. Tobisu, K. Koh, T. Furukawa, N. Chatani, *Angew. Chem., Int. Ed.*, 2012, **51**, 11363; (b) B. Zhang, C. Mück-Lichtenfeld, C. G. Daniliuc, A. Studer, *Angew. Chem., Int. Ed.*, 2013, **52**, 10792; (c) D. Leifert, C. G. Daniliuc, A. Studer, *Org. Lett.*, 2013, **15**, 6286; (d) X. Sun, S. Yu, *Org. Lett.*, 2014, **16**, 2938; (e) B. Zhang, C. G. Daniliuc, A. Studer, *Org. Lett.*, 2014, **16**, 250; (f) J. Liu, C. Fan, H. Yin, C. Qin, G. Zhang, X. Zhang, H. Yi, A. Lei, *Chem. Commun.*, 2014, **50**, 2145; (g) X. Li, M. Fang, P. Hu, G. Hong, Y. Tang, X. Xu, *Adv. Synth. Catal.*, 2014, **356**, 2103.
- (2) (a) Y. Cheng, X. Yuan, H. Jiang, R. Wang, J. Ma, Y. Zhang, S. Yu, *Adv. Synth. Catal.*, 2014, **356**, 2859; (b) J. Li, Y. He, S. Luo, J. Lei, J. Wang, Z. Xie, Q. Zhu, *J. Org. Chem.*, 2015, **80**, 2223.
- (3) A. R. Jagdale, J. H. Park, S. W. Youn, *J. Org. Chem.*, 2011, **76**, 7204.
- (4) (a) D. Leifert, C. G. Daniliuc, A. Studer, *Org. Lett.*, 2013, **15**, 6286; (b) J. Liu, C. Fan, H. Yin, C. Qin, G. Zhang, X. Zhang, H. Yi, A. Lei, *Chem. Commun.*, 2014, **50**, 2145.

## 7. Copies of $^1\text{H}$ NMR and $^{13}\text{C}$ NMR spectra



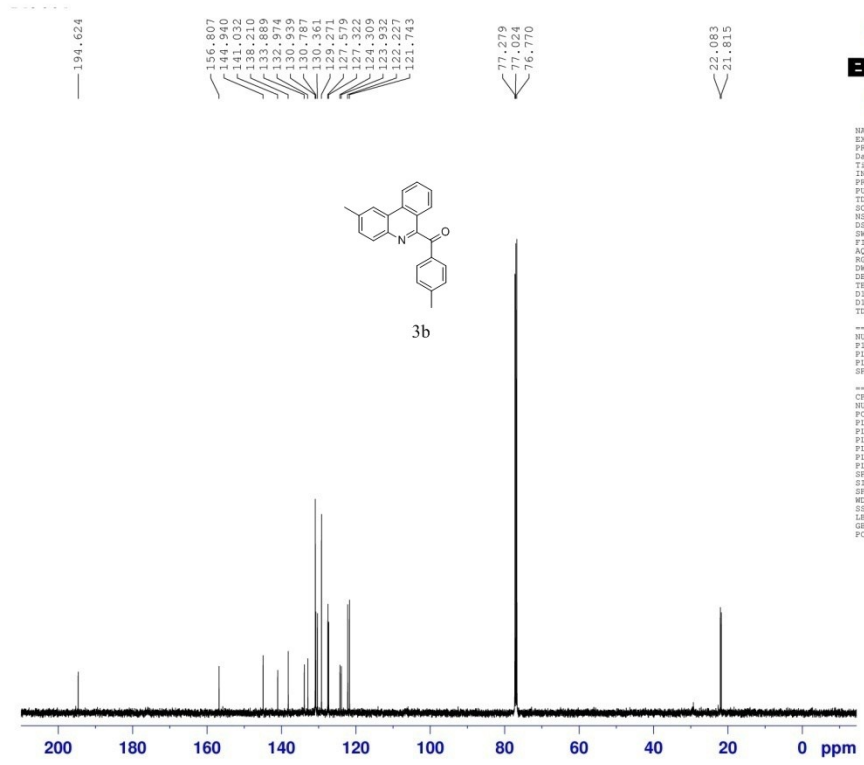


**BRUKER**

```

NAME      12-20
EXPNO    38
PROCNO   1
Date_    20131220
Time     14.55
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  zg30
TD       65536
SOLVENT  CDCl3
NS       8
DS       2
SWH      8278.146 Hz
FIDRES   0.126314 Hz
AQ       3.9584243 sec
RG       322.5
DW       60.400 usec
DE       6.50 usec
TE       298.0 K
D1       1.00000000 sec
TD0      1

===== CHANNEL f1 =====
NUC1     1H
P1       12.58 usec
PL1     0.00 dB
PL1W    10.8764686 W
SFO1    400.1324710 MHz
SI       32768
SF      400.1300094 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
  
```



**BRUKER**

```

NAME      149664
EXPNO    1
PROCNO   1
Date_    20131221
Time     16.56
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       331
DS       4
SWH      29761.904 Hz
FIDRES   0.484131 Hz
AQ       1.1010548 sec
RG       203
DW       16.800 usec
DE       6.50 usec
TE       298.0 K
D1       2.00000000 sec
D11     0.30000000 sec
TD0      1

===== CHANNEL f1 =====
NUC1     13C
P1       13.84 usec
PL1     2.50 dB
PL1W    46.89624786 W
SFO1    125.7703643 MHz

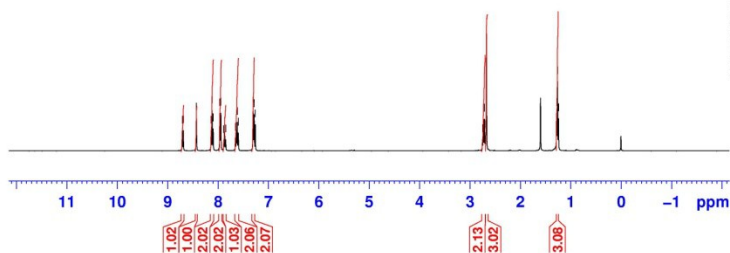
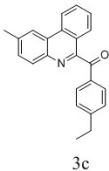
===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2   80.00 usec
PL2     2.50 dB
PL12    17.40 dB
PL13    17.40 dB
PL1W    13.02389851 W
PL12W   0.42143336 W
PL13W   0.42143336 W
SFO2    500.1320005 MHz
SI       32768
SF      125.7577930 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
  
```

149663

8.710  
8.690  
8.430  
8.137  
8.117  
8.097  
7.965  
7.946  
7.887  
7.868  
7.849  
7.849  
7.656  
7.638  
7.619  
7.599  
7.303  
7.284  
7.260

2.751  
2.732  
2.713  
2.694  
2.673

1.280  
1.262  
1.243



```

NAME      2013-12-20
EXPNO    37
PROCNO    1
Date_     20131220
Time      14.48
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         8
DS         2
SWH       8278.146 Hz
FIDRES    0.126314 Hz
AQ         3.9584243 sec
RG         362
DW         60.400 usec
DE         6.50 usec
TE         298.0 K
D1         1.00000000 sec
TDO        1
  
```

```

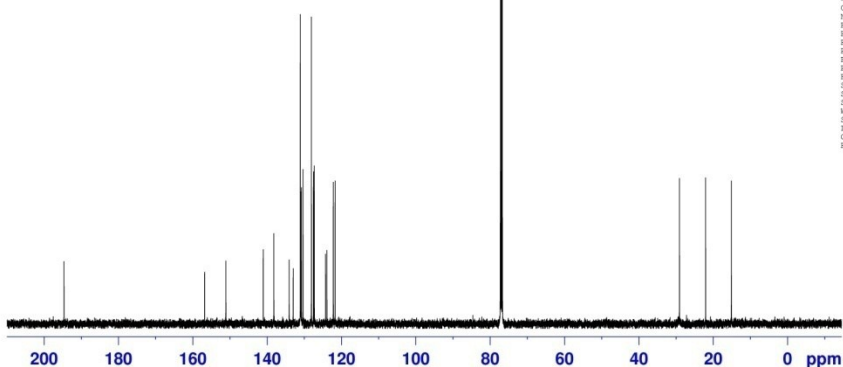
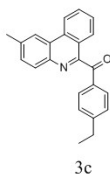
----- CHANNEL f1 -----
NUC1      1H
P1        12.58 usec
PL1       0.00 dB
PL1W      10.87646866 W
SF01      400.1324710 MHz
SI        32768
SF        400.1300094 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
  
```

194.632

156.840  
151.100  
141.043  
138.211  
134.071  
131.076  
131.076  
130.933  
130.792  
130.376  
128.122  
128.122  
127.388  
124.322  
123.954  
122.230  
121.753

77.288  
77.033  
76.779

29.124  
22.092  
15.146



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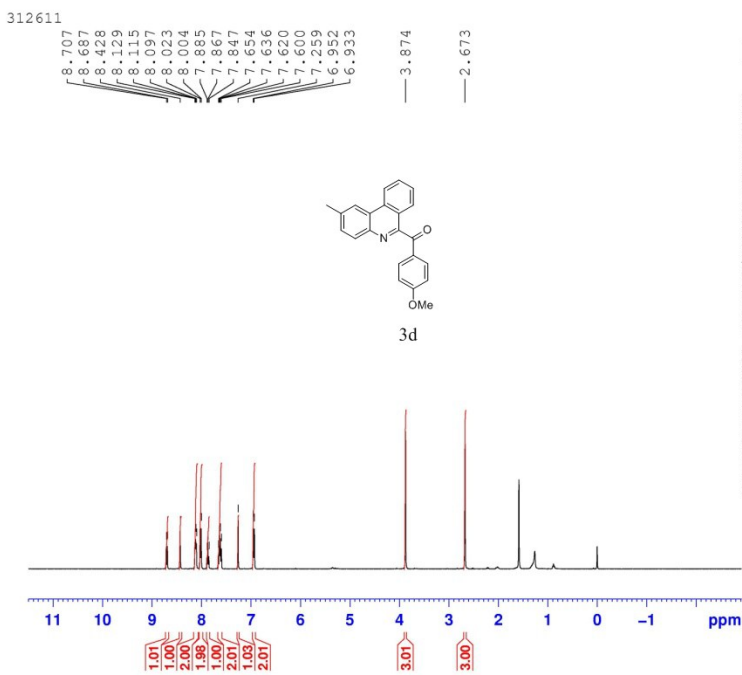
NAME      149663
EXPNO    1
PROCNO    1
Date_     20131221
Time      16.35
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         4
DS         4
SWH       29761.904 Hz
FIDRES    0.454131 Hz
AQ         1.1010583 sec
RG         203
DW         16.800 usec
DE         6.50 usec
TE         298.0 K
D1         2.00000000 sec
D11        0.33000000 sec
TDO        1
  
```

```

----- CHANNEL f1 -----
NUC1      13C
P1        13.84 usec
PL1       2.50 dB
PL1W      46.89624786 W
SF01      125.7703643 MHz
  
```

```

----- CHANNEL f2 -----
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       2.50 dB
PL12      17.40 dB
PL13      17.40 dB
PL1W      13.0239981 W
PL12W     0.42143336 W
PL13W     0.42143336 W
SF02      500.1320005 MHz
SI        32768
SF        125.7577919 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
  
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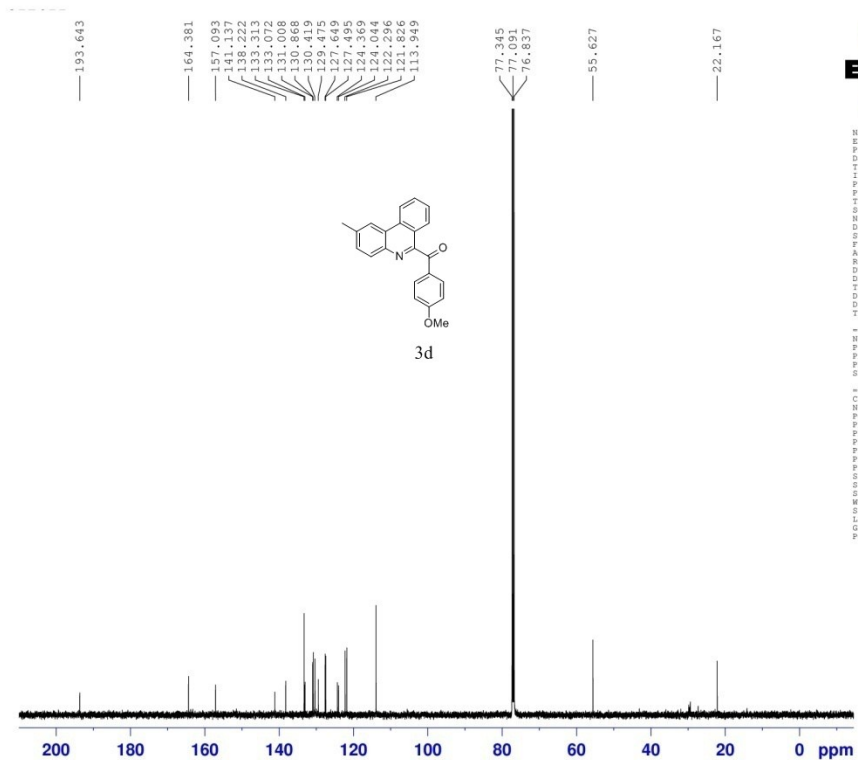


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NAME      2013-12-12
EXPNO    82
PROCNO    1
Date_     20131212
Time      19.05
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         8
DS         2
SWH        8278.146 Hz
FIDRES     0.126314 Hz
AQ         3.9584243 sec
RG         362
DW         60.400 usec
DE         6.50 usec
TE         298.0 K
D1         1.00000000 sec
TDO        1
  
```

```

===== CHANNEL f1 =====
NUC1      1H
P1        12.58 usec
PL1       0.00 dB
PL1W      10.87646866 W
SFO1      400.1324710 MHz
SI        32768
SF        400.1300095 MHz
WDS       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
  
```



```

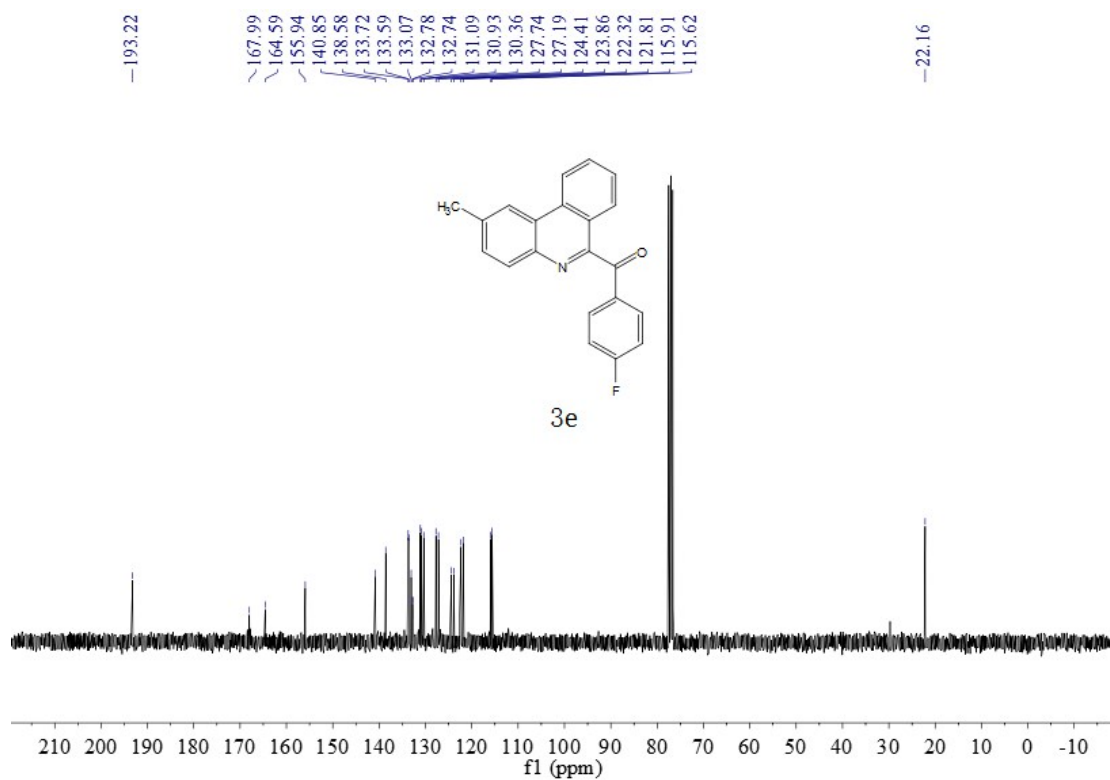
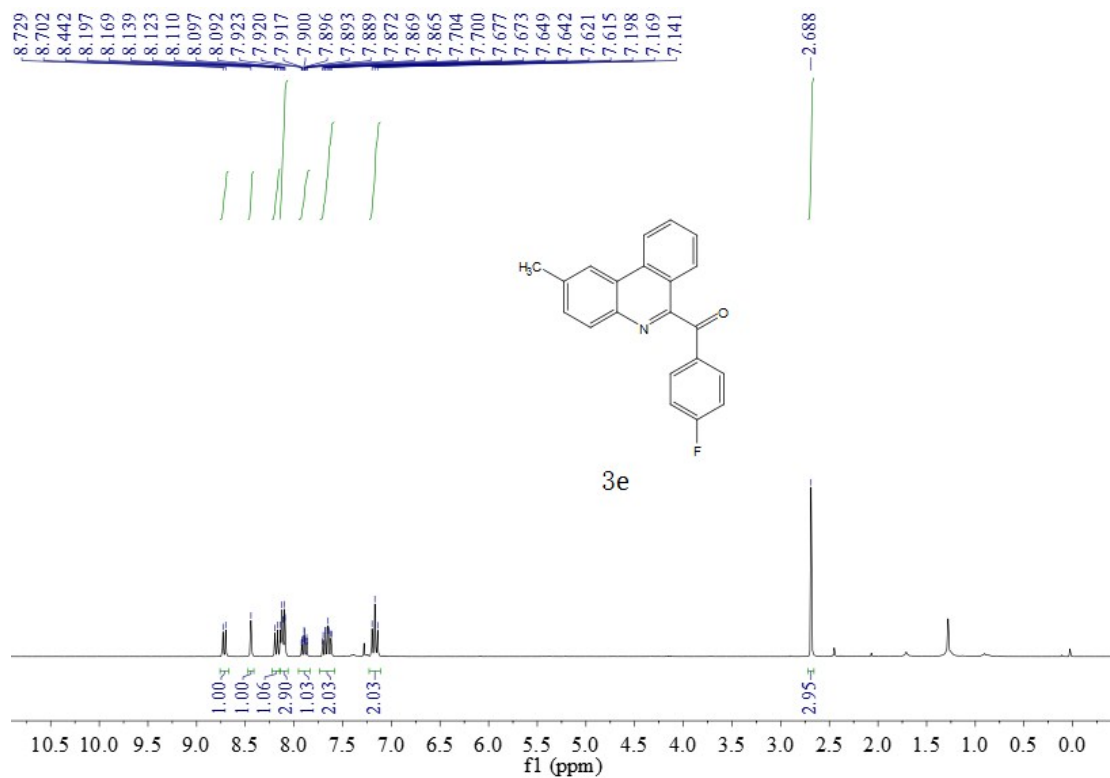
NAME      312611
EXPNO    1
PROCNO    1
Date_     20131221
Time      19.41
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         1024
DS         4
SWH        29761.904 Hz
FIDRES     0.454131 Hz
AQ         1.1010548 sec
RG         203
DW         16.800 usec
DE         6.50 usec
TE         298.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TDO        1
  
```

```

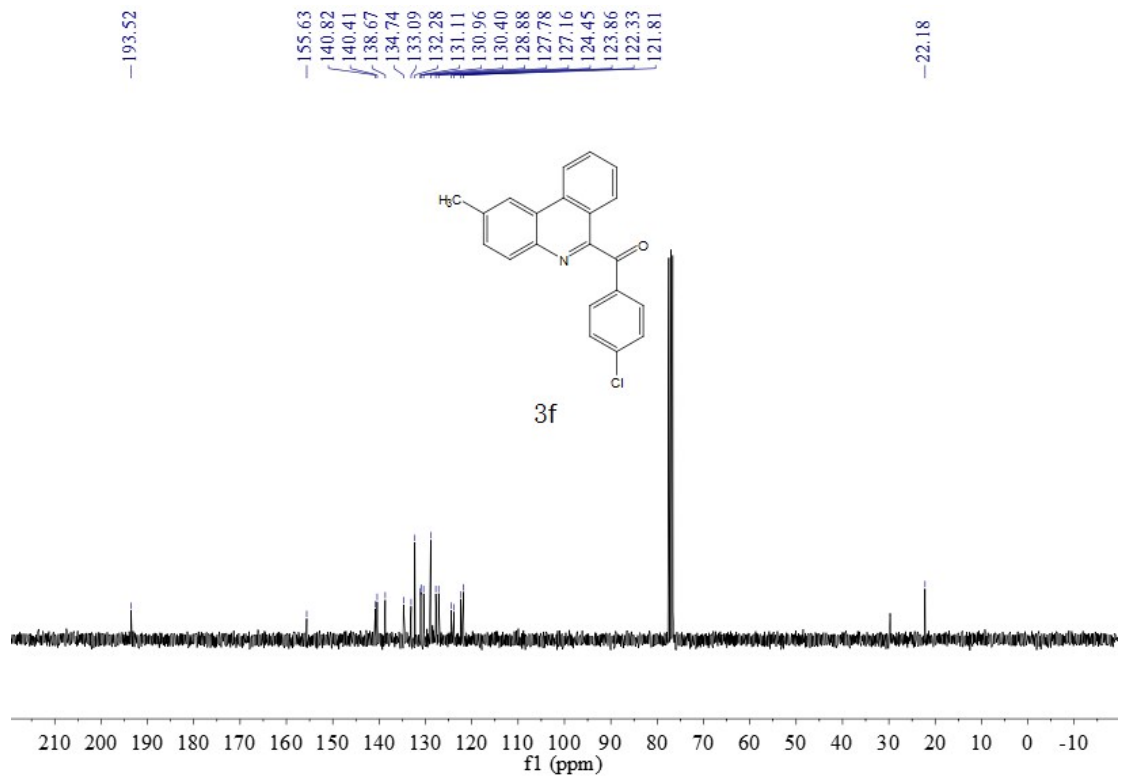
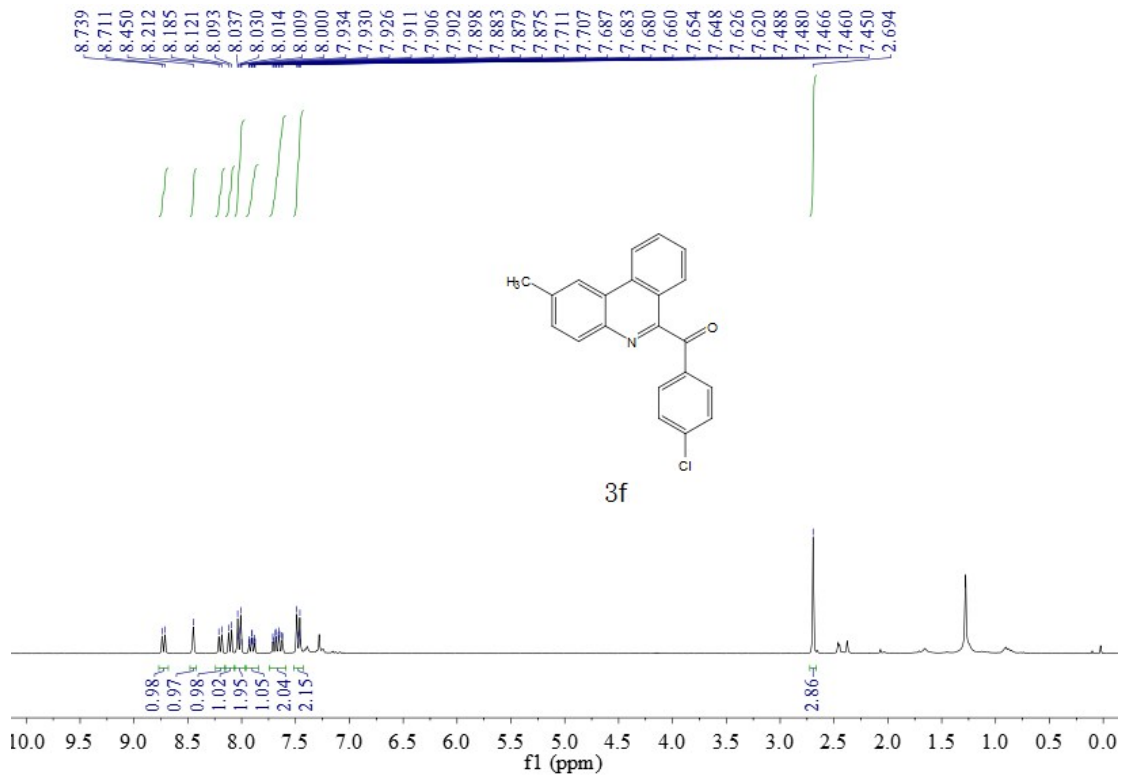
===== CHANNEL f1 =====
NUC1      13C
P1        13.84 usec
PL1       2.50 dB
PL1W      46.89624786 W
SFO1      125.7703643 MHz
  
```

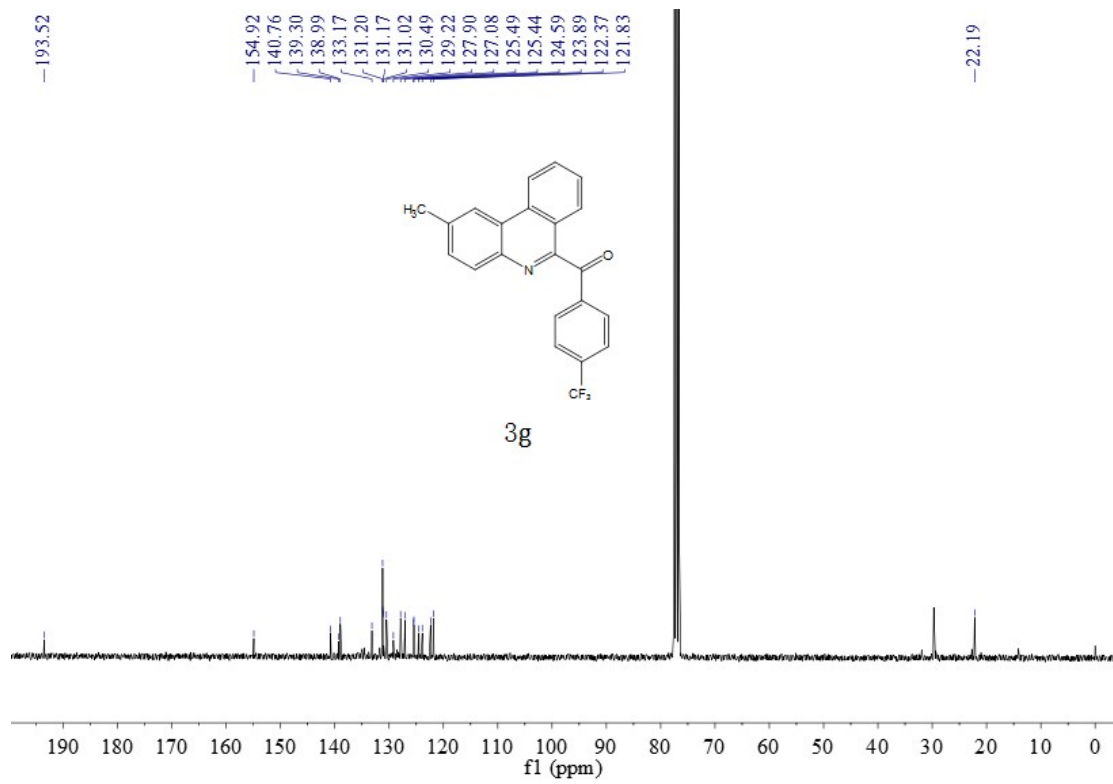
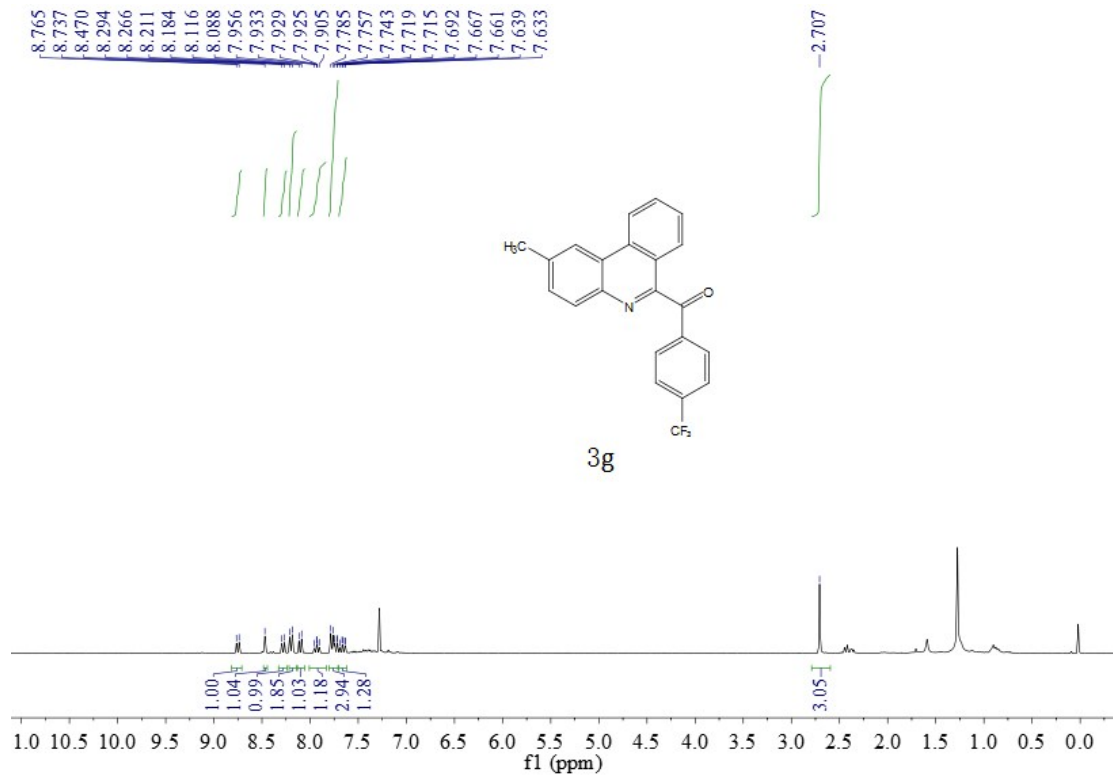
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===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       2.50 dB
PL12      17.40 dB
PL13      17.40 dB
PL1W      13.0239981 W
PL12W     0.42143336 W
PL13W     0.42143336 W
SFO2      500.1320005 MHz
SI        32768
SF        125.7577817 MHz
WDS       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
  
```

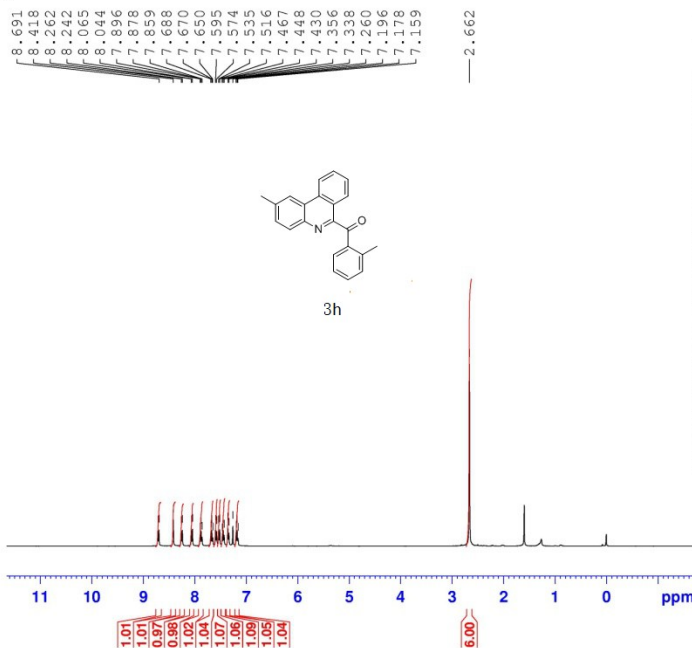








312511

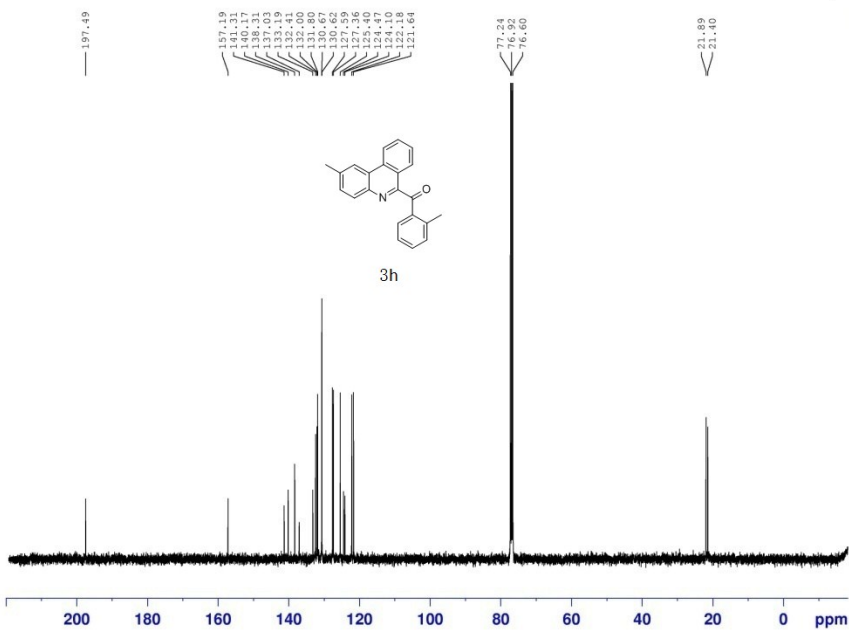


```

NAME      2013-12-11
EXPNO    52
PROCNO    1
Date_     20131211
Time      16.46
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         8
DS         2
SWH        8278.146 Hz
FIDRES     0.126314 Hz
AQ         3.9584243 sec
RG         362
DW         60.400 usec
DE         6.50 usec
TE         298.0 K
D1         1.0000000 sec
TDO        1

===== CHANNEL f1 =====
NUC1      1H
P1        12.58 usec
PL1       0.00 dB
PL1W      10.87646866 W
SFO1      400.1324710 MHz
SI        32768
SF        400.1300094 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
  
```

313511

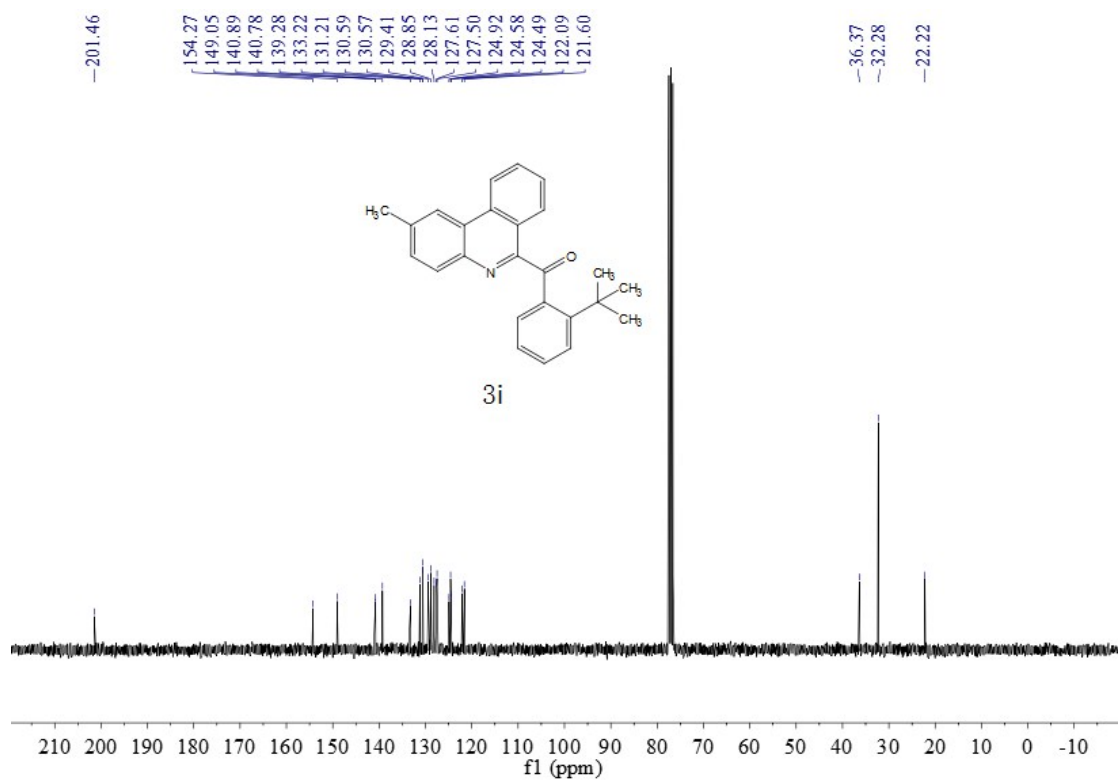
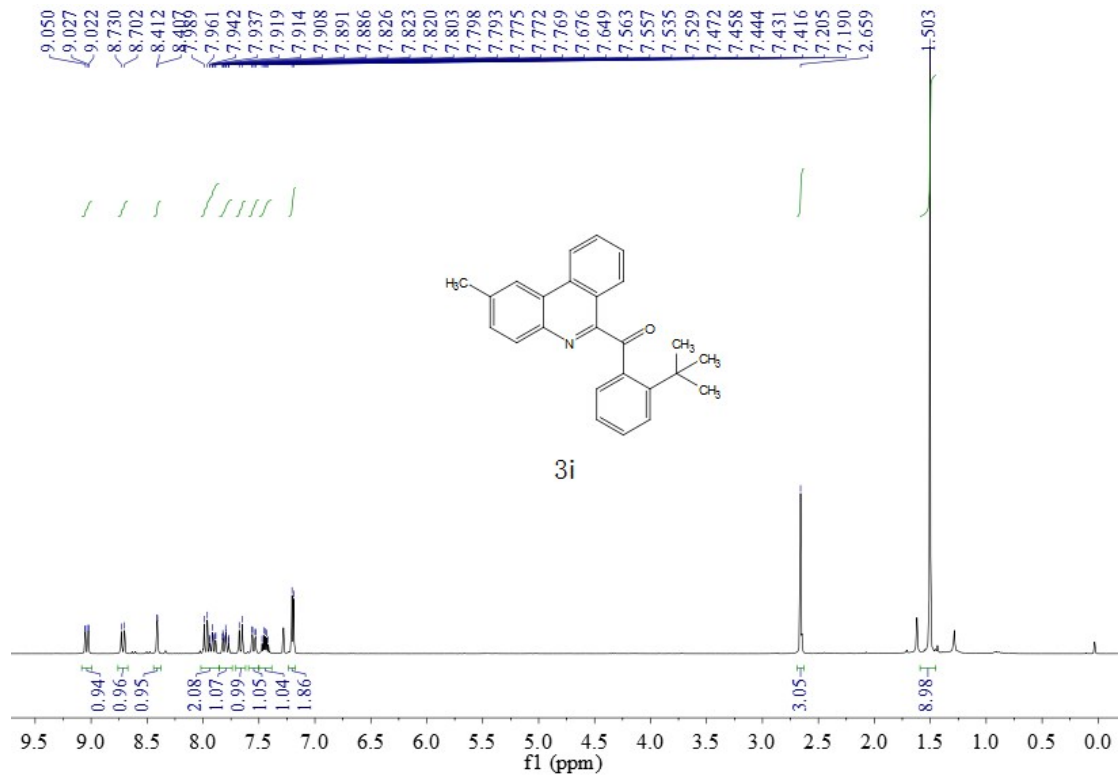


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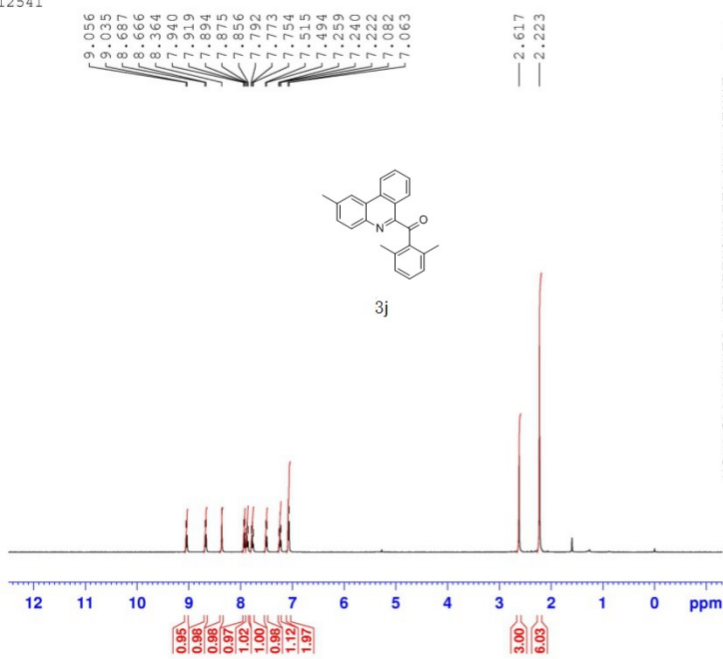
NAME      13-19
EXPNO    1
PROCNO    1
Date_     20131219
Time      8.54
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         1024
DS         4
SWH        23980.814 Hz
FIDRES     0.263928 Hz
AQ         1.3664756 sec
RG         1024
DW         20.850 usec
DE         6.50 usec
TE         313.1 K
D1         2.0000000 sec
D11        0.0300000 sec
TDO        1

===== CHANNEL f1 =====
NUC1      13C
P1        10.23 usec
PL1       0.00 dB
PL1W      38.69385206 W
SFO1      100.6282828 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      13C
P2        80.00 usec
PL2       0.00 dB
PL2W      14.07 dB
PL3       0.00 dB
PL3W      10.87646866 W
PL1W      0.26883630 W
SFO2      400.1326800 MHz
SI        32768
SF        100.6127659 MHz
WDW       RM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
  
```



312541



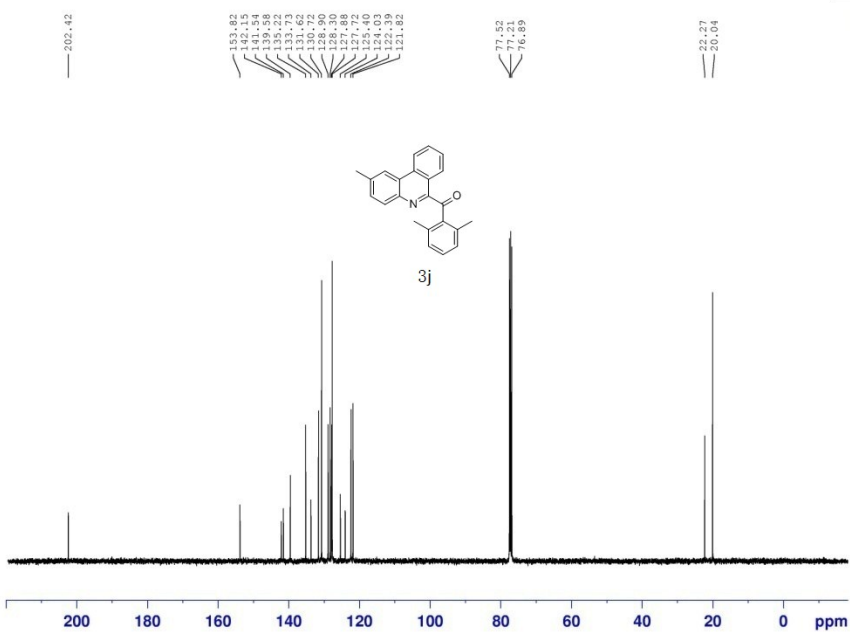
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NAME      2013-12-11
EXPNO    53
PROCNO   1
Date_    20131211
Time     16.54
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  zg30
TD        65536
SOLVENT  CDCl3
NS        8
DS        2
SWH       8278.146 Hz
FIDRES   0.126314 Hz
AQ        3.9584243 sec
RG        128
DW        60.400 usec
DE        6.50 usec
TE        298.0 K
D1        1.00000000 sec
TDO       1

===== CHANNEL f1 =====
NUC1      1H
P1        12.58 usec
PL1       0.00 dB
PL1W      10.87646866 W
SF01      400.1324710 MHz
SI        32768
SF        400.1300182 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00

```

312541



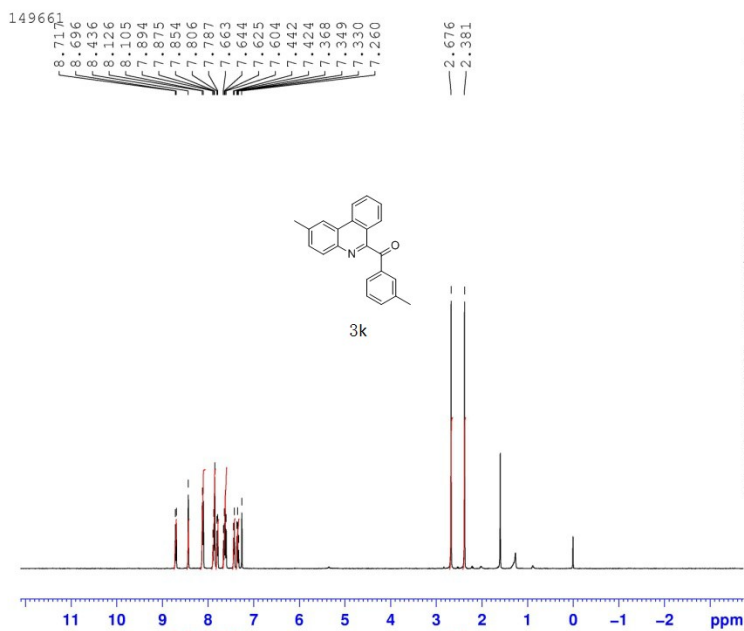
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NAME      13-13
EXPNO    87
PROCNO   1
Date_    20131213
Time     23.44
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD        45336
SOLVENT  CDCl3
NS        4
DS        4
SWH       23982.814 Hz
FIDRES   0.265928 Hz
AQ        1.3664756 sec
RG        1024
DW        20.850 usec
DE        6.50 usec
TE        311.9 K
D1        2.00000000 sec
D11       0.03000000 sec
TDO       1

===== CHANNEL f1 =====
NUC1      13C
P1        10.23 usec
PL1       0.00 dB
PL1W      38.6938206 W
SF01      100.6282898 MHz

===== CHANNEL f2 =====
CPROG2   waltz16
NUC2      1H
P2        80.00 usec
PL2       0.00 dB
PL2W      14.07 dB
PL3       0.00 dB
PL3W      10.87646866 W
PL1W      0.26883630 W
SF02      400.1326500 MHz
SI        32768
SF        100.6127401 MHz
WDW       RM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40

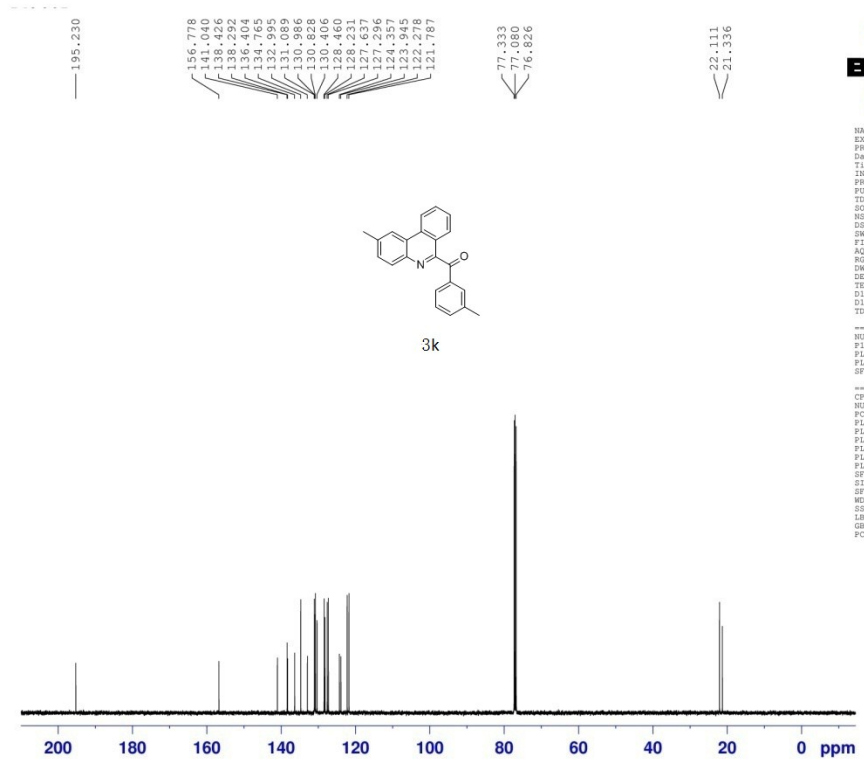
```



```

NAME      2013-12-20
EXPNO    36
PROCNO    1
Date_     20131220
Time      14.40
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         8
DS         2
SWH       8278.146 Hz
FIDRES    0.126314 Hz
AQ         3.9584243 sec
RG         362
DW         60.400 usec
DE         6.50 usec
TE         298.0 K
D1         1.00000000 sec
TDO        1

===== CHANNEL f1 =====
NUC1       1H
P1         12.58 usec
PL1        0.00 dB
PL1W       10.87646866 W
SFO1       400.1324710 MHz
SI         32768
SF         400.1300094 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```



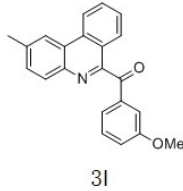
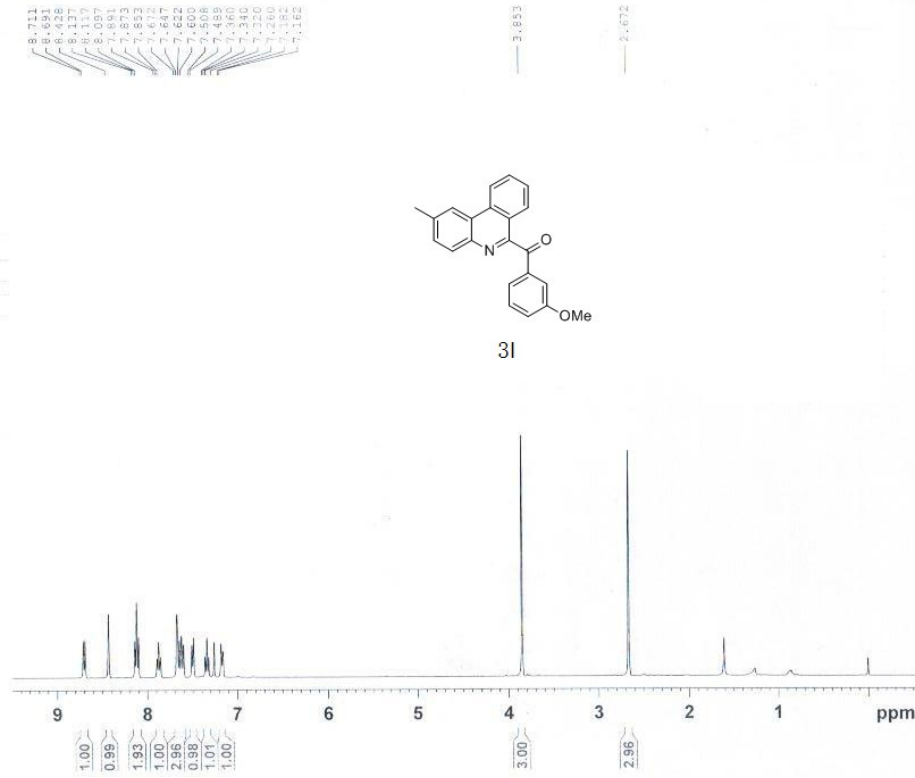
```

NAME      149661
EXPNO    2
PROCNO    1
Date_     20131221
Time      15.41
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         320
DS         4
SWH       29761.904 Hz
FIDRES    0.454131 Hz
AQ         1.010583 sec
RG         203
DW         16.800 usec
DE         6.50 usec
TE         298.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TDO        1

===== CHANNEL f1 =====
NUC1       13C
P1         13.84 usec
PL1        2.50 dB
PL1W       46.89624786 W
SFO1       125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2     80.00 usec
PL2        2.50 dB
PL2W      17.40 dB
PL3        7.40 dB
PL3W      13.0239881 W
PL12W     0.42143336 W
PL13W     0.42143336 W
SFO2       500.1320005 MHz
SI         32768
SF         125.7577903 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```

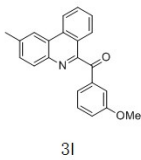
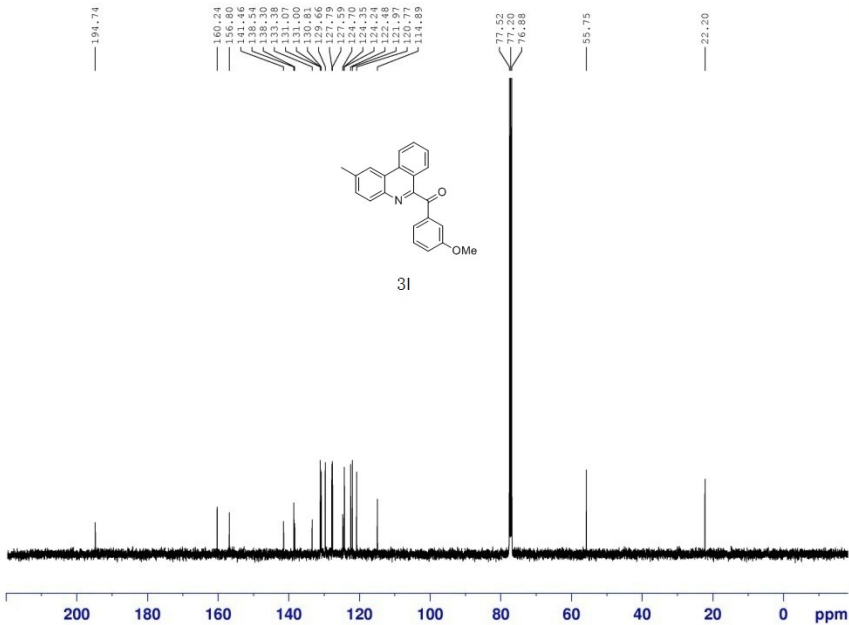
31263



```

NAME      Nov29-2013
EXPNO    45
PROCNO   1
Date_    2011119
Time     22:52
INSTRUM  spect
PROBHD   5 mm EBBB-BA-
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        8
DS        2
SWH       8278.146 Hz
FIDRES   0.176514 Hz
AQ        1.9584253 sec
RG        753
WDW       EM
SSB       0.000000 sec
DE        6.50 usec
TE        298.0 K
D1        1.0000000 sec
D11       1
TD0       1
===== CHANNEL f1 =====
NUC1      13C
P1        12.50 usec
PL1       0.00 dB
PC1M      10.07646866 MHz
SF1       400.1324710 MHz
SI        32768
SF        400.1300000 MHz
MEM       5M
SOLVENT  CDCl3
IR        0.000000
GB        5
PC        1.20
    
```

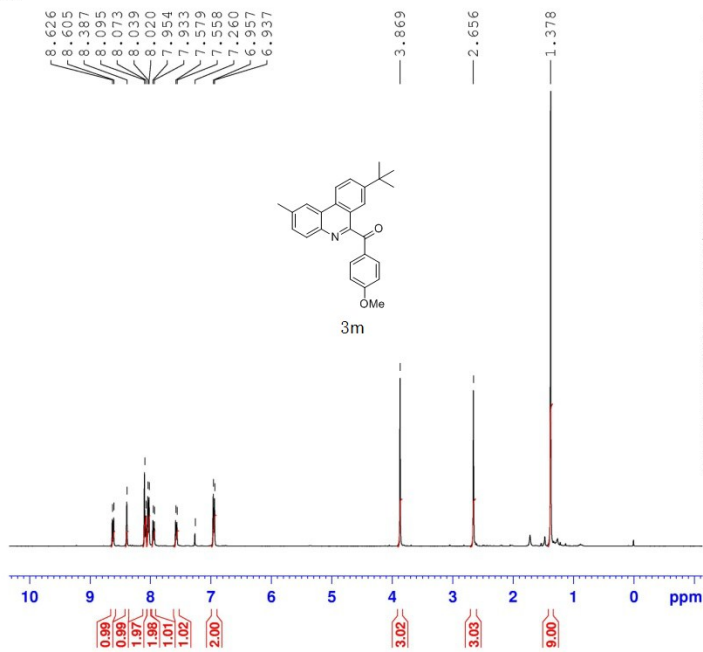
31263



```

NAME      12-11
EXPNO    46
PROCNO   1
Date_    2011111
Time     22:12
INSTRUM  spect
PROBHD   5 mm PABBO-BA-
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        1024
DS        4
SWH       23980.814 Hz
FIDRES   0.263998 Hz
AQ        1.3461975 sec
RG        1024
WDW       EM
SSB       0.000000 sec
DE        6.50 usec
TE        312.5 K
D1        2.0000000 sec
D11       0.0000000 sec
TD0       1
===== CHANNEL f1 =====
NUC1      13C
P1        10.25 usec
PL1       0.00 dB
PC1M      100.6288288 MHz
SF1       100.6288288 MHz
===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2      1H
PCPD2    80.00 usec
PL2      0.00 dB
PL12     16.07 dB
PL13     0.00 dB
PL1W     10.07646866 MHz
PL2W     5.04898266 MHz
PL13W    10.07646866 MHz
SF02     100.1316051 MHz
SI        32768
SF        100.6127375 MHz
MEM       8M
SOLVENT  CDCl3
IR        1.00 Hz
GB        0
PC        1.40
    
```

312921

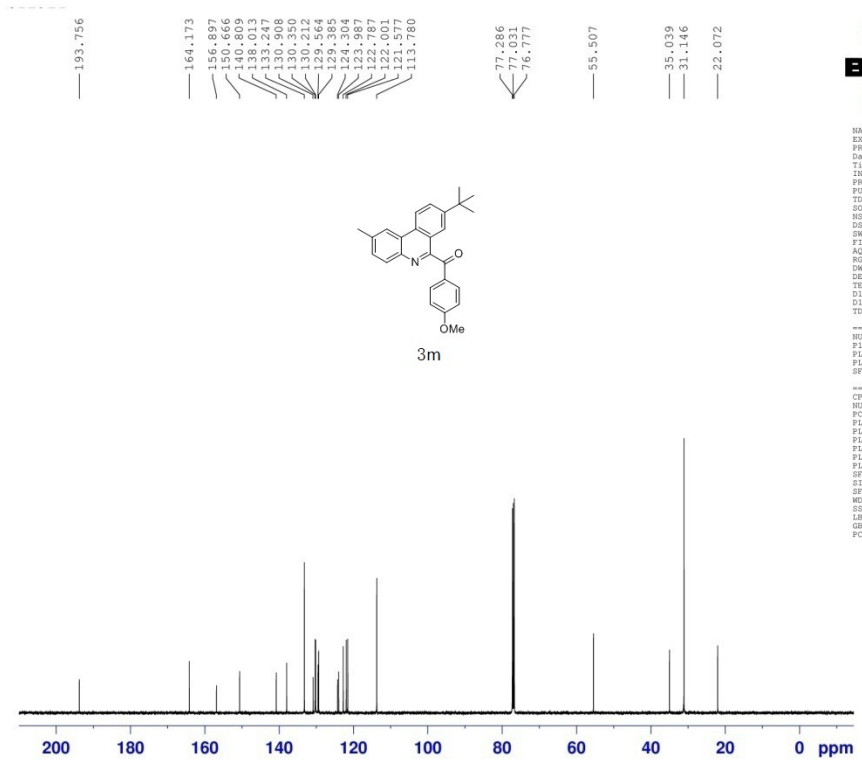


**BRUKER**

```

NAME      2013-12-12
EXPNO    20
PROCNO    1
Date_     20131212
Time      11.26
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         8
DS         2
SWH        8278.146 Hz
FIDRES     0.126314 Hz
AQ         3.9584243 sec
RG         90.5
DW         60.400 usec
DE         6.50 usec
TE         298.0 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1      1H
P1         12.58 usec
PL1        0.00 dB
PL1W      10.87646866 W
SFO1      400.1324710 MHz
SI         32768
SF         400.1300093 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
    
```



**BRUKER**

```

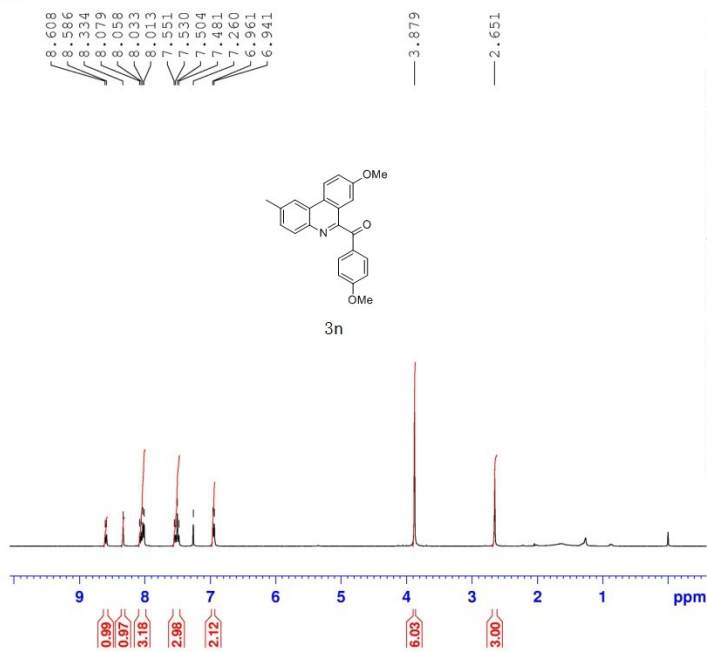
NAME      312921
EXPNO    1
PROCNO    1
Date_     20131221
Time      18.41
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         658
DS         4
SWH        29761.904 Hz
FIDRES     0.454131 Hz
AQ         1.1010548 sec
RG         203
DW         16.800 usec
DE         6.50 usec
TE         298.0 K
D1         2.00000000 sec
D11        0.33000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1      13C
P1         13.84 usec
PL1        2.50 dB
PL1W      46.89624786 W
SFO1      125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2        2.50 dB
PL2W      17.40 dB
PL3        7.40 dB
PL3W      13.0239981 W
PL12W     0.42143336 W
PL13W     0.42143336 W
PL1W      8.42143336 W
SFO2      500.1320005 MHz
SI         32768
SF         125.7577950 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
    
```



149651

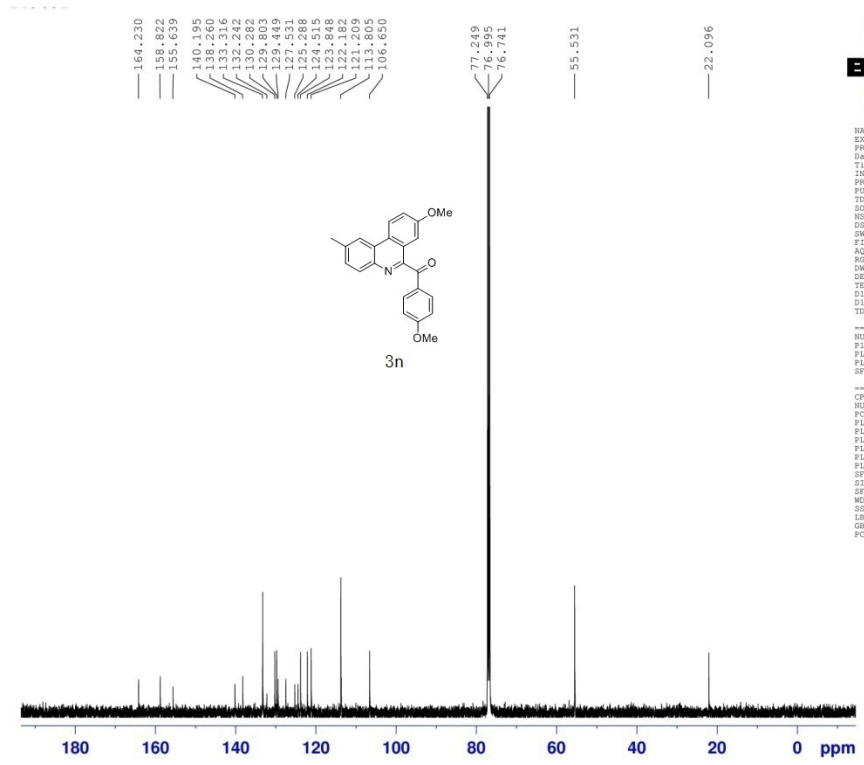


```

NAME      2013-12-10
EXPNO    78
PROCNO   1
Date_    20131210
Time     20.01
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  zg30
TD        65536
SOLVENT  CDCl3
NS        8
DS        2
SWH      8278.146 Hz
FIDRES   0.126314 Hz
AQ        3.9584243 sec
RG        362
DW        60.400 usec
DE        6.50 usec
TE        298.0 K
D1        1.00000000 sec
TDO       1
  
```

```

===== CHANNEL f1 =====
NUC1      1H
P1        12.58 usec
PL1       0.00 dB
PL1W     10.87646866 W
SFO1     400.1324710 MHz
SI        32768
SF        400.1300093 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
  
```



```

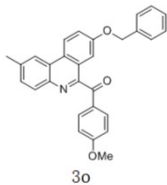
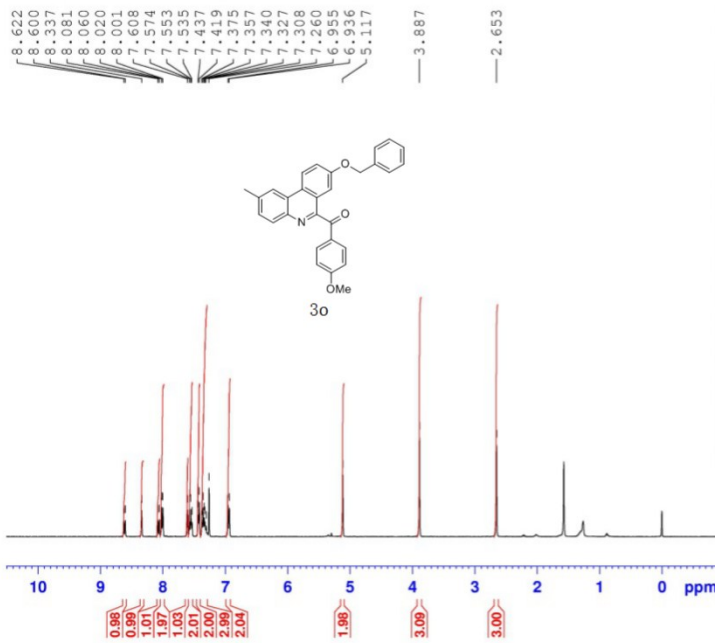
NAME      149651
EXPNO    1
PROCNO   1
Date_    20131221
Time     17.18
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        1024
DS        4
SWH      29761.904 Hz
FIDRES   0.454131 Hz
AQ        1.010548 sec
RG        203
DW        16.800 usec
DE        6.50 usec
TE        298.0 K
D1        2.00000000 sec
D11       0.03000000 sec
TDO       1
  
```

```

===== CHANNEL f1 =====
NUC1      13C
P1        13.84 usec
PL1       2.50 dB
PL1W     46.89624796 W
SFO1     125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2      1H
PCPD2    80.00 usec
PL2       2.50 dB
PL2W     17.40 dB
PL3       7.40 dB
PL3W     13.02399881 W
PL12W    0.42143336 W
PL13W    0.42143336 W
SFO2     500.1320005 MHz
SI        32768
SF        125.7577939 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
  
```

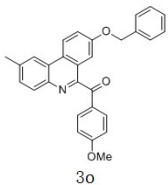
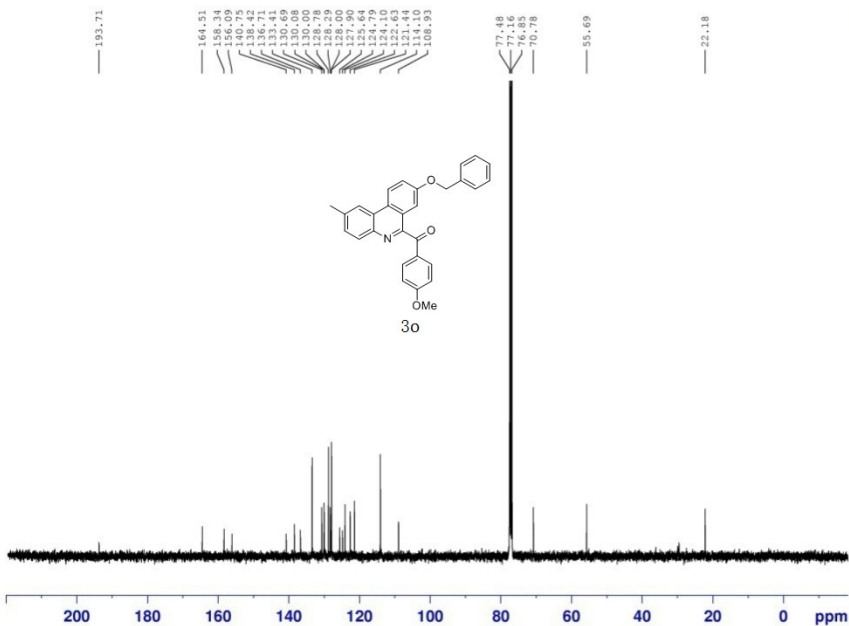
312771



NAME 2013-12-12  
EXPNO 17  
PROCNO 1  
Date\_ 20131212  
Time 11.04  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 8  
DS 2  
SWH 8278.146 Hz  
FIDRES 0.126314 Hz  
AQ 3.9584243 sec  
RG 362  
DW 60.400 usec  
DE 6.50 usec  
TE 298.0 K  
D1 1.0000000 sec  
TDO 1

----- CHANNEL f1 -----  
NUC1 1H  
P1 12.58 usec  
PL1 0.00 dB  
PL1W 10.87646866 W  
SFO1 400.1324710 MHz  
SI 32768  
SF 400.1300094 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

312771



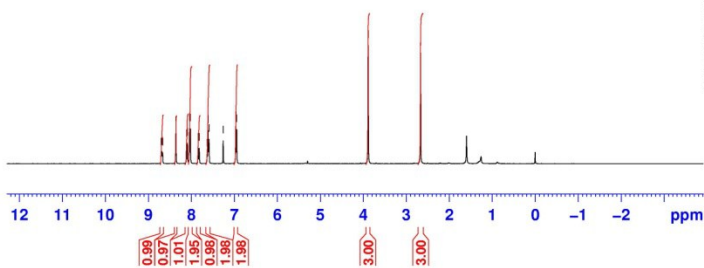
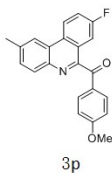
NAME 12-17  
EXPNO 1  
PROCNO 1  
Date\_ 20131217  
Time 2.24  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 2048  
DS 4  
SWH 23982.814 Hz  
FIDRES 0.963928 Hz  
AQ 1.3664756 sec  
RG 1239.2  
DW 20.850 usec  
DE 6.50 usec  
TE 311.4 K  
D1 2.0000000 sec  
D11 0.0300000 sec  
TDO 1

----- CHANNEL f1 -----  
NUC1 13C  
P1 10.23 usec  
PL1 0.00 dB  
PL1W 38.6938206 W  
SFO1 100.628298 MHz

----- CHANNEL f2 -----  
CPROG2 waltz16  
NUC2 1H  
P2 80.00 usec  
PL2 0.00 dB  
PL2W 14.07 dB  
P13 0.00 dB  
P13W 10.87646866 W  
P14W 0.24683620 W  
SFO2 400.1324710 MHz  
SI 32768  
SF 100.6127401 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

313041

8.703  
8.688  
8.680  
8.668  
8.361  
8.113  
8.092  
8.044  
8.024  
7.843  
7.819  
7.632  
7.591  
7.260  
6.966  
6.947  
— 3.885  
— 2.667

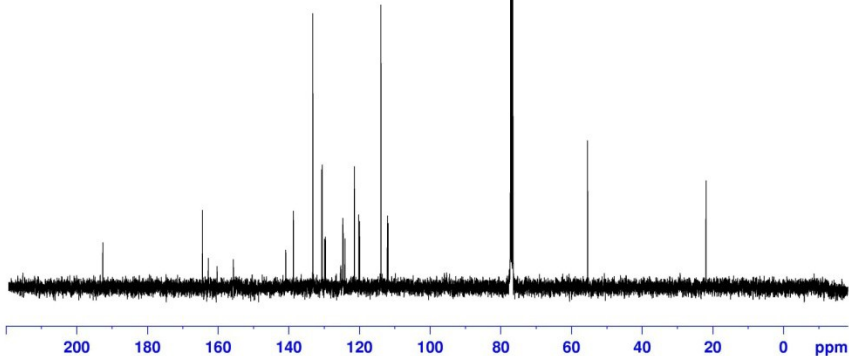
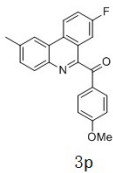


NAME 2013-12-11  
EXPNO 55  
PROCNO 1  
Date\_ 20131211  
Time 17.08  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 8  
DS 2  
SWH 8278.146 Hz  
FIDRES 0.126314 Hz  
AQ 3.9584243 sec  
RG 362  
DW 60.400 usec  
DE 6.50 usec  
TE 298.0 K  
D1 1.0000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 12.58 usec  
PL1 0.00 dB  
PL1W 10.87646866 W  
SFO1 400.1324710 MHz  
SI 32768  
SF 400.1300096 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

313041

192.62  
164.39  
162.77  
160.30  
155.66  
138.68  
133.19  
130.64  
129.84  
129.60  
124.71  
124.63  
121.42  
120.18  
119.90  
112.09  
111.87  
77.21  
76.89  
76.58  
— 55.43  
— 21.90

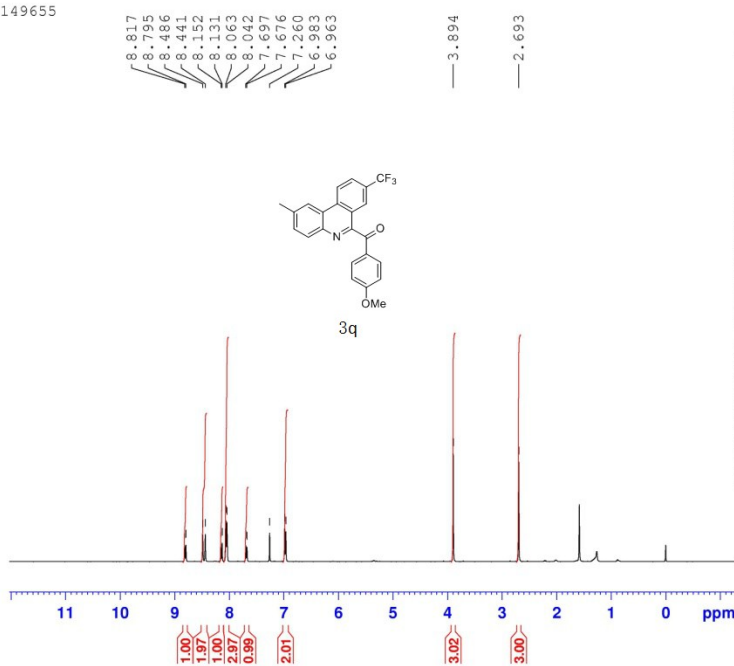


NAME 13-19  
EXPNO 84  
PROCNO 1  
Date\_ 20131219  
Time 7.48  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zgpg30  
TD 45336  
SOLVENT CDCl3  
NS 1024  
DS 4  
SWH 23982.814 Hz  
FIDRES 0.265993 Hz  
AQ 1.3664756 sec  
RG 612.7  
DW 20.850 usec  
DE 6.50 usec  
TE 313.3 K  
D1 2.0000000 sec  
D11 0.0300000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 13C  
P1 10.25 usec  
PL1 0.00 dB  
PL1W 38.6938206 W  
SFO1 100.6282898 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PROC2 80.00 usec  
P12 0.00 dB  
PL12 14.07 dB  
P13 0.00 dB  
PL13 10.87646866 W  
PL1W 0.26883636 W  
SFO2 400.1324710 MHz  
SI 32768  
SF 100.6127659 MHz  
WDW RM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

149655

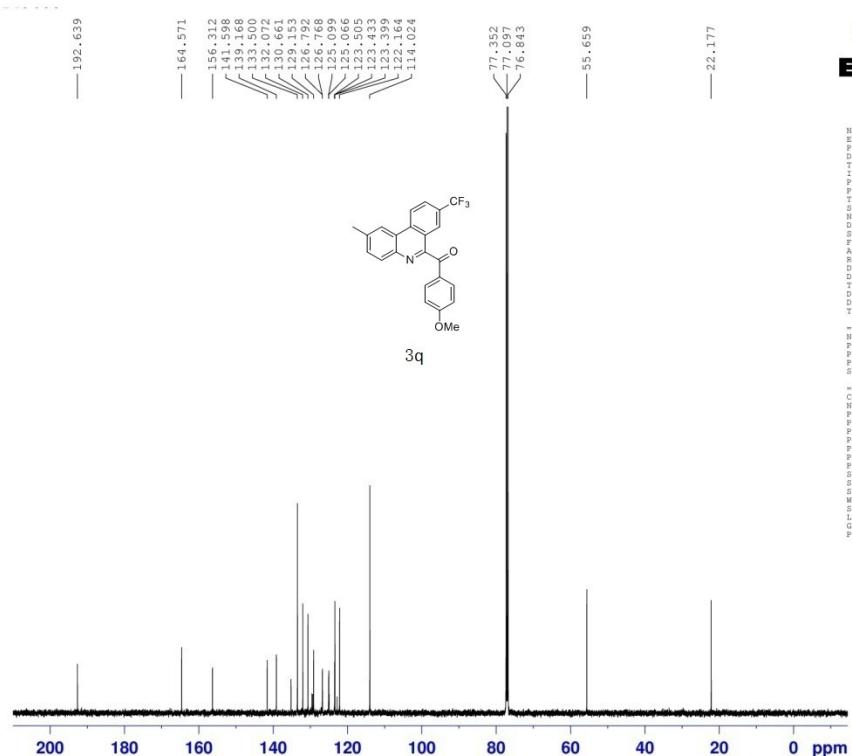


```

NAME      2013-12-13
EXPNO    48
PROCNO    1
Date_    20131213
Time     15.03
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD        65536
SOLVENT   CDCl3
NS        8
DS        2
SWH       8278.146 Hz
FIDRES    0.126314 Hz
AQ        3.9584243 sec
RG        362
DW        60.400 usec
DE        6.50 usec
TE        298.0 K
D1        1.00000000 sec
TDO       1
  
```

```

===== CHANNEL f1 =====
NUC1      1H
P1        12.58 usec
PL1       0.00 dB
PL1W      10.87646856 W
SFO1      400.1324710 MHz
SI        32768
SF        400.1300094 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
  
```



```

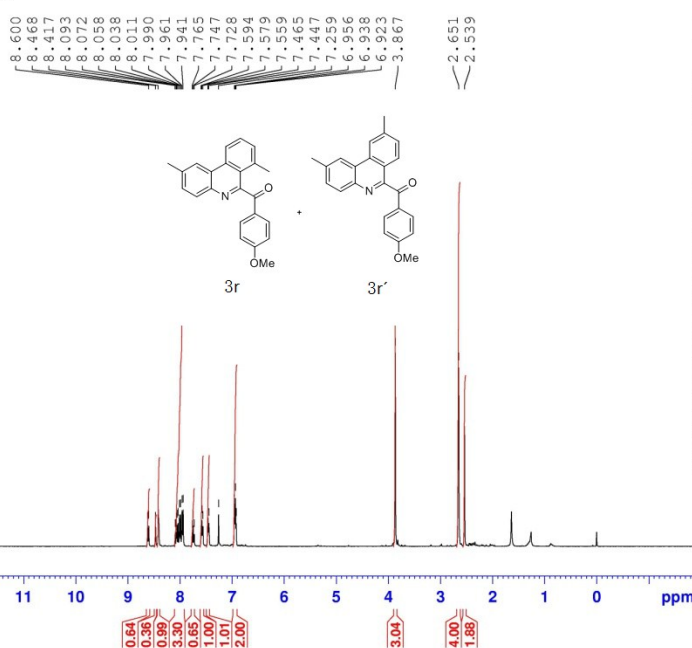
NAME      149655
EXPNO    1
PROCNO    1
Date_    20131218
Time     11.57
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS        781
DS        4
SWH       29761.904 Hz
FIDRES    0.454131 Hz
AQ        1.1010548 sec
RG        203
DW        16.800 usec
DE        6.50 usec
TE        298.0 K
D1        2.00000000 sec
D11       0.03000000 sec
TDO       1
  
```

```

===== CHANNEL f1 =====
NUC1      13C
P1        13.84 usec
PL1       2.50 dB
PL1W      46.89624786 W
SFO1      125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       2.50 dB
PL12      17.40 dB
PL13      7.40 dB
PL1W      13.0239881 W
PL12W     0.42143336 W
PL13W     0.42143336 W
SF        500.1320005 MHz
SI        32768
SF        125.7577820 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
  
```

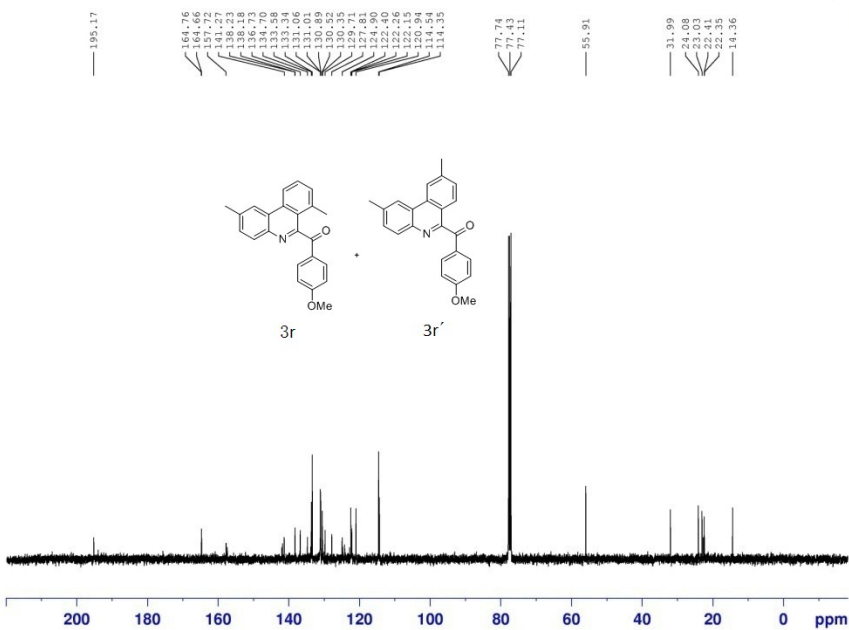
312901



NAME 2013-12-12  
 EXPNO 19  
 PROCNO 1  
 Date\_ 20131212  
 Time 11.19  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 8  
 DS 2  
 SWH 8278.146 Hz  
 FIDRES 0.126314 Hz  
 AQ 3.9584243 sec  
 RG 256  
 DW 60.400 usec  
 DE 6.50 usec  
 TE 298.0 K  
 D1 1.0000000 sec  
 TDO 1

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 12.58 usec  
 PL1 0.00 dB  
 PL1W 10.87646866 W  
 SF01 400.1324710 MHz  
 SI 32768  
 SF 400.1300096 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

312901



NAME 12-14  
 EXPNO 31  
 PROCNO 1  
 Date\_ 20131214  
 Time 21.05  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zgpg30  
 TD 45336  
 SOLVENT CDCl3  
 NS 600  
 DS 4  
 SWH 23982.814 Hz  
 FIDRES 0.265938 Hz  
 AQ 1.3664756 sec  
 RG 1149.4  
 DW 20.850 usec  
 DE 6.50 usec  
 TE 311.6 K  
 D1 2.0000000 sec  
 D11 0.0000000 sec  
 TDO 1

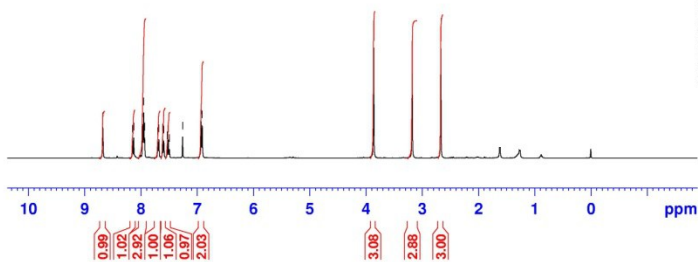
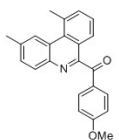
===== CHANNEL f1 =====  
 NUC1 13C  
 P1 10.23 usec  
 PL1 0.00 dB  
 PL1W 30.6930206 W  
 SF01 100.6282898 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 P2 80.00 usec  
 PL2 0.00 dB  
 PL2W 14.07 dB  
 PL3 0.00 dB  
 PL3W 10.87646866 W  
 PL4W 0.26883630 W  
 SF02 400.1324710 MHz  
 SF 400.1300096 MHz  
 SI 32768  
 SF 100.6127147 MHz  
 WDW RM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

313031

8.679  
8.151  
8.130  
7.971  
7.953  
7.937  
7.702  
7.684  
7.616  
7.595  
7.533  
7.514  
7.495  
7.260  
6.932  
6.913

3.861  
3.179  
2.670



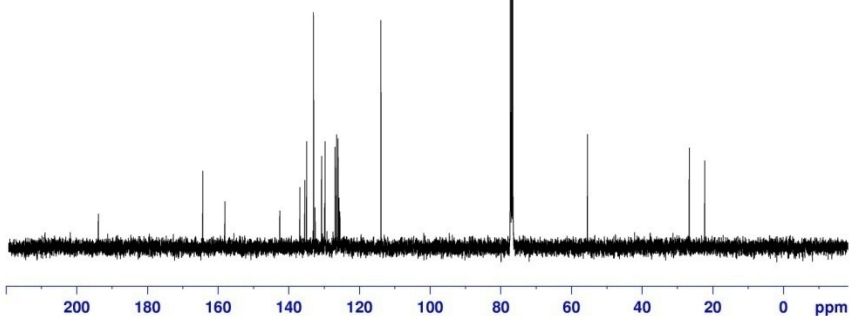
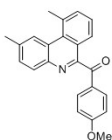
NAME 2013-12-12  
EXPNO 83  
PROCNO 1  
Date\_ 20131212  
Time 19.13  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 8  
DS 2  
SWH 8278.146 Hz  
FIDRES 0.126314 Hz  
AQ 3.9584243 sec  
RG 322.5  
DW 60.400 usec  
DE 6.50 usec  
TE 298.0 K  
D1 1.0000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 12.58 usec  
PL1 0.00 dB  
PL1W 10.87646866 W  
SFO1 400.1324710 MHz  
SI 32768  
SF 400.1300094 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

313031

193.92  
164.34  
158.12  
142.56  
136.90  
133.86  
133.00  
132.94  
129.85  
129.74  
126.45  
126.05  
125.33  
113.93

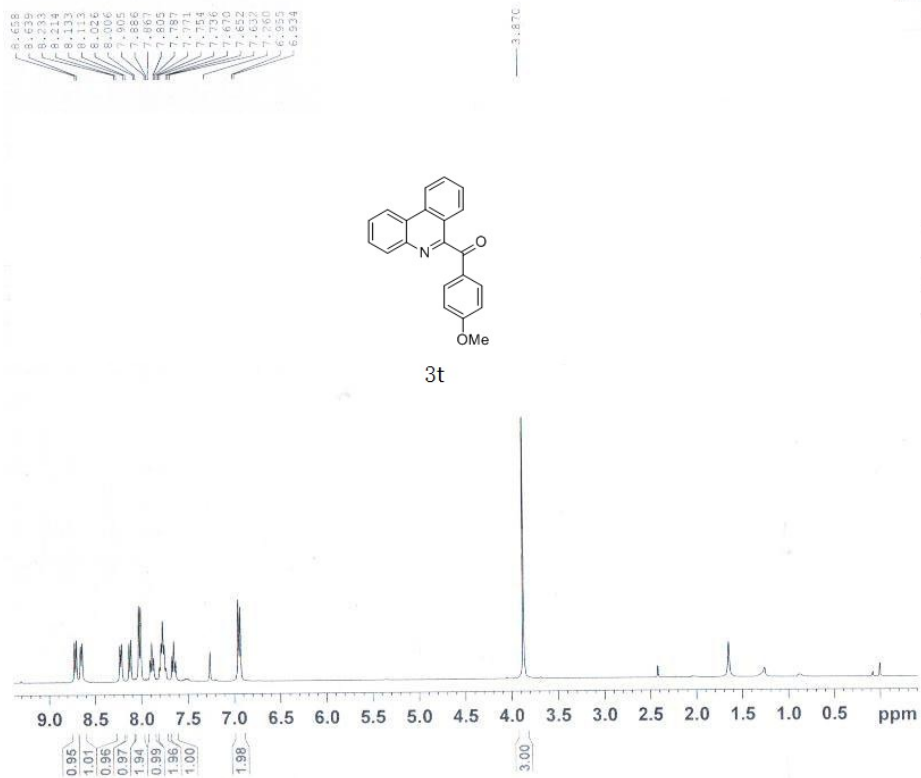
55.45  
26.67  
22.26



NAME 13-13  
EXPNO 101  
PROCNO 1  
Date\_ 20131213  
Time 1.55  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zgpg30  
TD 45336  
SOLVENT CDCl3  
NS 1024  
DS 4  
SWH 23982.814 Hz  
FIDRES 0.265993 Hz  
AQ 1.3664756 sec  
RG 612.7  
DW 20.850 usec  
DE 6.50 usec  
TE 312.0 K  
D1 2.0000000 sec  
D11 0.0300000 sec  
TD 1

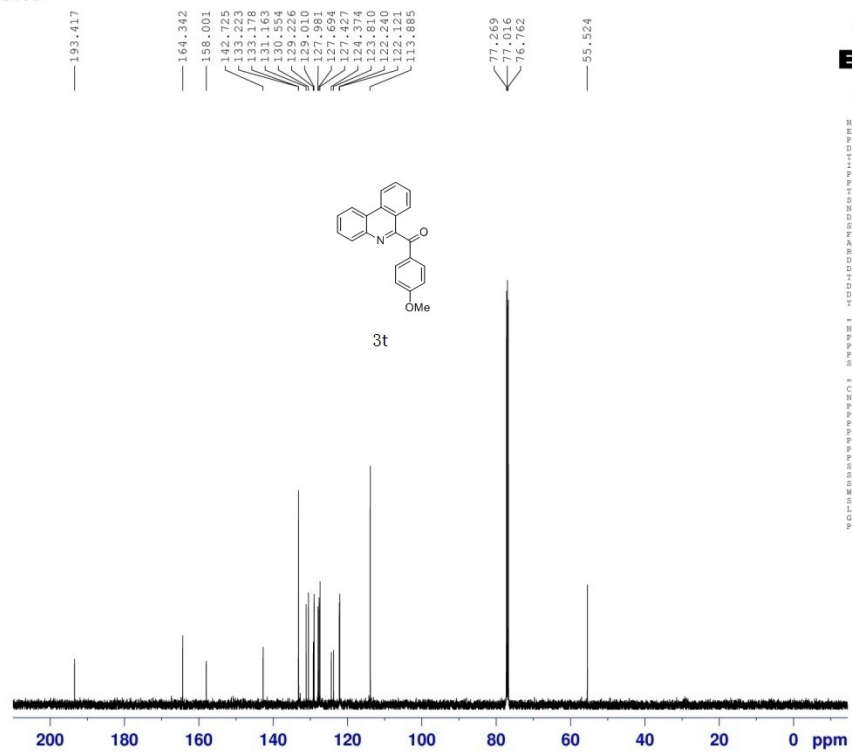
===== CHANNEL f1 =====  
NUC1 13C  
P1 10.23 usec  
PL1 0.00 dB  
PL1W 38.6930206 W  
SFO1 100.6282898 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 80.00 usec  
PL2 0.00 dB  
PL2W 14.07 dB  
PL3 0.00 dB  
PL3W 10.87646866 W  
PL1W 0.26883630 W  
PL1W 10.87646866 W  
SFO2 400.1326505 MHz  
SI 32768  
SF 100.6127625 MHz  
WDW RM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40



```
NAME      30x29-2013  
EXPNO      15  
PROCNO      1  
Date_      20131129  
Time        10.51  
INSTRUM     spect  
PROBHD      5 mm PABBO B9-  
PULPROG     zg30  
PC        65536  
SOLVENT      CDCl3  
NS           8  
DS           2  
SWH          8278.146 Hz  
FIDRES      0.176314 Hz  
AQ          1.358243 sec  
RG           756  
EQ          66.400 usec  
LB           6.50 usec  
TE           296.4 K  
D1           1.30000000 sec  
TDO           1
```

```
===== CHANNEL f1 =====  
NUC1      1H  
P1        12.58 usec  
PL1       0.20 dB  
FLW       13.87658862 MHz  
SFO1      400.1324710 MHz  
SI         32768  
SF        400.1308555 MHz  
WDW       EM  
SSB       0  
LB         6.30 Hz  
GB         0  
PC         1.00
```



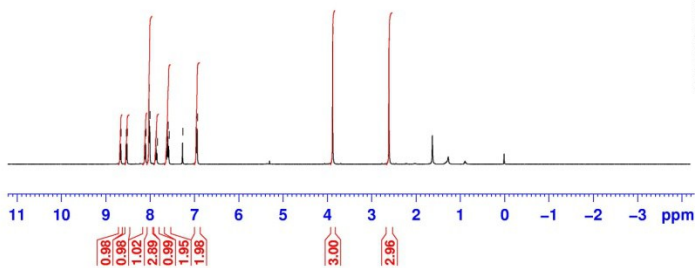
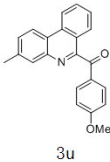
```
NAME      31317  
EXPNO      1  
PROCNO      1  
Date_      20131219  
Time        12.08  
INSTRUM     spect  
PROBHD      5 mm PABBO BB-  
PULPROG     zgpg30  
TD          65536  
SOLVENT      CDCl3  
NS           162  
DS           4  
SWH          29761.904 Hz  
FIDRES      0.454131 Hz  
AQ          1.1010548 sec  
RG           203  
SW          16.800 usec  
EQ          6.50 usec  
TE           298.1 K  
D1           2.00000000 sec  
D11         0.03000000 sec  
TDO           1
```

```
===== CHANNEL f1 =====  
NUC1      13C  
P1        13.84 usec  
PL1       2.50 dB  
FLW       46.89624786 MHz  
SFO1      125.7703643 MHz
```

```
===== CHANNEL f2 =====  
CFPROG2    waltz16  
NUC2      1H  
PCPRG2     80.00 usec  
PL2        2.50 dB  
PL12       17.40 dB  
PL13       17.40 dB  
PL1W       13.02359081 MHz  
FL1W       0.42435536 MHz  
FL12W      0.42435536 MHz  
SFO2       500.1320053 MHz  
SI         32768  
SF         125.7577866 MHz  
WDW       EM  
SSB       0  
LB         1.00 Hz  
GB         0  
PC         1.40
```

313181

8.653  
8.536  
8.515  
8.115  
8.094  
8.019  
8.000  
7.877  
7.858  
7.838  
7.631  
7.612  
7.593  
7.572  
7.260  
6.951  
6.931  
3.872  
2.602



```

NAME      2013-12-12
EXPNO    22
PROCNO    1
Date_     20131212
Time      11.41
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         8
DS         2
SWH       8278.146 Hz
FIDRES    0.126314 Hz
AQ         3.9584243 sec
RG         322.5
DW         60.400 usec
DE         6.50 usec
TE         298.0 K
D1         1.0000000 sec
TDO        1

```

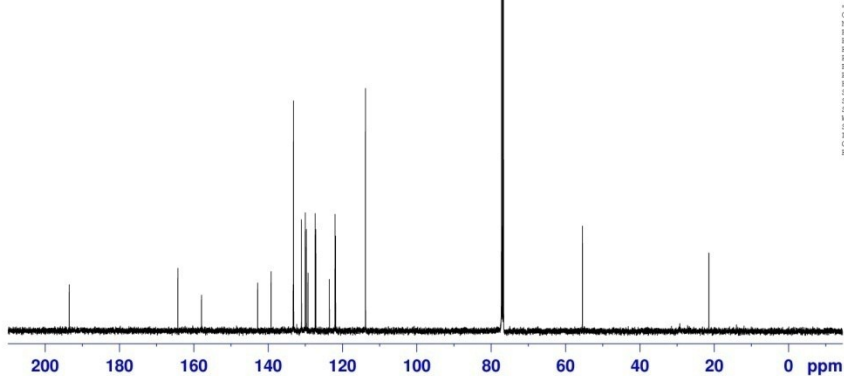
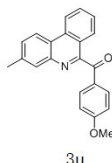
```

===== CHANNEL f1 =====
NUC1      1H
P1        12.58 usec
PL1       0.00 dB
PL1W      10.87646866 W
SFO1      400.1324710 MHz
SI        32768
SF        400.1300095 MHz
WDS       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00

```

313181

193.491  
164.277  
157.926  
142.846  
139.260  
133.298  
133.170  
130.687  
130.057  
129.748  
129.306  
127.385  
127.210  
122.061  
122.033  
121.886  
113.843  
77.242  
76.988  
76.734  
55.513  
21.480



```

NAME      31381
EXPNO    1
PROCNO    1
Date_     20131219
Time      11.33
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         858
DS         4
SWH       29761.904 Hz
FIDRES    0.454131 Hz
AQ         1.1010548 sec
RG         203
DW         16.800 usec
DE         6.50 usec
TE         298.1 K
D1         2.0000000 sec
D11        0.3300000 sec
TDO        1

```

```

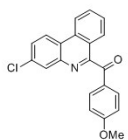
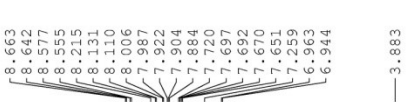
===== CHANNEL f1 =====
NUC1      13C
P1        13.84 usec
PL1       2.50 dB
PL1W      46.89624786 W
SFO1      125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       2.50 dB
PL12      17.40 dB
PL13      7.40 dB
PL1W      13.0239881 W
PL12W     0.42143336 W
PL13W     0.42143336 W
SFO2      500.1320005 MHz
SI        32768
SF        125.7577966 MHz
WDS       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40

```



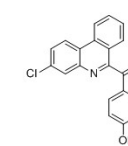
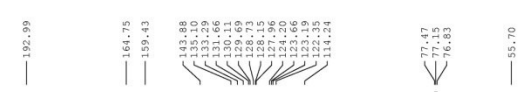
313131



NAME 2013-12-12  
 EXPNO 84  
 PROCNO 1  
 Date\_ 20131212  
 Time 19.20  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 8  
 DS 2  
 SWH 8278.146 Hz  
 FIDRES 0.126314 Hz  
 AQ 3.9584243 sec  
 RG 362  
 DW 60.400 usec  
 DE 6.50 usec  
 TE 298.0 K  
 D1 1.0000000 sec  
 TDO 1

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 12.58 usec  
 PL1 0.00 dB  
 PL1W 10.87646866 W  
 SF01 400.1324710 MHz  
 SI 32768  
 SF 400.1300097 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

313131



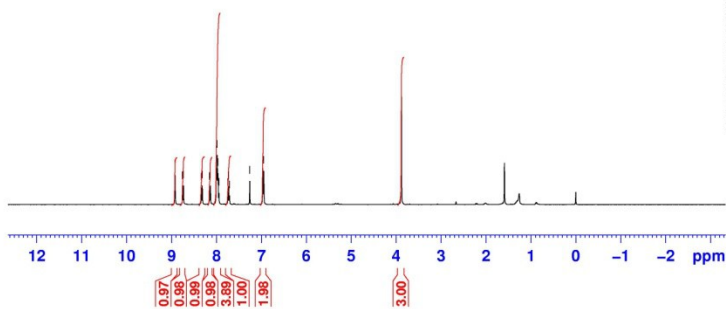
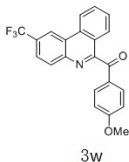
NAME 12-17  
 EXPNO 3  
 PROCNO 1  
 Date\_ 20131217  
 Time 6.06  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zgpg30  
 TD 45336  
 SOLVENT CDCl3  
 NS 2048  
 DS 4  
 SWH 23982.814 Hz  
 FIDRES 0.265928 Hz  
 AQ 1.3664756 sec  
 RG 1024  
 DW 20.850 usec  
 DE 6.50 usec  
 TE 312.1 K  
 D1 2.0000000 sec  
 D11 0.0000000 sec  
 TDO 1

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 10.23 usec  
 PL1 0.00 dB  
 PL1W 38.6938206 W  
 SF01 100.6282898 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 POC2 80.00 usec  
 PL2 0.00 dB  
 PL2 14.07 dB  
 PL3 0.00 dB  
 PL3W 10.87646866 W  
 PL1W 0.26883630 W  
 SF02 400.1324710 MHz  
 SF 400.1300097 MHz  
 SI 32768  
 SF 100.6127401 MHz  
 WDW RM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

313051

8.924  
8.757  
8.736  
8.338  
8.317  
8.157  
8.137  
8.004  
7.966  
7.969  
7.946  
7.748  
7.730  
7.710  
7.260  
6.969  
6.949  
— 3.884

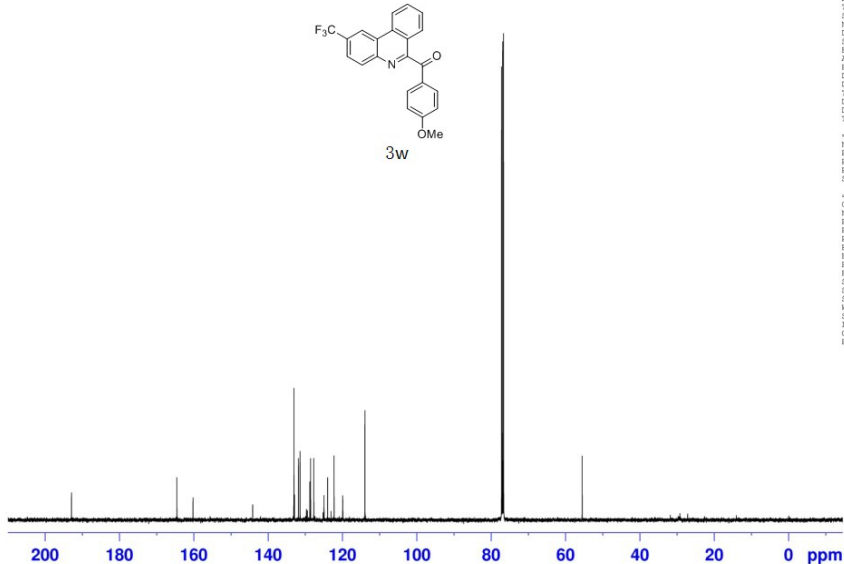
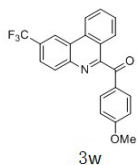


NAME 2013-12-11  
EXPNO 56  
PROCNO 1  
Date\_ 20131211  
Time 17.15  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 8  
DS 2  
SWH 8278.146 Hz  
FIDRES 0.126314 Hz  
AQ 3.9584243 sec  
RG 362  
DW 60.400 usec  
DE 6.50 usec  
TE 298.0 K  
D1 1.0000000 sec  
TDO 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 12.58 usec  
PL1 0.00 dB  
PL1W 10.87646866 W  
SFO1 400.1324710 MHz  
SI 32768  
SF 400.1300096 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

313051

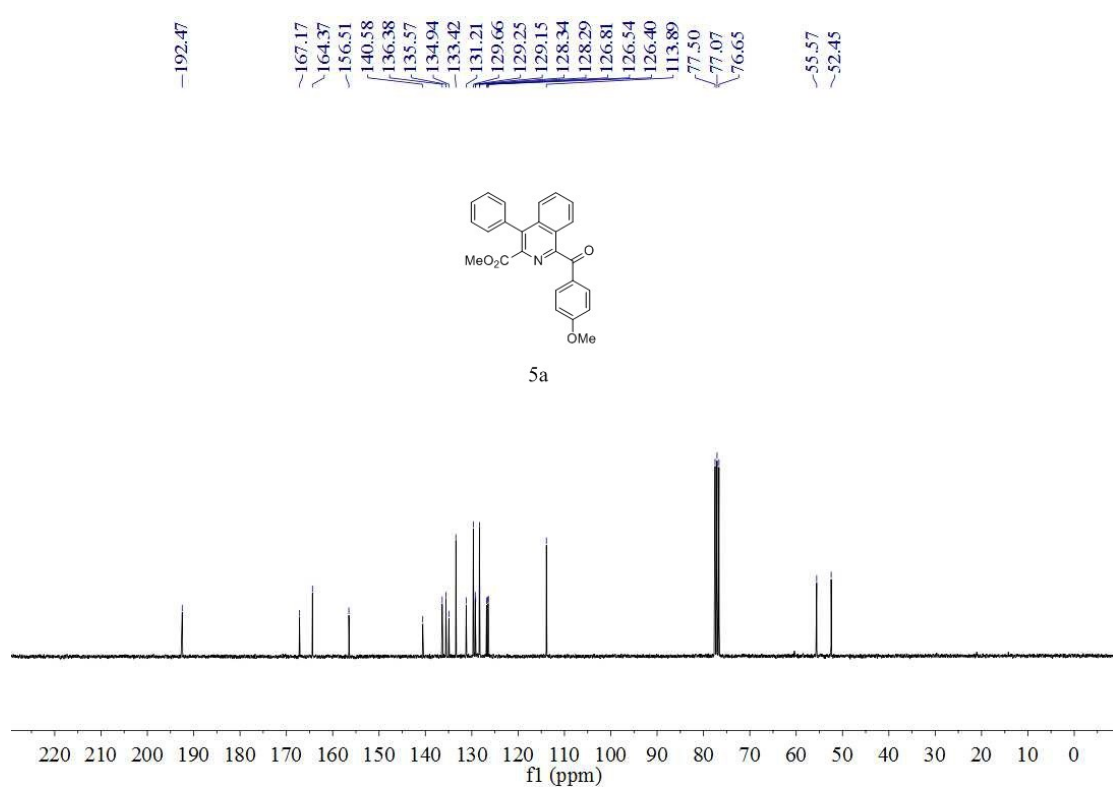
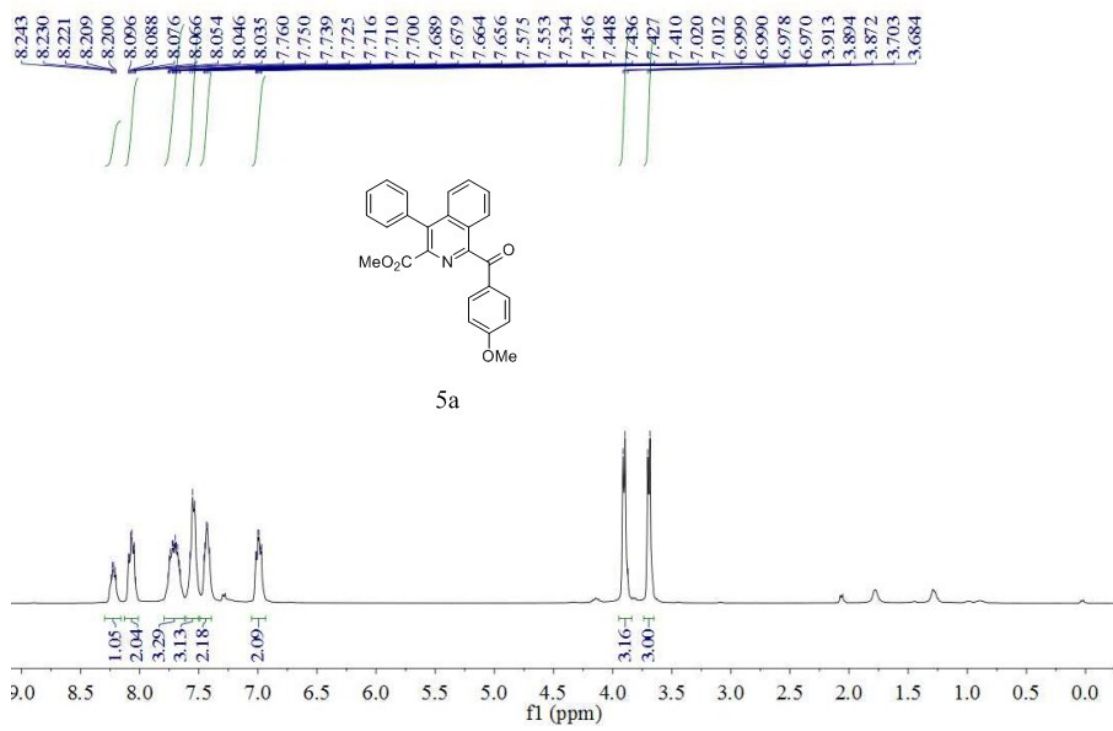
192.884  
164.586  
160.203  
144.201  
133.126  
132.958  
131.905  
131.491  
128.843  
128.752  
127.752  
125.027  
125.001  
124.051  
122.293  
122.283  
119.935  
119.925  
114.005  
77.230  
76.975  
76.721  
55.557

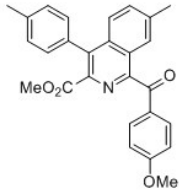
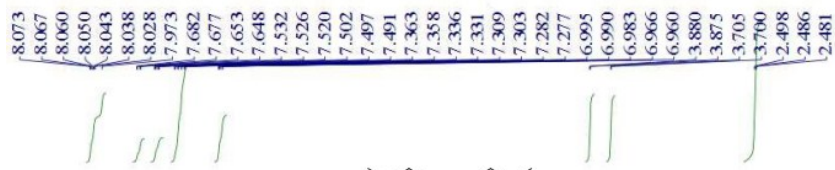


NAME 313051  
EXPNO 2  
PROCNO 1  
Date\_ 20131211  
Time 20.33  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 1024  
DS 4  
SWH 29761.904 Hz  
FIDRES 0.454131 Hz  
AQ 1.1010548 sec  
RG 203  
DW 16.800 usec  
DE 6.50 usec  
TE 297.9 K  
D1 2.0000000 sec  
D11 0.3300000 sec  
TDO 1

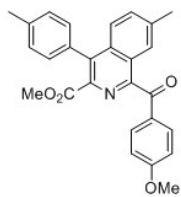
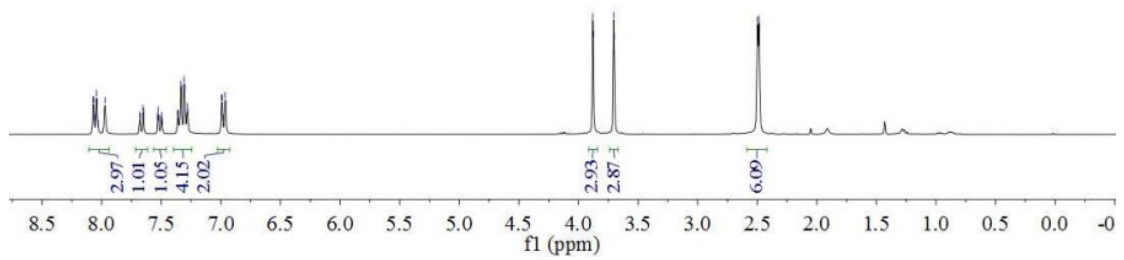
===== CHANNEL f1 =====  
NUC1 13C  
P1 13.84 usec  
PL1 2.50 dB  
PL1W 46.89624796 W  
SFO1 125.7703643 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 80.00 usec  
PL2 2.50 dB  
PL12 17.40 dB  
PL13 7.40 dB  
PL1W 13.02359581 W  
PL1W 0.42143336 W  
PL1W 0.42143336 W  
SFO2 500.1320096 MHz  
SI 32768  
SF 125.7577966 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

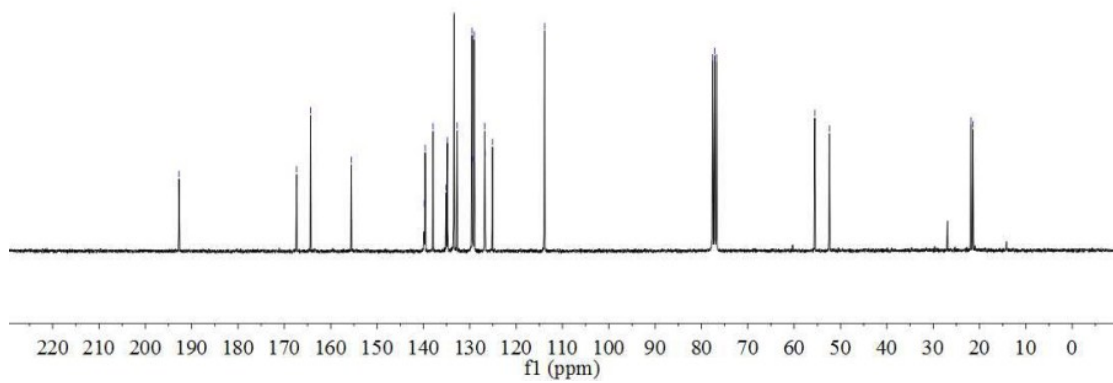


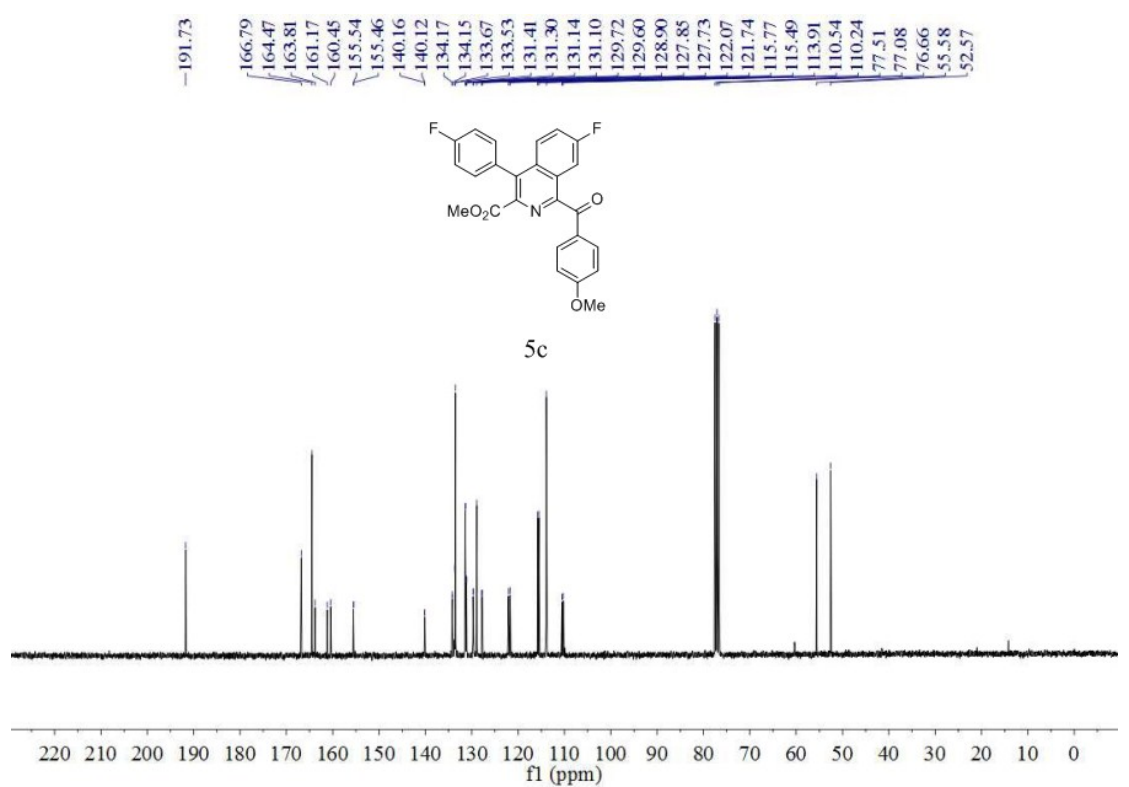
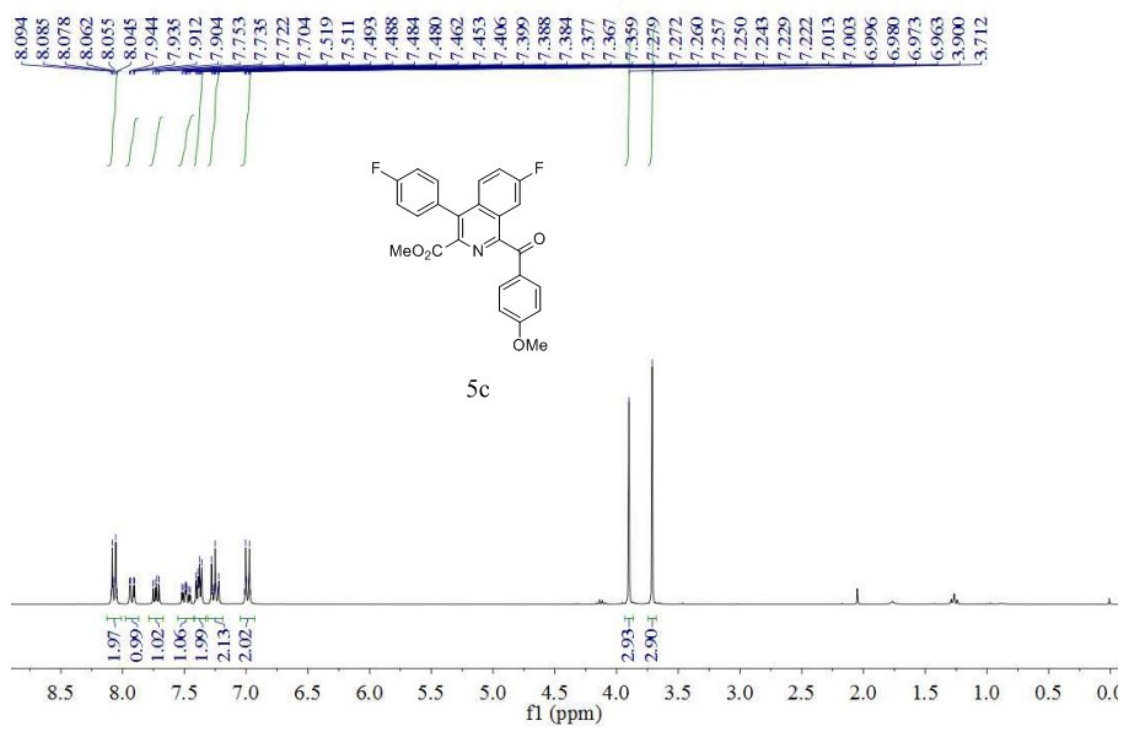


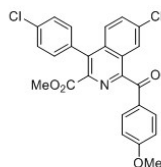
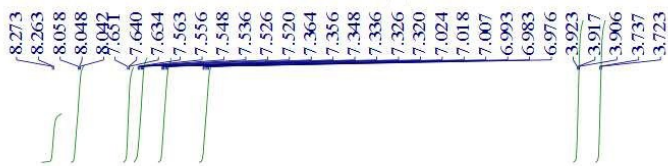
5b



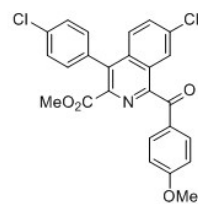
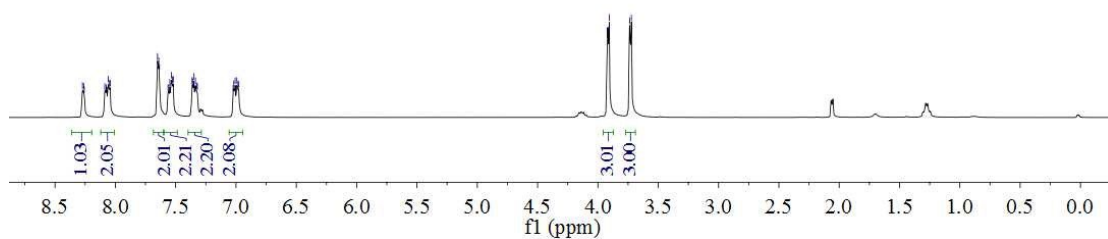
5b



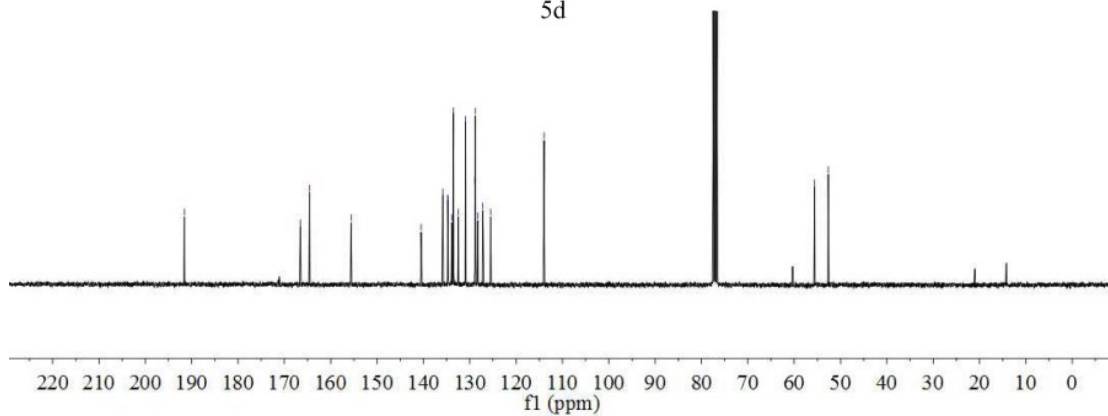


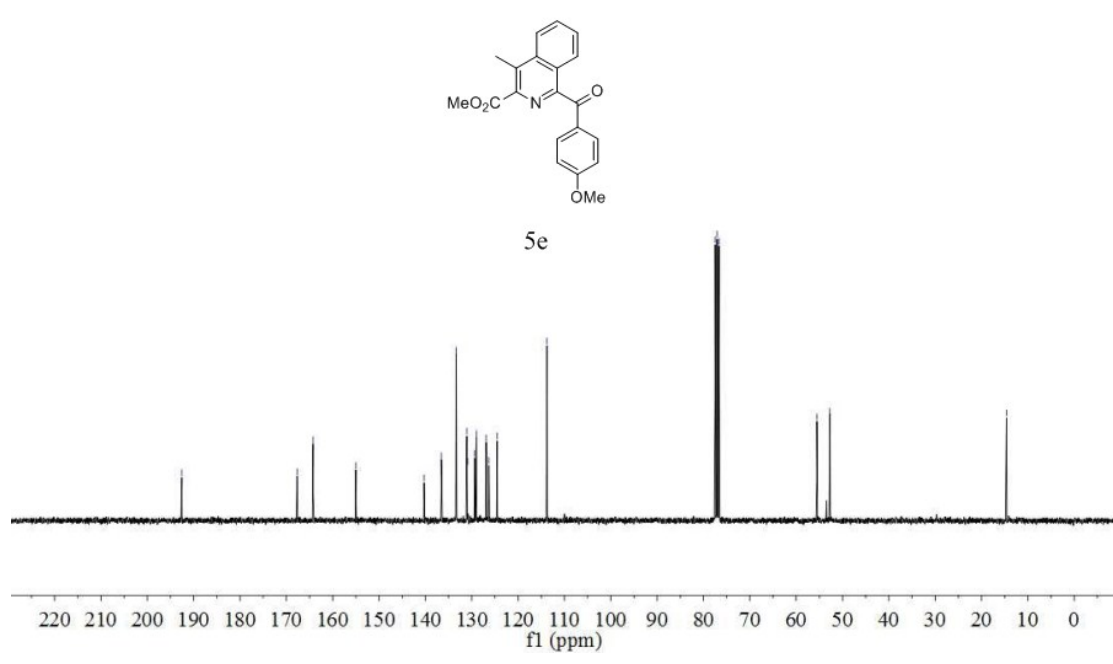
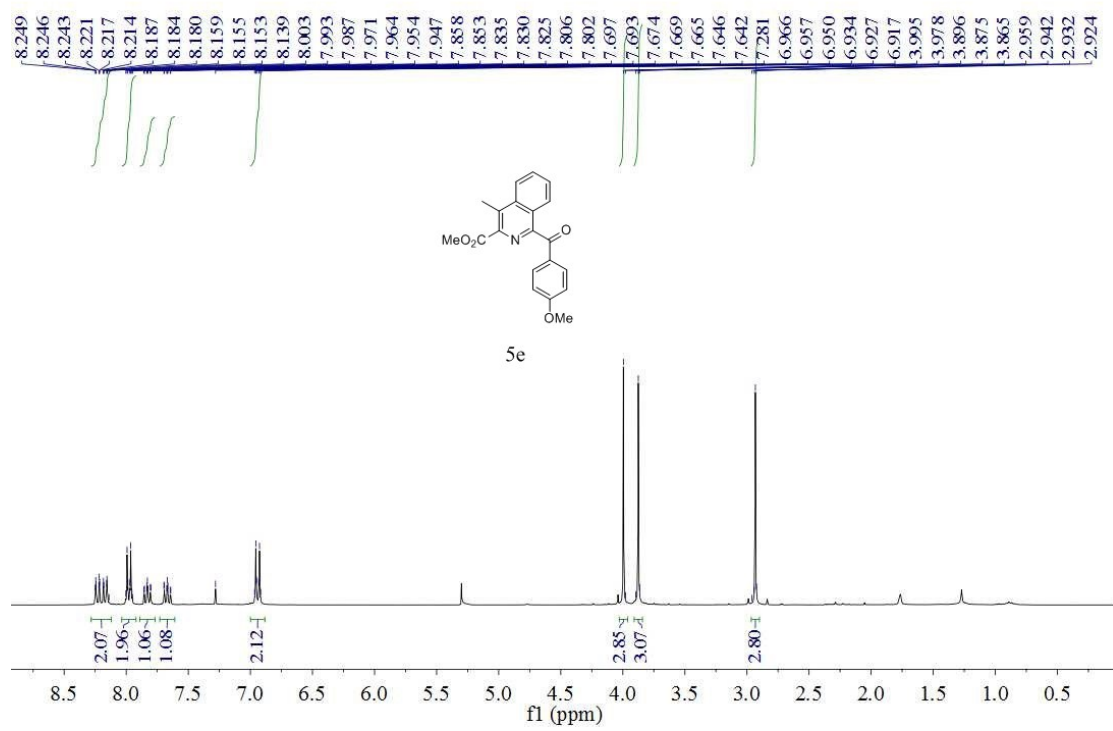


5d



5d



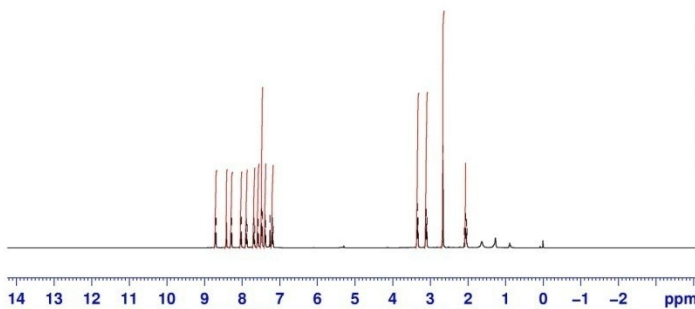
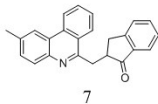


1496391

8.716  
8.696  
8.418  
8.299  
8.278  
8.043  
8.022  
7.905  
7.897  
7.857  
7.796  
7.668  
7.668  
7.592  
7.571  
7.507  
7.485  
7.465  
7.396  
7.377  
7.259  
7.214  
7.196  
7.177  
3.356  
3.339  
3.323  
3.124  
3.106  
3.087  
2.661  
2.065  
2.068  
2.060  
2.024



NAME 2013-12-12  
EXPNO 85  
PROCNO 1  
Date\_ 20131212  
Time 19.28  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 8  
DS 2  
SWH 8278.146 Hz  
FIDRES 0.126314 Hz  
AQ 3.9584243 sec  
RG 256  
DW 60.400 usec  
DE 6.50 usec  
TE 298.0 K  
D1 1.00000000 sec  
TDO 1



0.99  
1.00  
0.97  
0.97  
1.00  
1.02  
1.07  
2.04  
1.07  
1.05  
1.97  
1.96  
3.00  
1.08

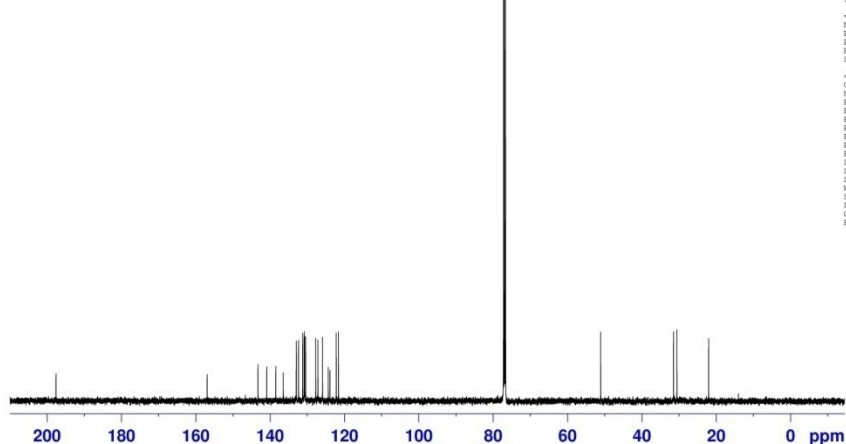
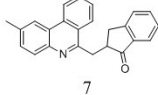
----- CHANNEL f1 -----  
NUC1 1H  
P1 12.58 usec  
PL1 0.00 dB  
PL1W 10.87646866 W  
SFO1 400.1324710 MHz  
SI 32768  
SF 400.1300094 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

1496391

197.587  
156.945  
156.785  
140.970  
138.509  
136.512  
133.042  
133.003  
131.292  
131.292  
130.895  
130.773  
130.482  
127.754  
125.989  
124.409  
123.940  
122.249  
121.687  
77.240  
76.986  
76.732  
51.118  
31.508  
30.591  
22.078



NAME 1496391  
EXPNO 1  
PROCNO 1  
Date\_ 20131218  
Time 11.10  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 884  
DS 4  
SWH 29761.904 Hz  
FIDRES 0.454131 Hz  
AQ 1.010583 sec  
RG 203  
DW 16.800 usec  
DE 6.50 usec  
TE 298.0 K  
D1 2.00000000 sec  
D11 0.33000000 sec  
TDO 1



----- CHANNEL f1 -----  
NUC1 13C  
P1 13.84 usec  
PL1 2.50 dB  
PL1W 46.89624786 W  
SFO1 125.7703643 MHz  
----- CHANNEL f2 -----  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 80.00 usec  
PL2 2.50 dB  
PL12 17.40 dB  
PL13 7.40 dB  
PL1W 13.0239881 W  
PL1W 0.42143336 W  
PL1W 0.42143336 W  
SFO2 500.1320005 MHz  
SI 32768  
SF 125.7577966 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40