

# **Ru(II)-Catalyzed Amidation of 2-Arylpyridines with Isocyanates via C-H Activation**

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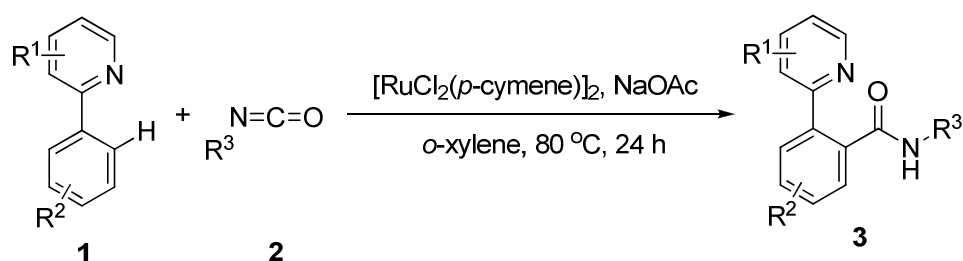
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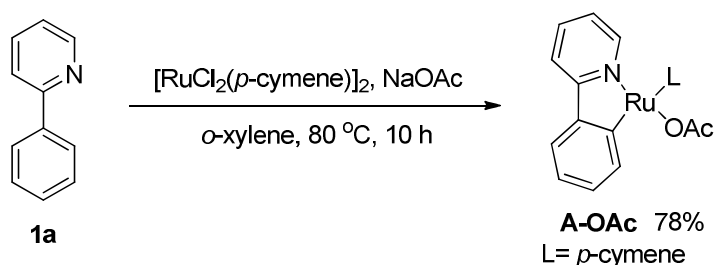
**General:** All reactions were conducted under a nitrogen atmosphere on a dual-manifold Schlenk line unless otherwise mentioned and in oven-dried glass wares. All solvents were dried according to known methods and distilled prior to use.<sup>1</sup> Starting materials 2-arylpyridines **1b-q** were synthesized according to the literature procedures.<sup>2</sup> Other reagents were commercially available and used as purchased.

**General Procedure for the Ru(II)-Catalyzed Amidation of 2-Arylpyridines with Isocyanates via C-H Activation.**



A sealed tube containing  $[\text{RuCl}_2(p\text{-cymene})]_2$  (5.0 mol %) and NaOAc (30 mol %) was evacuated and purged with nitrogen gas three times. Then, *o*-xylene (2 mL), 2-arylpyridines **1** (1.00 mmol), isocyanates **2** (1.8 mmol) were sequentially added to the system via syringe under a nitrogen atmosphere and the reaction mixture was allowed to stir at 80 °C for 24 h. When the reaction was complete, the mixture was cooled and diluted with  $\text{CH}_2\text{Cl}_2$  (10 mL). The mixture was filtered through a Celite pad and the Celite pad was washed with  $\text{CH}_2\text{Cl}_2$  (50 mL). The filtrate was concentrated and the residue was purified by column chromatography (silica gel, hexane–EtOAc) to give the corresponding pure amidation product **3**.

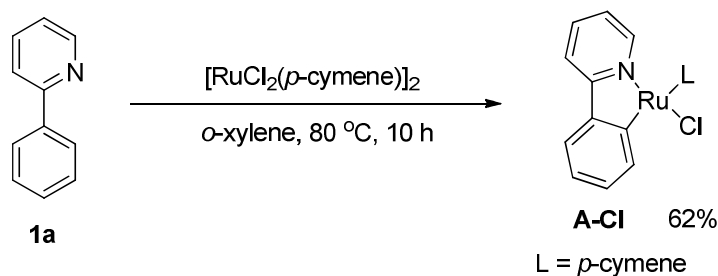
**Synthesis of Complex A-OAc**



A seal tube initially fitted with a septum containing  $[\text{RuCl}_2(p\text{-cymene})]_2$  (50 mg, 0.081 mmol) and NaOAc (40 mg, 0.490 mmol) was evacuated and purged with

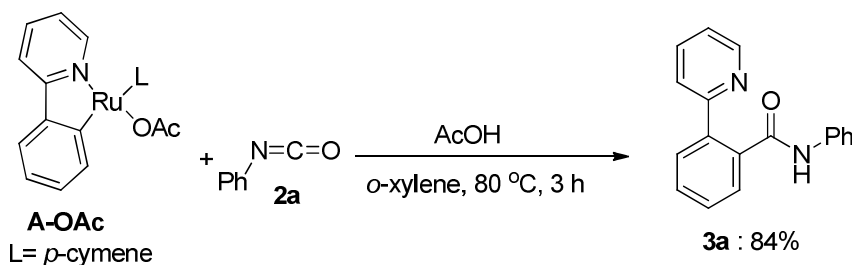
nitrogen gas three times. Then, *o*-xylene (2 mL) and 2-phenylpyridine (25 mg, 0.162 mmol) were sequentially added to the system via syringe under a nitrogen atmosphere and the reaction mixture was stirred at 80 °C for 10 h. After cooling to ambient temperature, the mixture was filtered through a short Celite pad and the Celite pad was washed with DCM several times. The filtrate was concentrated by vacuum and the residue was carefully washed with Hexane and redissolved in EtOAc/Hexane. The combined filtrate was concentrated in vacuum to afford the desired product **A-OAc** in good solid.

#### Synthesis of Complex A-Cl



A seal tube initially fitted with a septum containing  $[\text{RuCl}_2(\textit{p}\text{-cymene})]_2$  (50 mg, 0.081 mmol) was evacuated and purged with nitrogen gas three times. Then, *o*-xylene (2 mL) and 2-phenylpyridine (25 mg, 0.162 mmol) were sequentially added to the system via syringe under a nitrogen atmosphere and the reaction mixture was stirred at 80 °C for 10 h. After cooling to ambient temperature, the mixture was filtered through a short Celite pad and the Celite pad was washed with DCM several times. The filtrate was concentrated by vacuum and purified by column chromatography (neutral alumina ( $\text{Al}_2\text{O}_3$ )) and eluted with EtOAc (100 %). The solvent was removed under vacuum to afford **A-Cl** in orange solid.

#### Reaction of Complex A-OAc with Phenylisocyanate (2a).

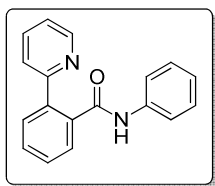


A seal tube initially fitted with a septum containing complex **A-OAc** (50 mg, 0.111 mmol) was evacuated and purged with nitrogen gas three times. Phenylisocyanate (**2a**) (23 mg, 0.200 mmol), HOAc (0.111 mmol) and *o*-xylene (2 mL) was added to the

system and the reaction mixture was stirred at 80 °C for 3 h. After cooling to ambient temperature, the mixture was filtered through a short Celite pad and the Celite pad was washed with DCM several times. The filtrate was concentrated and the residue was purified by column chromatography (silica gel, hexane–EtOAc) to give the corresponding pure amidation product **3a** in 84% yield.

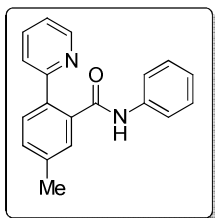
The spectral data and a copy of  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra of all compounds are listed below.

***N*-Phenyl-2-(pyridin-2-yl)benzamide (3a)**



Brown solid; m.p. 104-106 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.83 (s, NH), 8.59 (d,  $J = 4$  Hz, 1 H), 7.74 (t,  $J = 7.2$  Hz, 2 H), 7.52-7.41 (m, 6 H), 7.27-7.21 (m, 3 H), 7.04 (t,  $J = 7.2$  Hz, 1 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  167.4 (CO), 158.0 (C), 148.2 (CH), 138.1 (C), 137.7 (C), 137.4 (CH), 136.3 (C), 130.3 (CH), 130.2 (CH), 129.1 (CH), 128.9 (CH), 128.8 (2 CH), 124.3 (CH), 124.1 (CH), 122.7(CH), 119.9 (2 CH); HRMS (ESI/[2M+Na] $^+$ ) calcd for  $\text{C}_{26}\text{H}_{18}\text{N}_2$  571.2110 found 571.2098; IR (KBr): 3316, 2923, 1658, 1316 and 763  $\text{cm}^{-1}$ .

**5-Methyl-*N*-phenyl-2-(pyridin-2-yl)benzamide (3b)**

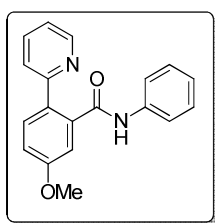


Brown solid; m.p. 154-156 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  9.24 (s, NH), 8.50 (d,  $J = 4.8$  Hz, 1 H), 7.66 (td,  $J = 1.6$  Hz,  $J = 8$  Hz, 1 H), 7.48-7.41 (m, 4 H), 7.30 (d,  $J =$



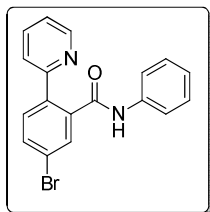
8 Hz, 1 H), 7.23-7.15 (m, 4 H), 7.01 (t,  $J = 7.6$  Hz, 1 H), 2.31 (s, 3 H);  **$^{13}\text{C}$  NMR** (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  167.7 (CO), 158.2 (C), 148.5 (CH), 138.5 (C), 138.3 (C), 136.8 (CH), 135.9 (C), 135.3 (C), 130.8 (CH), 130.0 (CH), 129.7 (CH), 128.6 (2 CH), 124.0 (CH), 123.9 (CH), 122.2 (CH), 119.9 (2 CH), 20.9 ( $\text{CH}_3$ ); **HRMS** (ESI/[2M+Na] $^+$ ) calcd for  $\text{C}_{38}\text{H}_{32}\text{N}_4\text{NaO}_2$  599.2423 found 599.2415; IR (KBr): 3255, 2923, 1658, 1434 and 817  $\text{cm}^{-1}$

### 5-Methoxy-*N*-phenyl-2-(pyridin-2-yl)benzamide (3c)



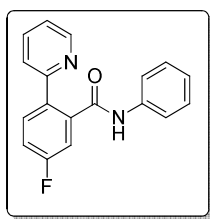
Yellow solid; m.p. 149-151  $^{\circ}\text{C}$ ;  **$^1\text{H}$  NMR** (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  9.09 (s, NH), 8.55 (d,  $J = 4.4$  Hz, 1 H), 7.69 (td,  $J = 1.6$  Hz,  $J = 7.6$  Hz, 1 H), 7.46-7.39 (m, 4 H), 7.27-7.17 (m, 4 H), 7.06-6.98 (m, 2 H), 3.81 (s, 3 H);  **$^{13}\text{C}$  NMR** (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  167.3 (CO), 159.7 (C), 158.1 (C), 148.5 (CH), 138.1 (C), 137.4 (C), 136.9 (CH), 131.7 (CH), 130.6 (C), 128.8 (2 CH), 124.2 (CH), 124.1 (CH), 122.1 (CH), 119.9 (2 CH), 116.7 (CH), 113.9 (CH), 55.4 ( $\text{CH}_3$ ); **HRMS** (ESI/[2M+Na] $^+$ ) calcd for  $\text{C}_{38}\text{H}_{32}\text{N}_4\text{NaO}_4$  631.2321 found 631.2339; IR (KBr): 3264, 2923, 1673, 1542 and 817  $\text{cm}^{-1}$

### 5-Bromo-*N*-phenyl-2-(pyridin-2-yl)benzamide (3d)



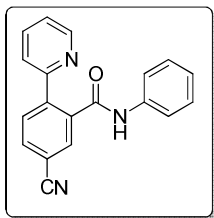
Brown solid; m.p. 130-132 °C; **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>): δ 9.52 (s, NH), 8.55 (d, *J* = 4.4 Hz, 1 H), 7.75-7.71 (m, 2 H), 7.52-7.46 (m, 2 H), 7.38 (d, *J* = 8 Hz, 2 H), 7.29 (d, *J* = 8 Hz, 1 H), 7.24 (t, *J* = 8 Hz, 1 H), 7.16 (t, *J* = 7.6 Hz, 2 H), 7.00 (t, *J* = 7.6 Hz, 1 H); **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>): δ 166.1 (CO), 157.2 (C), 148.6 (CH), 138.0 (C), 137.8 (C), 137.2 (CH), 137.1 (C), 133.2 (CH), 132.1 (CH), 131.7 (CH), 128.7 (2 CH), 124.3 (CH), 123.9 (CH), 122.8 (CH), 122.7 (C), 120.1 (2 CH); **HRMS** (ESI/[2M+Na]<sup>+</sup>) calcd for C<sub>36</sub>H<sub>26</sub>Br<sub>2</sub>N<sub>4</sub>NaO<sub>2</sub> 727.0320 found 729.0324; IR (KBr): 3235, 2925, 1653, 1473 and 717 cm<sup>-1</sup>

**5-Fluoro-*N*-phenyl-2-(pyridin-2-yl)benzamide (3e)**



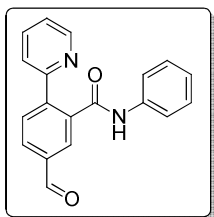
Yellow solid; m.p. 128-130 °C; **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>): δ 9.35 (s, NH), 8.52 (d, *J* = 4.8 Hz, 1 H), 7.71 (td, *J* = 1.6 Hz, *J* = 8 Hz, 1 H), 7.47-7.37 (m, 5 H), 7.24-7.18 (m, 3 H), 7.11 (td, *J* = 2.8 Hz, *J* = 8 Hz, 1 H), 7.02 (t, *J* = 7.6 Hz, 1 H); **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>): δ 166.1 (CO), 162.4 (d, *J*<sub>C-F</sub> = 248 Hz, C), 157.3 (C), 148.6 (CH), 138.1 (d, *J*<sub>C-F</sub> = 7.3 Hz, C), 137.9 (C), 137.1 (CH), 134.3 (d, *J*<sub>C-F</sub> = 3.6 Hz, C), 132.2 (d, *J*<sub>C-F</sub> = 8 Hz, CH), 128.7 (2 CH), 124.3 (CH), 124.1 (CH), 122.6 (CH), 120.1 (2 CH), 117.2 (d, *J*<sub>C-F</sub> = 21.3 Hz, CH), 116.3 (d, *J*<sub>C-F</sub> = 22.7 Hz, CH); **HRMS** (ESI/[2M+Na]<sup>+</sup>) calcd for C<sub>36</sub>H<sub>26</sub>F<sub>2</sub>N<sub>4</sub>NaO<sub>2</sub> 607.1922 found 607.1911; IR (KBr): 3289, 2923, 1658, 1542 and 640 cm<sup>-1</sup>

**5-Cyano-*N*-phenyl-2-(pyridin-2-yl)benzamide (3f)**



Brown solid; m.p. 165-167 °C; **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>): δ 9.05 (s, NH), 8.66 (d, *J* = 4 Hz, 1 H), 8.02 (s, 1 H), 7.81 (t, *J* = 7.6 Hz, 1 H), 7.75 (d, *J* = 8 Hz, 1 H), 7.62 (d, *J* = 8 Hz, 1 H), 7.55 (d, *J* = 7.6 Hz, 1 H), 7.40 (d, *J* = 7.6 Hz, 2 H), 7.35 (t, *J* = 6.8 Hz, 1 H), 7.25 (t, *J* = 8 Hz, 2 H), 7.08 (t, *J* = 7.2 Hz, 1 H); **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>): δ 165.3 (CO), 156.4 (C), 149.0 (CH), 142.2 (C), 137.6 (C), 137.5 (CH), 137.4 (C), 133.3 (CH), 133.0 (CH), 131.0 (CH), 128.9 (2 CH), 124.7 (CH), 124.1 (CH), 123.6 (CH), 120.0 (2 CH), 117.6 (C), 112.7 (C); **HRMS** (ESI/[2M+Na]<sup>+</sup>) calcd for C<sub>18</sub>H<sub>14</sub>N<sub>2</sub>O<sub>2</sub> 621.2015 found 621.2014; IR (KBr): 3247, 2923, 2229, 1658 and 1326 cm<sup>-1</sup>

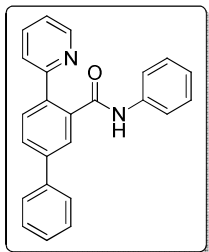
#### 5-Formyl-*N*-phenyl-2-(pyridin-2-yl)benzamide (3g)



Yellow solid; m.p. 148-150 °C; **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>): δ 10.01 (s, CHO), 8.98 (s, NH), 8.63 (d, *J* = 4.4 Hz, 1 H), 8.21 (s, 1 H), 7.97 (dd, *J* = 1.6 Hz, *J* = 8 Hz, 1 H), 7.78 (t, *J* = 7.6 Hz, 1 H), 7.62 (d, *J* = 8 Hz, 1 H), 7.54 (d, *J* = 7.6 Hz, 1 H), 7.41 (d, *J* = 8 Hz, 2 H), 7.31 (t, *J* = 8 Hz, 1 H), 7.24 (t, *J* = 8 Hz, 2 H), 7.05 (t, *J* = 7.6 Hz, 1 H); **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>): δ 191.0 (CO), 166.3 (CO), 157.0 (C), 148.9 (CH), 143.5 (C), 137.8 (C), 137.3 (CH), 136.0 (C), 131.2 (CH), 131.1 (CH), 130.2 (CH), 128.9 (3 CH), 124.5 (CH), 124.2 (CH), 123.3 (CH), 120.0 (2 CH); **HRMS**

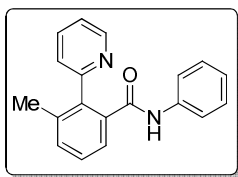
(ESI/[2M+Na]<sup>+</sup>) calcd for C<sub>38</sub>H<sub>28</sub>N<sub>4</sub>NaO<sub>4</sub> 627.2008 found 627.2001; IR (KBr): 3252, 2923, 1697, 1658 and 1596 cm<sup>-1</sup>

***N*-Phenyl-4-(pyridin-2-yl)-[1,1'-biphenyl]-3-carboxamide (3h)**



Yellow solid; m.p. 157-159 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 9.67 (s, NH), 8.63 (d, *J* = 5.2 Hz, 1 H), 7.84 (s, 1 H), 7.74 (t, *J* = 7.6 Hz, 1 H), 7.57 (d, *J* = 8 Hz, 1 H), 7.51 (d, *J* = 8 Hz, 1 H), 7.46 (d, *J* = 8.8 Hz, 2 H), 7.36-7.32 (m, 6 H), 7.26 (t, *J* = 8 Hz, 1 H), 7.17 (t, *J* = 7.2 Hz, 2 H), 6.99 (t, *J* = 7.2 Hz, 1 H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 167.7 (CO), 158.0 (C), 148.6 (CH), 141.2 (C), 139.4 (C), 138.4 (C), 137.1 (CH), 137.0 (C), 136.7 (C), 130.7 (CH), 128.8 (2 CH), 128.7 (2 CH), 128.6 (CH), 127.9 (CH), 127.8 (CH), 126.8 (2 CH), 124.1 (CH), 124.0 (CH), 122.5 (CH), 120.0 (2 CH); HRMS (ESI/[2M+Na]<sup>+</sup>) calcd for C<sub>48</sub>H<sub>36</sub>N<sub>4</sub>NaO<sub>2</sub> 723.2736 found 723.2749; IR (KBr): 3214, 2923, 1658, 1542 and 748 cm<sup>-1</sup>

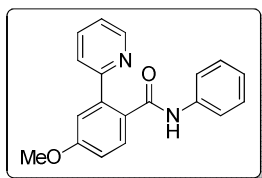
**3-Methyl-*N*-phenyl-2-(pyridin-2-yl)benzamide (3i)**



White solid; m.p. 122-124 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 8.68 (d, *J* = 4.4 Hz, 1 H), 7.91 (s, NH), 7.70 (td, *J* = 1.6 Hz, *J* = 7.6 Hz, 1 H), 7.59 (t, *J* = 4.8 Hz, 1H), 7.36-7.21 (m, 6 H), 7.17 (t, *J* = 7.6 Hz, 2 H), 6.99 (t, *J* = 7.2 Hz, 1 H), 2.10 (s, 3 H);

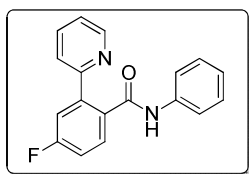
**$^{13}\text{C}$  NMR** (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  167.3 (CO), 158.4 (C), 149.1 (CH), 137.8 (C), 137.5 (C), 137.0 (CH), 136.7 (C), 136.6 (C), 132.2 (CH), 128.7 (2 CH), 128.4 (CH), 126.1 (CH), 125.0 (CH), 124.0 (CH), 122.5 (CH), 119.5 (2 CH), 20.0 ( $\text{CH}_3$ ); **HRMS** (ESI/[2M+Na] $^+$ ) calcd for  $\text{C}_{38}\text{H}_{32}\text{N}_4\text{NaO}_2$  599.2423 found 599.2407; IR (KBr): 3287, 2915, 1658, 1535 and  $686\text{ cm}^{-1}$

#### 4-Methoxy-*N*-phenyl-2-(pyridin-2-yl)benzamide (3j)



Yellow solid; m.p. 152-154 °C;  **$^1\text{H}$  NMR** (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  9.38 (s, NH), 8.47 (d,  $J = 4.4\text{ Hz}$ , 1 H), 7.68 (t,  $J = 7.6\text{ Hz}$ , 1 H), 7.62 (d,  $J = 8.4\text{ Hz}$ , 1 H), 7.45 (d,  $J = 7.6\text{ Hz}$ , 1 H), 7.38 (d,  $J = 7.6\text{ Hz}$ , 2 H), 7.20-7.15 (m, 3 H), 6.98 (t,  $J = 7.6\text{ Hz}$ , 1 H), 6.87-6.81 (m, 2 H), 3.77 (s, 3 H);  **$^{13}\text{C}$  NMR** (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  167.2 (CO), 160.6 (2 C), 158.2 (C), 148.4 (CH), 140.0 (C), 138.4 (C), 136.8 (CH), 131.0 (CH), 128.6 (3 CH), 124.2 (CH), 123.8 (CH), 122.5 (CH), 120.0 (CH), 115.3 (CH), 113.8 (CH), 55.2 ( $\text{CH}_3$ ); **HRMS** (ESI/[2M+Na] $^+$ ) calcd for  $\text{C}_{38}\text{H}_{32}\text{N}_4\text{NaO}_4$  631.2321 found 631.2302; IR (KBr): 3287, 2923, 1643, 1535 and  $601\text{ cm}^{-1}$

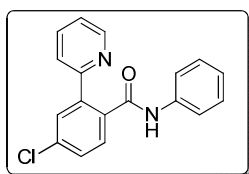
#### 4-Fluoro-*N*-phenyl-2-(pyridin-2-yl)benzamide (3k)<sup>3</sup>



White solid; m.p. 142-144 °C;  **$^1\text{H}$  NMR** (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  9.11 (s, NH), 8.56 (d,  $J = 4.4\text{ Hz}$ , 1 H), 7.75-7.68 (m, 2 H), 7.48 (d,  $J = 7.6\text{ Hz}$ , 1 H), 7.33 (d,  $J = 8\text{ Hz}$ , 2 H),

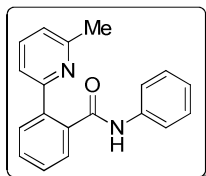
7.27-7.12 (m, 4 H), 7.08-7.00 (m, 2 H); **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>): δ 166.6 (CO), 163.2 (d,  $J_{C-F}$  = 249.6 Hz, C), 157.0 (C), 148.8 (CH), 140.7 (d,  $J_{C-F}$  = 7.3 Hz, C), 138.0 (C), 137.1 (CH), 132.4 (C), 131.6 (d,  $J_{C-F}$  = 8.7 Hz, CH), 128.8 (2 CH), 124.2 (CH), 124.1 (CH), 123.0 (CH), 119.9 (2 CH), 117.1 (d,  $J_{C-F}$  = 22.7 Hz, CH), 115.5 (d,  $J_{C-F}$  = 21.3 Hz, CH); **HRMS** (ESI/[2M+Na]<sup>+</sup>) calcd for C<sub>36</sub>H<sub>26</sub>F<sub>2</sub>N<sub>4</sub>NaO<sub>2</sub> 607.1922 found 607.1902; IR (KBr): 3213, 2923, 1658, 1434 and 663 cm<sup>-1</sup>

#### 4-Chloro-*N*-phenyl-2-(pyridin-2-yl)benzamide (3l)



Yellow solid; m.p. 144-146 °C; **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>): δ 8.93 (s, NH), 8.59 (d,  $J$  = 4.4 Hz, 1 H), 7.76-7.69 (m, 2 H), 7.50-7.46 (m, 2 H), 7.42-7.38 (m, 3 H), 7.28-7.20 (m, 3 H), 7.04 (t,  $J$  = 7.2 Hz, 1 H); **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>): δ 166.5 (CO), 157.0 (C), 148.8 (CH), 139.7 (C), 137.9 (C), 137.2 (CH), 136.3 (C), 134.6 (C), 130.9 (CH), 130.2 (CH), 128.9 (2 CH), 128.8 (CH), 124.4 (CH), 124.2 (CH), 123.1 (CH), 119.9 (2 CH); **HRMS** (ESI/[2M+Na]<sup>+</sup>) calcd for C<sub>36</sub>H<sub>26</sub>Cl<sub>2</sub>N<sub>4</sub>NaO<sub>2</sub> 639.1331 found 639.1341; IR (KBr): 3214, 2924, 1658, 1496 and 701 cm<sup>-1</sup>

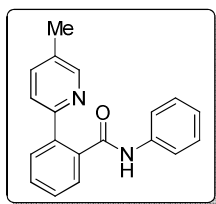
#### 2-(6-Methylpyridin-2-yl)-*N*-phenylbenzamide (3m)



Yellow solid; m.p. 103-105 °C; **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>): δ 8.63 (s, NH), 7.71 (d,  $J$  = 7.6 Hz, 1 H), 7.55 (t,  $J$  = 7.6 Hz, 1 H), 7.49 (t,  $J$  = 7.6 Hz, 2 H), 7.44-7.38 (m, 3

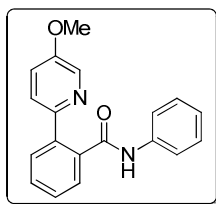
H), 7.24 (t,  $J = 8$  Hz, 3 H), 7.06-7.03 (m, 2 H), 2.49 (s, 3 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  167.6 (CO), 157.4 (C), 157.3 (C), 138.3 (C), 137.9 (C), 136.8 (CH), 136.0 (C), 129.9 (CH), 129.8 (CH), 128.8 (CH), 128.5 (2 CH), 128.2 (CH), 123.8 (CH), 121.8 (CH), 120.6 (CH), 119.7 (2 CH), 24.2 ( $\text{CH}_3$ ); **HRMS** (ESI/[2M+Na] $^+$ ) calcd for  $\text{C}_{38}\text{H}_{32}\text{N}_4\text{NaO}_2$  599.2423 found 599.2414; IR (KBr): 3286, 3046, 2923, 1658 and  $1442\text{ cm}^{-1}$

### 2-(5-Methylpyridin-2-yl)-*N*-phenylbenzamide (3n)



Yellow solid; m.p. 192-194 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  9.05 (s, NH), 8.40 (s, 1 H), 7.73 (d,  $J = 7.2$  Hz, 1 H), 7.51 (d,  $J = 8.4$  Hz, 1 H), 7.45-7.37 (m, 6 H), 7.24 (t,  $J = 7.2$  Hz, 2 H), 7.03 (t,  $J = 7.2$  Hz, 1 H), 2.31 (s, 3 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  167.7 (CO), 155.5 (C), 148.9 (CH), 138.3 (C), 138.1 (C), 137.5 (CH), 136.1 (C), 132.1 (C), 130.2 (2 CH), 129.2 (CH), 128.7 (2 CH), 128.4 (CH), 124.0 (CH), 123.6 (CH), 119.9 (2 CH), 18.1 ( $\text{CH}_3$ ); **HRMS** (ESI/[2M+Na] $^+$ ) calcd for  $\text{C}_{38}\text{H}_{32}\text{N}_4\text{NaO}_2$  599.2423 found 599.2416; IR (KBr): 3258, 2915, 1658, 1542 and  $717\text{ cm}^{-1}$

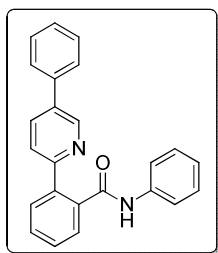
### 2-(5-Methoxypyridin-2-yl)-*N*-phenylbenzamide (3o)



Yellow solid; m.p. 163-165 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.47 (s, NH), 8.39 (d,  $J = 2.8$  Hz, 1 H), 7.78 (d,  $J = 7.2$  Hz, 1 H), 7.51-7.43 (m, 6 H), 7.29-7.21 (m, 3 H),

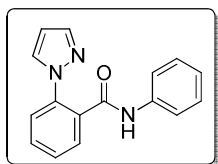
7.06 (t,  $J = 7.2$  Hz, 1 H), 3.84 (s, 3 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  167.7 (CO), 155.0 (C), 150.4 (C), 138.2 (C), 137.8 (C), 136.5 (CH), 136.2 (C), 130.3 (CH), 130.2 (CH), 129.2 (CH), 128.9 (2 CH), 128.3 (CH), 124.5 (CH), 124.1 (CH), 121.2 (CH), 119.8 (2 CH), 55.6 ( $\text{CH}_3$ ); HRMS (ESI/[2M+Na] $^+$ ) calcd for  $\text{C}_{38}\text{H}_{32}\text{N}_4\text{NaO}_4$  631.2321 found 631.2310; IR (KBr): 3264, 2923, 1658, 1496 and  $794\text{ cm}^{-1}$

***N*-Phenyl-2-(5-phenylpyridin-2-yl)benzamide (3p)**



Yellow solid; m.p. 198-200 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.83 (sd,  $J = 2$  Hz, 1 H), 8.62 (s, NH), 7.91 (dd,  $J = 2.4$  Hz,  $J = 8$  Hz, 1 H), 7.79 (d,  $J = 7.2$  Hz, 1 H), 7.60-7.51 (m, 4 H), 7.50-7.37 (m, 7 H), 7.26 (t,  $J = 7.6$  Hz, 2 H), 7.05 (t,  $J = 7.6$  Hz, 1 H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  167.7 (CO), 156.8 (C), 147.1 (CH), 138.2 (C), 137.9 (C), 137.1 (C), 136.3 (C), 135.4 (C), 135.2 (CH), 130.3 (CH), 130.2 (CH), 129.2 (CH), 129.0 (2 CH), 128.8 (2 CH), 128.7 (CH), 128.2 (CH), 127.0 (2 CH), 124.2 (CH), 123.9 (CH), 119.9 (2 CH); HRMS (ESI/[2M+Na] $^+$ ) calcd for  $\text{C}_{48}\text{H}_{36}\text{N}_4\text{NaO}_2$  723.2736 found 723.2750; IR (KBr): 3215, 2923, 1658, 1542 and  $817\text{ cm}^{-1}$

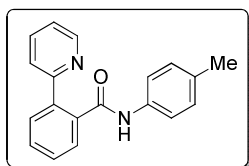
***N*-Phenyl-2-(1H-pyrazol-1-yl)benzamide (3q)**





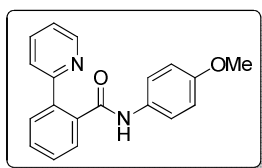
White solid; m.p. 158-160 °C;  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.66 (s, NH), 7.83 (dd,  $J = 2$  Hz,  $J = 7.2$  Hz, 1 H), 7.73 (s, 1 H), 7.68 (s, 1 H), 7.52-7.34 (m, 5 H), 7.24 (t,  $J = 8$  Hz, 2 H), 7.05 (t,  $J = 7.2$  Hz, 1 H), 6.43 (s, 1 H);  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  164.5 (CO), 141.2 (CH), 137.8 (C), 137.1 (C), 132.8 (C), 131.7 (CH), 131.2 (CH), 130.4 (CH), 128.9 (CH), 128.7 (2 CH), 126.5 (CH), 124.4 (CH), 120.0 (2 CH), 107.7 (CH); **HRMS** (ESI/[2M+Na] $^+$ ) calcd for  $\text{C}_{32}\text{H}_{26}\text{N}_6\text{NaO}_2$  549.2015 found 549.2008; IR (KBr): 3256, 2923, 1658, 1326 and 779  $\text{cm}^{-1}$

**2-(Pyridin-2-yl)-*N*-(*p*-tolyl)benzamide (3r)**



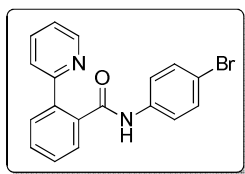
Yellow solid; m.p. 122-124 °C;  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.61 (d,  $J = 4.8$  Hz, 1 H), 8.51 (s, NH), 7.77 (d,  $J = 7.2$  Hz, 1 H), 7.71 (td,  $J = 1.6$  Hz,  $J = 8$  Hz, 1 H), 7.51-7.45 (m, 4 H), 7.28 (d,  $J = 8$  Hz, 2 H), 7.24 (t,  $J = 8$  Hz, 1 H), 7.05 (d,  $J = 8$  Hz, 2 H), 2.26 (s, 3 H);  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  167.4 (CO), 158.2 (C), 148.6 (CH), 138.1 (C), 136.8 (CH), 136.3 (C), 135.6 (C), 133.6 (C), 130.2 (CH), 130.1 (CH), 129.2 (2 CH), 129.1 (CH), 128.6 (CH), 124.1 (CH), 122.4 (CH), 119.9 (2 CH), 20.7 ( $\text{CH}_3$ ); **HRMS** (ESI/[2M+Na] $^+$ ) calcd for  $\text{C}_{38}\text{H}_{32}\text{N}_4\text{NaO}_2$  599.2423 found 599.2418; IR (KBr): 3268, 2923, 1658, 1511 and 802  $\text{cm}^{-1}$

***N*-(4-Methoxyphenyl)-2-(pyridin-2-yl)benzamide (3s)**



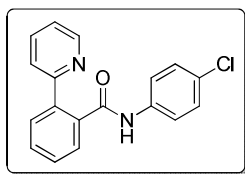
Brown solid; m.p. 111-113 °C;  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  9.15 (s, NH), 8.46 (d,  $J = 4.8$  Hz, 1 H), 7.66 (td,  $J = 1.6$  Hz,  $J = 7.6$  Hz, 1 H), 7.61 (d,  $J = 7.6$  Hz, 1 H), 7.44 (d,  $J = 8$  Hz, 1 H), 7.37-7.27 (m, 5 H), 7.16 (t,  $J = 6.8$  Hz, 1 H), 6.71 (d,  $J = 9.2$  Hz, 2 H), 3.70 (s, 3 H);  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  167.5 (CO), 158.0 (C), 156.1 (C), 148.5 (CH), 138.0 (C), 136.7 (CH), 136.2 (C), 131.3 (C), 130.0 (CH), 129.9 (CH), 128.8 (CH), 128.4 (CH), 123.9 (CH), 122.3 (CH), 121.7 (2 CH), 113.7 (2 CH), 55.2 ( $\text{CH}_3$ ); **HRMS** (ESI/[2M+Na] $^+$ ) calcd for  $\text{C}_{38}\text{H}_{32}\text{N}_4\text{NaO}_4$  631.2321 found 631.2340; IR (KBr): 3279, 2923, 1658, 1511 and 786  $\text{cm}^{-1}$

***N*-(4-Bromophenyl)-2-(pyridin-2-yl)benzamide (3t)**



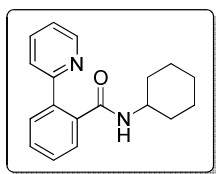
White solid; m.p. 180-182 °C;  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  9.61 (s, NH), 8.53 (d,  $J = 4.8$  Hz, 1 H), 7.73 (t,  $J = 7.6$  Hz, 1 H), 7.60 (d,  $J = 7.6$  Hz, 1 H), 7.46 (d,  $J = 7.6$  Hz, 1 H), 7.42-7.22 (m, 8 H);  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  167.6 (CO), 158.2 (C), 148.3 (CH), 138.2 (C), 137.4 (C), 137.1 (CH), 135.8 (C), 131.5 (2 CH), 130.3 (CH), 130.2 (CH), 129.0 (CH), 128.4 (CH), 124.1 (CH), 122.5 (CH), 121.5 (2 CH), 116.5 (C); **HRMS** (ESI/[2M+Na] $^+$ ) calcd for  $\text{C}_{36}\text{H}_{26}\text{Br}_2\text{N}_4\text{NaO}_2$  727.0320 found 729.0309; IR (KBr): 3247, 2923, 1658, 1535 and 794  $\text{cm}^{-1}$

***N*-(4-Chlorophenyl)-2-(pyridin-2-yl)benzamide (3u)**



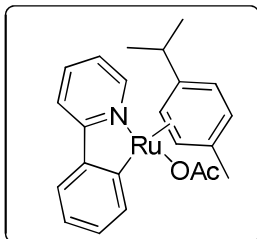
White solid; m.p. 133-135 °C; **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>): δ 9.49 (s, NH), 8.53 (d, *J* = 4.8 Hz, 1 H), 7.73 (t, *J* = 7.6 Hz, 1 H), 7.62 (d, *J* = 7.6 Hz, 1 H), 7.46 (d, *J* = 7.6 Hz, 1 H), 7.43-7.30 (m, 5 H), 7.24 (t, *J* = 7.6 Hz, 1 H), 7.13 (d, *J* = 8.4 Hz, 2 H); **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>): δ 167.7 (CO), 158.2 (C), 148.3 (CH), 138.2 (C), 137.1 (CH), 136.9 (C), 135.8 (C), 130.3 (CH), 130.2 (CH), 129.0 (CH), 128.9 (C), 128.6 (2 CH), 128.5 (CH), 124.1 (CH), 122.5 (CH), 121.1 (2 CH); **HRMS** (ESI/[2M+Na]<sup>+</sup>) calcd for C<sub>36</sub>H<sub>26</sub>Cl<sub>2</sub>N<sub>4</sub>NaO<sub>2</sub> 639.1331 found 639.1327; IR (KBr): 3239, 2923, 1658, 1535 and 1095 cm<sup>-1</sup>

***N*-Cyclohexyl-2-(pyridin-2-yl)benzamide (3v)**



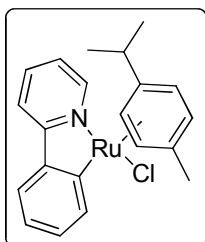
Yellow paste; **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>): δ 8.63-8.61 (m, 1 H), 7.72-7.68 (m, 1 H), 7.61 (dd, *J* = 1.6 Hz, *J* = 7.2 Hz, 1 H), 7.51-7.39 (m, 4 H), 7.27-7.25 (m, 1 H), 5.82 (d, *J* = 7.2 Hz, 1 H), 3.78-3.70 (m, 1 H), 1.70-1.66 (m, 2 H), 1.56-1.48 (m, 2 H), 1.31-1.20 (m, 3 H), 1.09-0.98 (m, 1 H), 0.90-0.80 (m, 2 H); **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>): δ 168.6 (CO), 158.3 (C), 149.0 (CH), 138.2 (C), 136.6 (C), 136.5 (CH), 129.8 (CH), 129.7 (CH), 128.5 (CH), 128.4 (CH), 124.0 (CH), 122.3 (CH), 48.3 (CH), 32.5 (2 CH<sub>2</sub>), 29.6 (CH<sub>2</sub>), 25.3 (CH<sub>2</sub>), 24.5 (CH<sub>2</sub>); **HRMS** (ESI/[2M+Na]<sup>+</sup>) calcd for C<sub>36</sub>H<sub>40</sub>N<sub>4</sub>NaO<sub>2</sub> 583.3049 found 583.3035; IR (KBr): 3245, 2923, 1654, 1596 and 817 cm<sup>-1</sup>

**Complex A-OAc**



**$^1\text{H}$  NMR** (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  9.58 (d,  $J = 5.6$  Hz, 1 H), 8.35 (d,  $J = 7.2$  Hz, 1 H), 7.57 (d,  $J = 7.6$  Hz, 2 H), 7.24 (t,  $J = 6.4$  Hz, 2 H), 7.12-7.09 (m, 2 H), 5.66 (d,  $J = 6$  Hz, 1 H), 5.61 (d,  $J = 6$  Hz, 1 H), 5.42 (d,  $J = 5.6$  Hz, 1 H), 5.18 (d,  $J = 6$  Hz, 1 H), 2.58-2.17 (m, 1 H), 1.96 (s, 3 H), 1.65 (s, 3 H), 0.87-0.71 (m, 6 H);  **$^{13}\text{C}$  NMR** (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  180.4 (C), 166.9 (CO), 158.0 (C), 156.3 (CH), 139.4 (C), 135.1 (CH), 131.3 (CH), 127.7 (CH), 123.5 (CH), 122.6 (CH), 121.3 (CH), 118.3 (CH), 99.6 (C), 99.4 (C), 90.0 (CH), 89.4 (CH), 86.7 (CH), 82.4 (CH), 31.9 (CH), 22.6 ( $\text{CH}_3$ ), 22.4 ( $\text{CH}_3$ ), 21.3 ( $\text{CH}_3$ ), 18.7 ( $\text{CH}_3$ ); Chemical Formula:  $\text{C}_{23}\text{H}_{25}\text{NO}_2\text{Ru}$ ; IR (KBr): 2923, 1828, 1697, 1558, 1519, 1095 and  $509\text{ cm}^{-1}$

#### Complex A-Cl

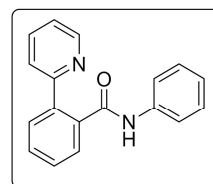
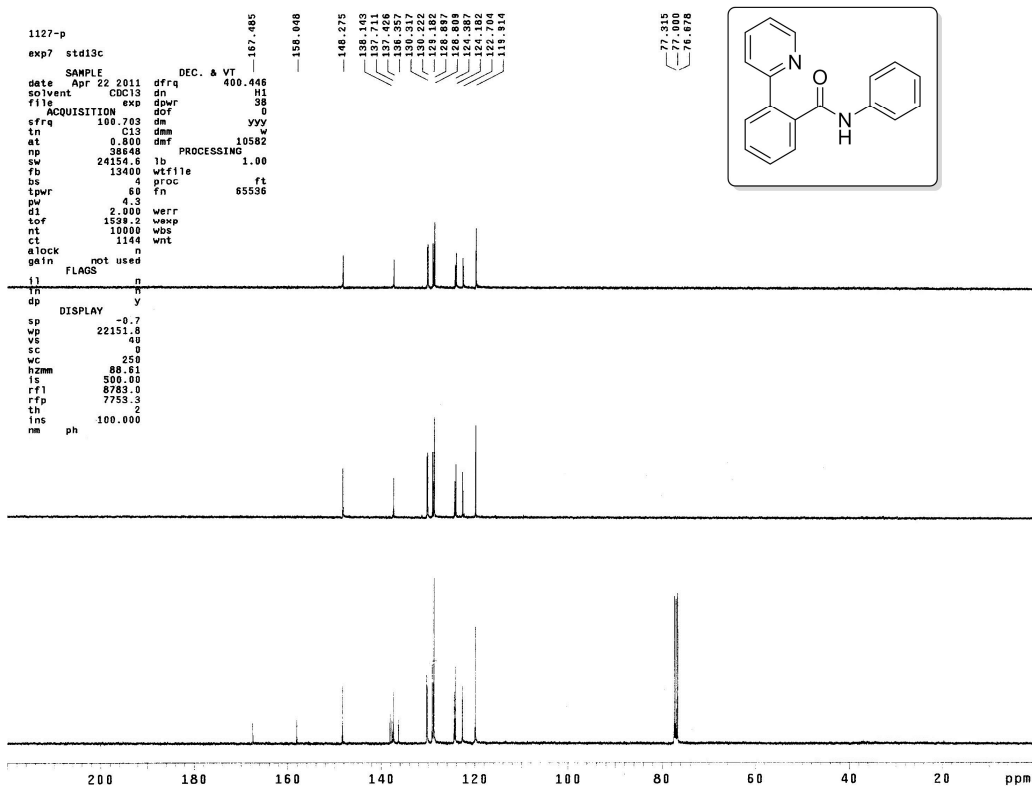
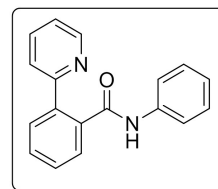
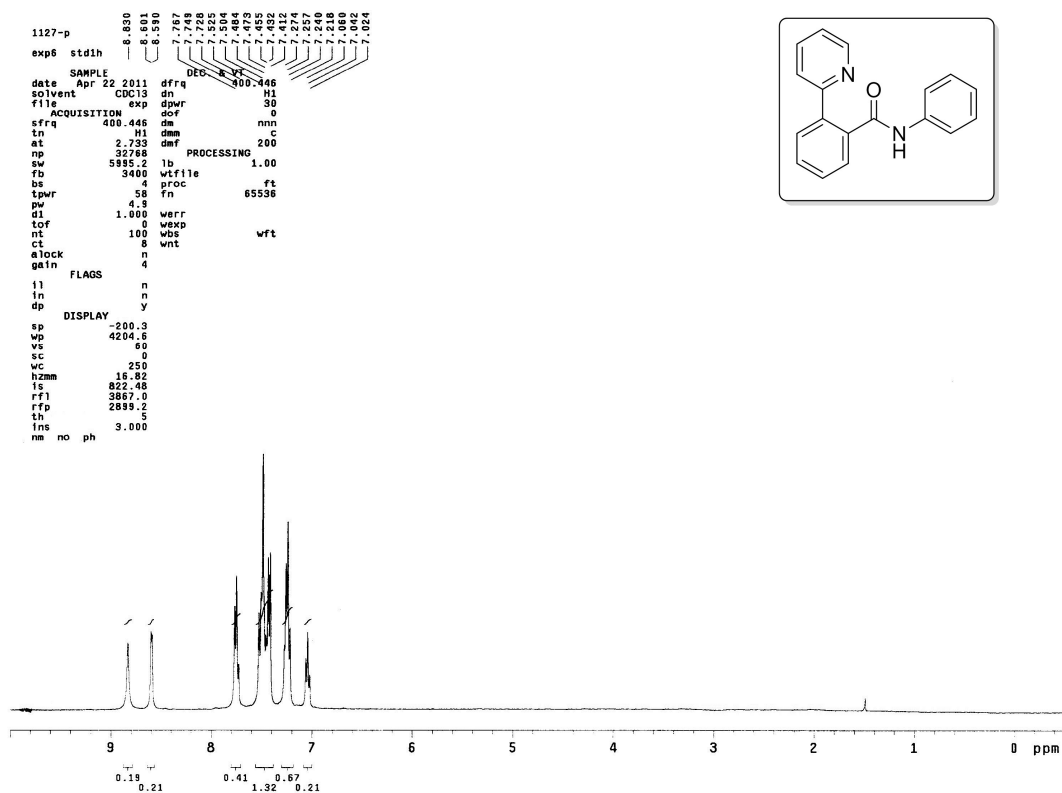


**$^1\text{H}$  NMR** (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  9.30 (dd,  $J = 1.2$  Hz,  $J = 4.4$  Hz, 1 H), 8.02 (dd,  $J = 1.2$  Hz,  $J = 8$  Hz, 1 H), 7.68-7.58 (m, 3 H), 7.13 (t,  $J = 7.2$  Hz, 1 H), 6.98-6.84 (m, 2 H), 5.49 (t,  $J = 6$  Hz, 2 H), 5.12 (d,  $J = 6$  Hz, 1 H), 4.99 (d,  $J = 6$  Hz, 1 H), 2.63-2.56 (m, 1 H), 2.17 (s, 3 H), 1.00 (d,  $J = 6.8$  Hz, 3 H), 0.89 (d,  $J = 6.8$  Hz, 3 H);  **$^{13}\text{C}$  NMR** (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  181.2 (C), 165.1 (C), 154.4 (CH), 143.1 (C), 139.4 (CH), 136.5 (CH), 129.2 (CH), 123.7 (CH), 122.3 (CH), 121.2 (CH), 118.6 (CH), 100.5 (C), 100.3 (C), 90.6 (CH), 89.4 (CH), 84.0 (CH), 82.0 (CH), 30.6 (CH), 22.3 ( $\text{CH}_3$ ), 21.5 ( $\text{CH}_3$ ), 18.6 ( $\text{CH}_3$ ); Chemical Formula:  $\text{C}_{21}\text{H}_{22}\text{ClNRu}$ ; IR (KBr): 2923, 2360, 1542, 1049, 786, 624 and  $563\text{ cm}^{-1}$

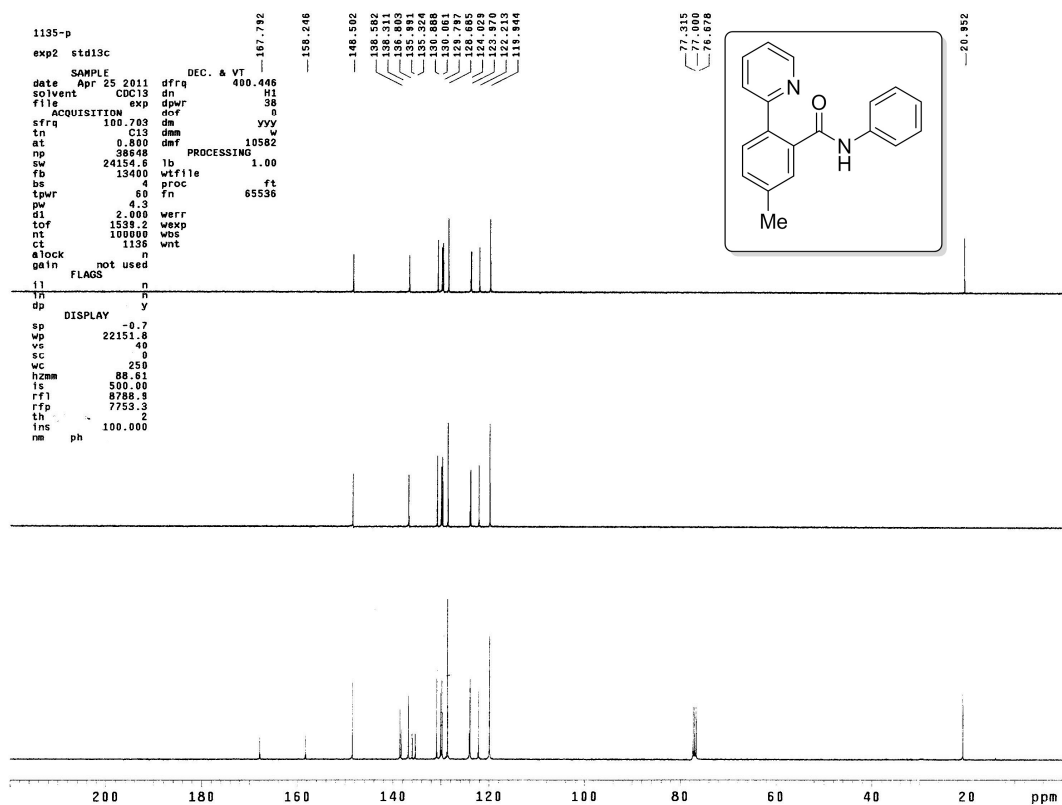
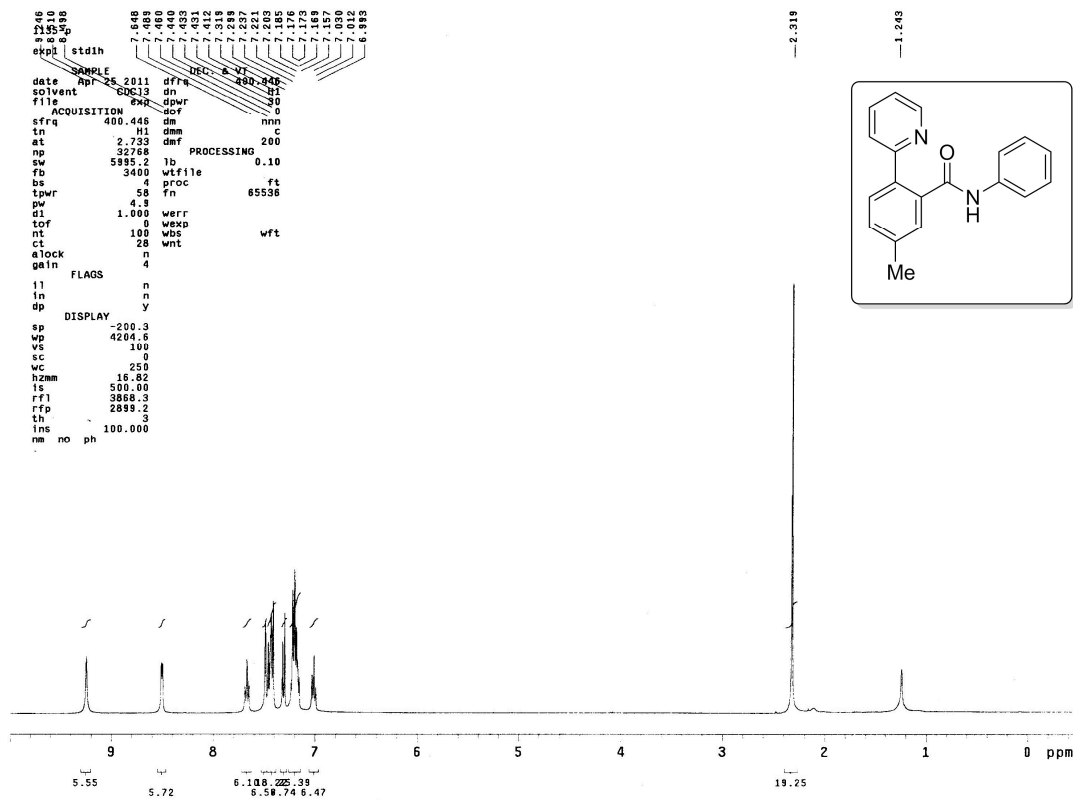
## Reference:

1. Perrin, D. D.; Armarego, W. L. F. *In Purification of Laboratory Chemicals*, 3rd ed.; Pergamon Press: New York, 1988.
2. Pan, C.; Liu, M.; Zhang, L.; Wu, H.; Ding, J.; Cheng, J. *Catal. Commun.* **2008**, *9*, 508.
3. (a) Ackermann, L.; Lygin, A. V.; Hofmann, N. *Angew. Chem., Int. Ed.* **2011**, *50*, 6379. (b) Ackermann, L.; Fenner, S. *Org. Lett.* **2011**, *13*, 6548. (c) Ackermann, L.; Diers, E.; Manvar, A. *Org. Lett.* **2012**, *14*, 1154.

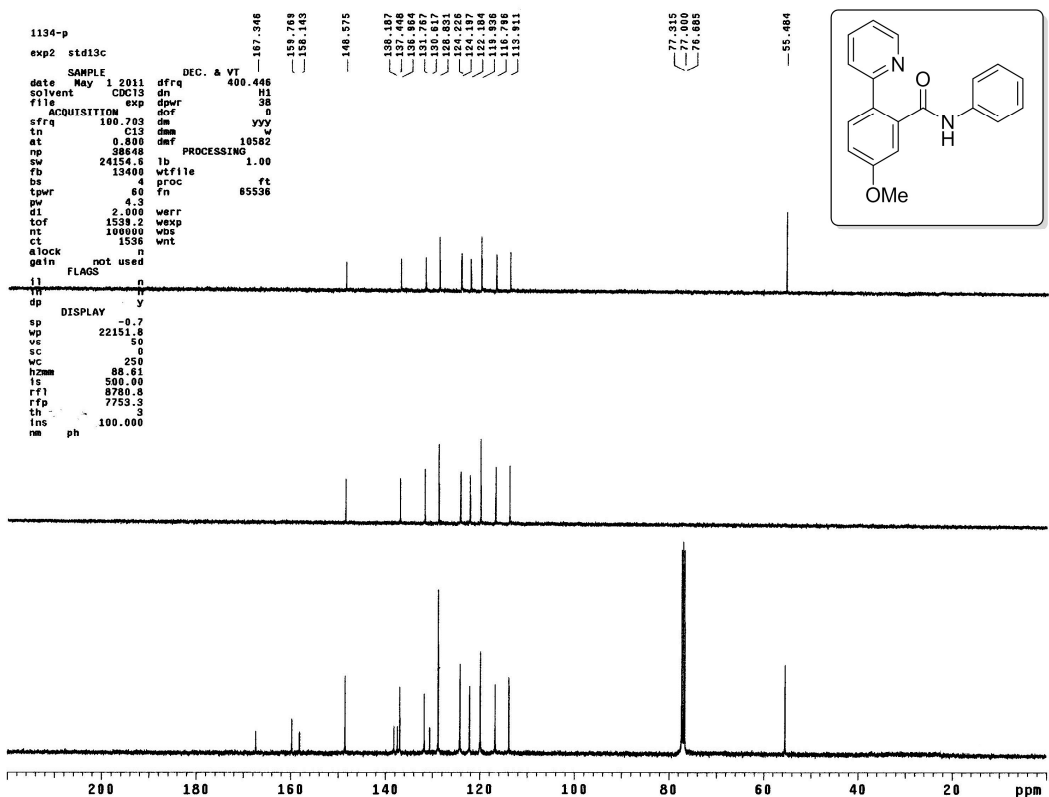
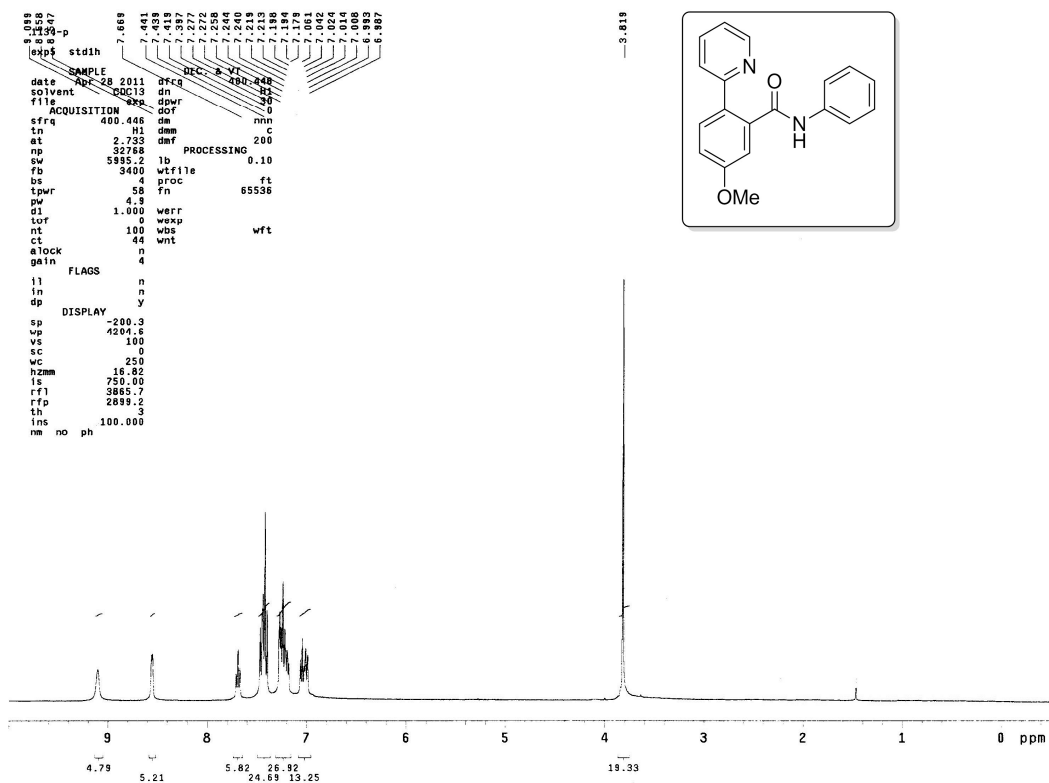
$^1\text{H}$  and  $^{13}\text{C}$  NMR spectra of compound **3a**.



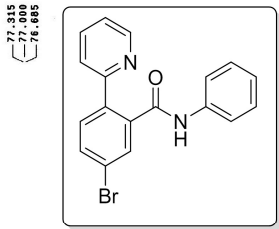
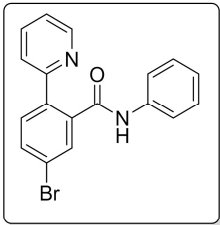
$^1\text{H}$  and  $^{13}\text{C}$  NMR spectra of compound **3b**.

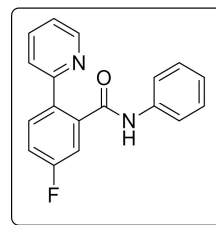
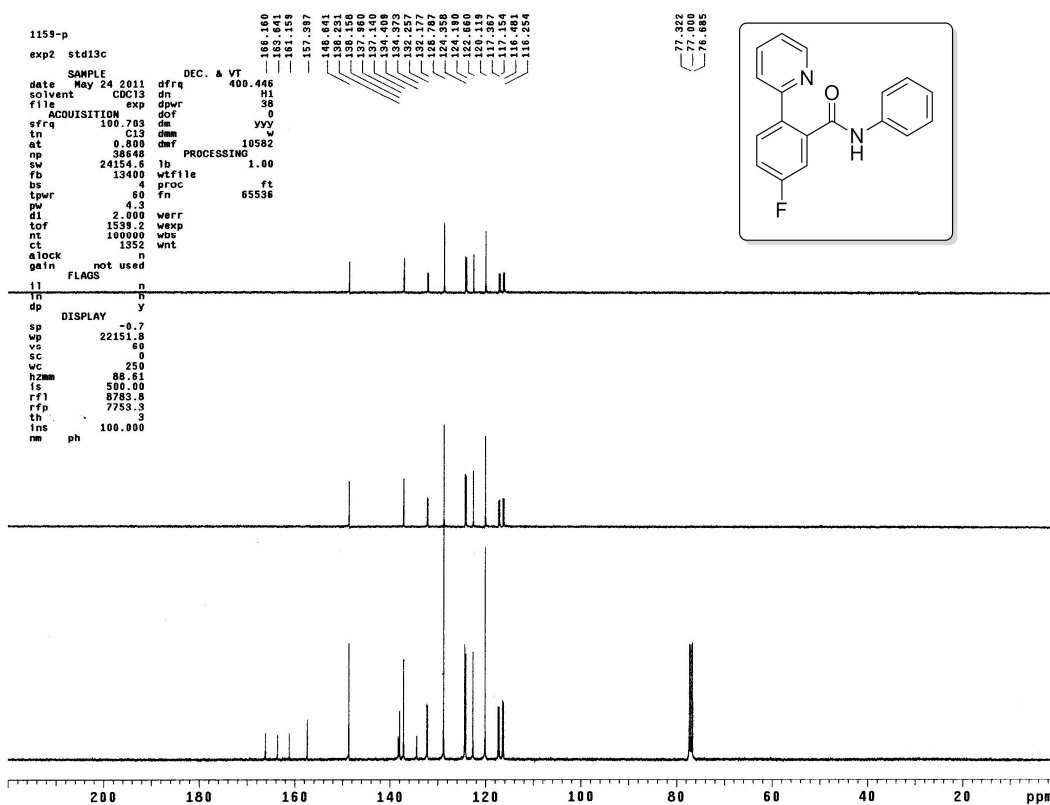
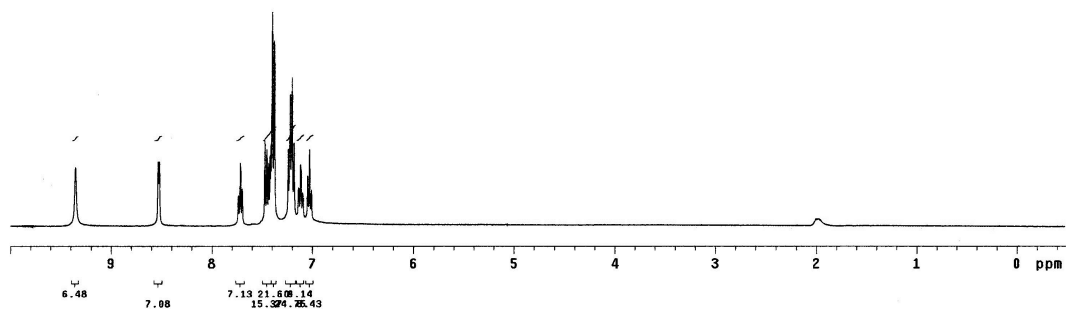
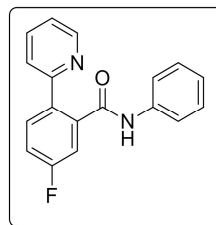


$^1\text{H}$  and  $^{13}\text{C}$  NMR spectra of compound **3c**.

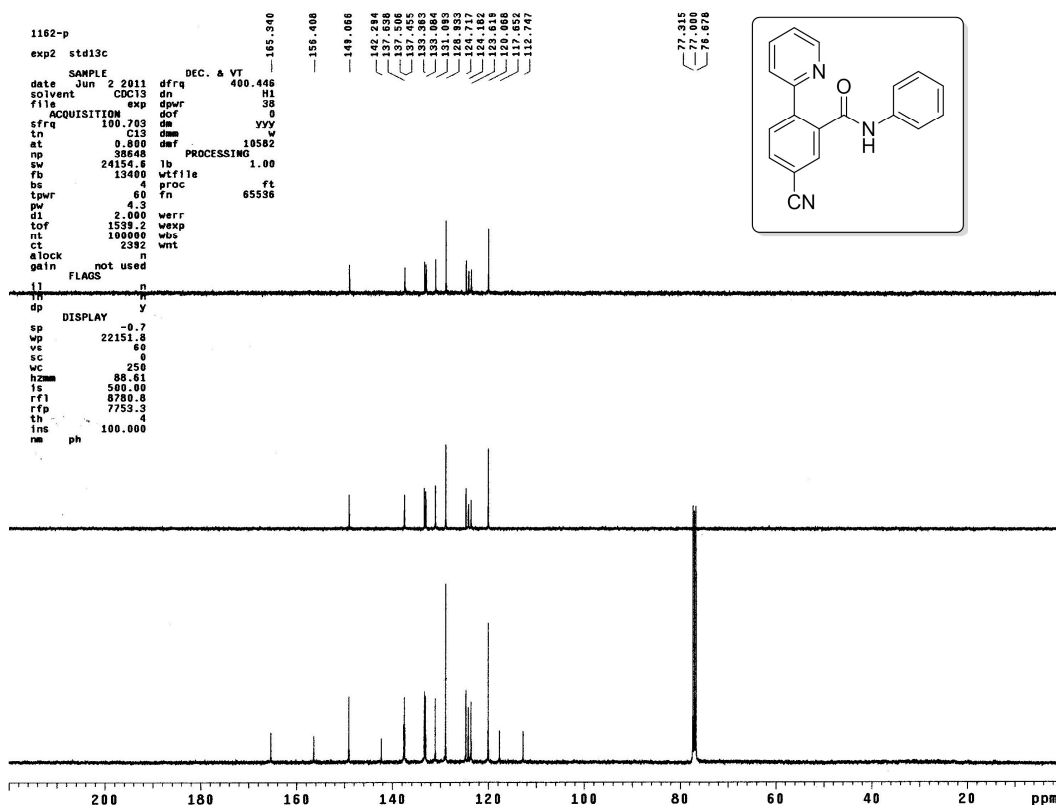
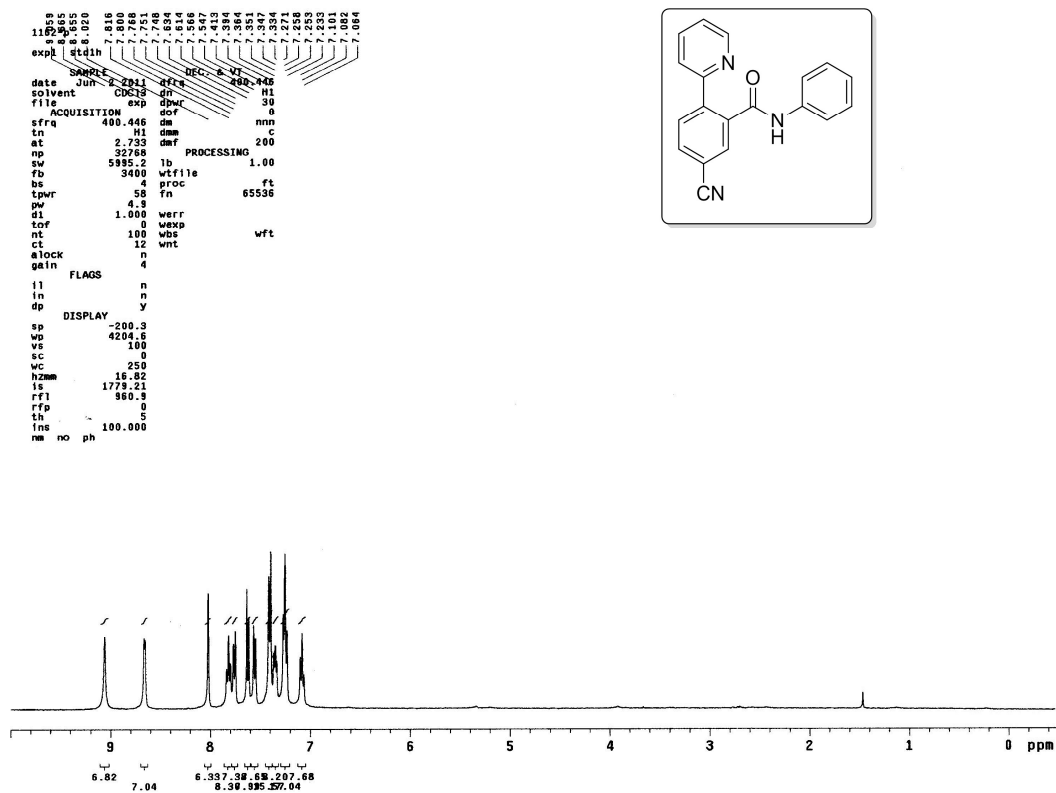




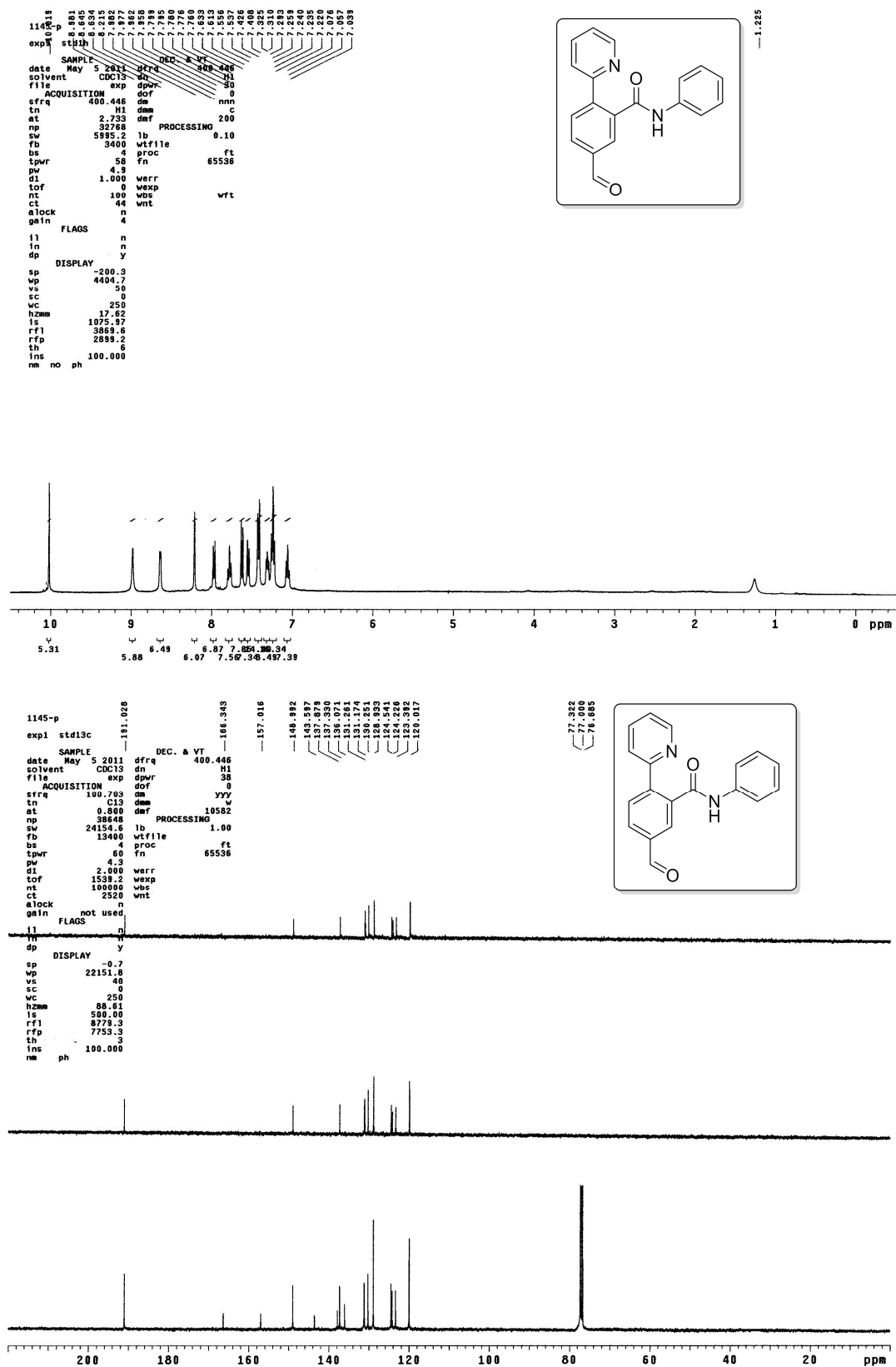
<sup>1</sup>H and <sup>13</sup>C NMR spectra of compound **3d**.

[illegible]

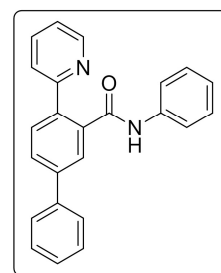
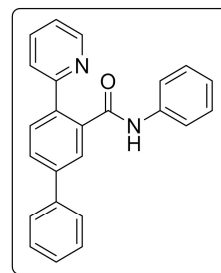
$^1\text{H}$  and  $^{13}\text{C}$  NMR spectra of compound **3f**.



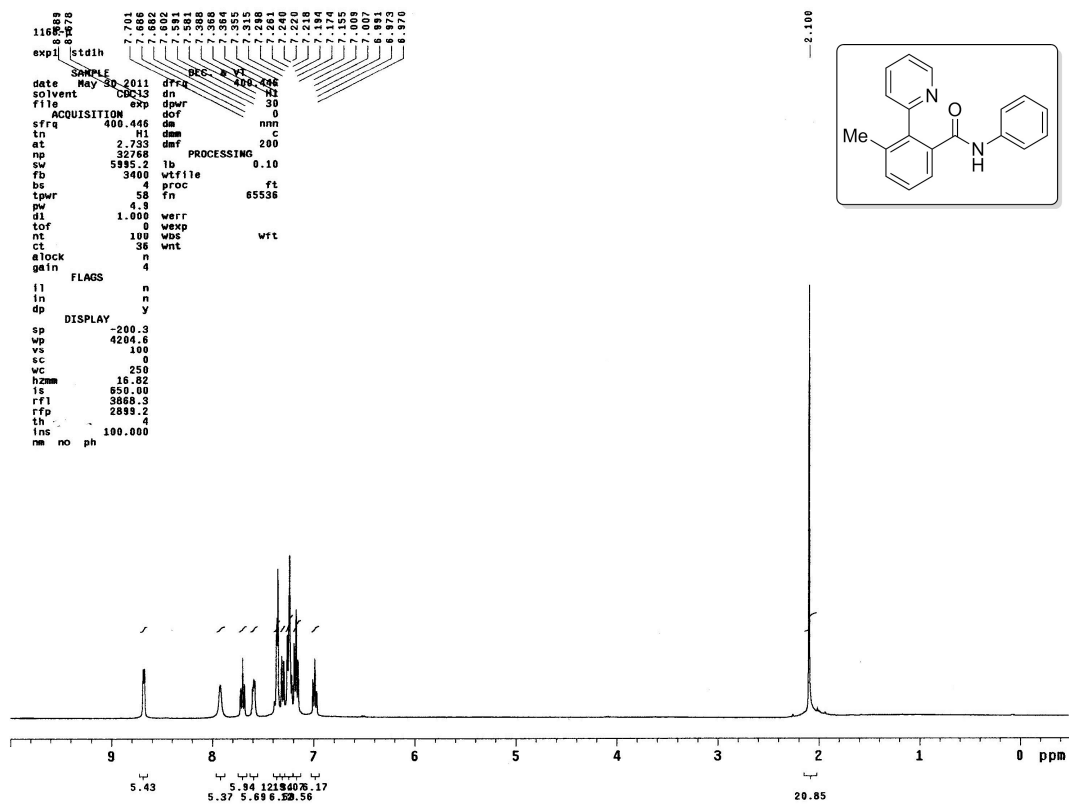
$^1\text{H}$  and  $^{13}\text{C}$  NMR spectra of compound **3g**.



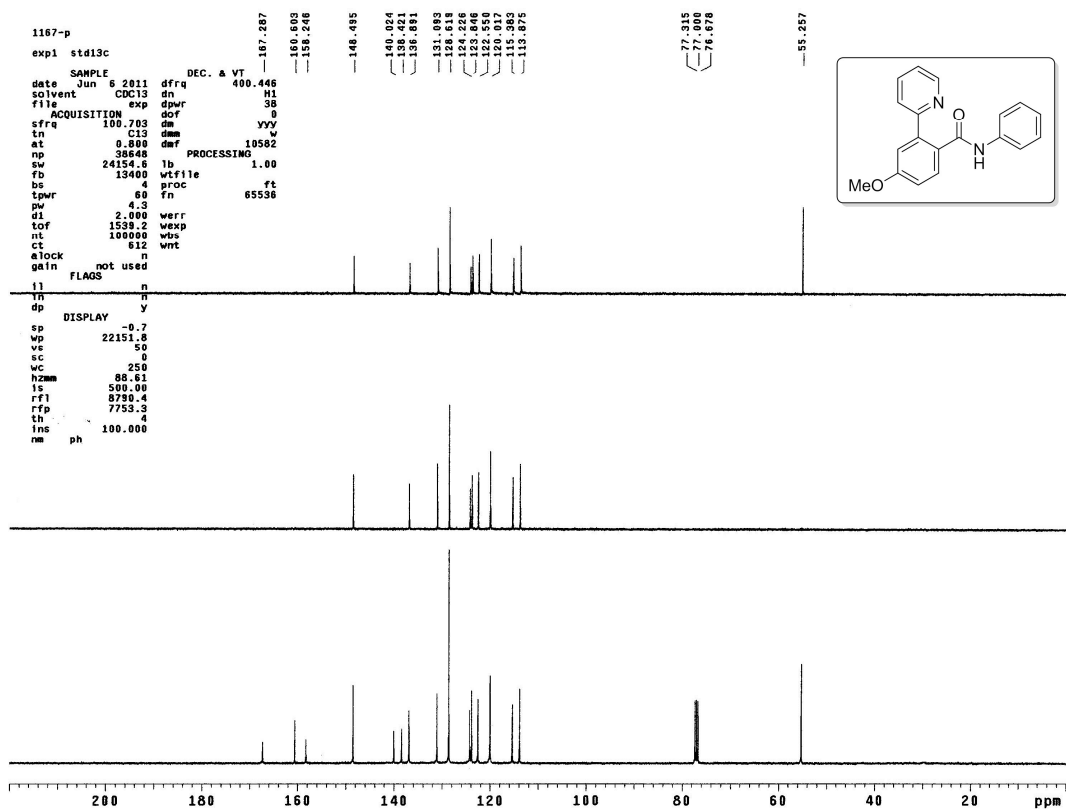
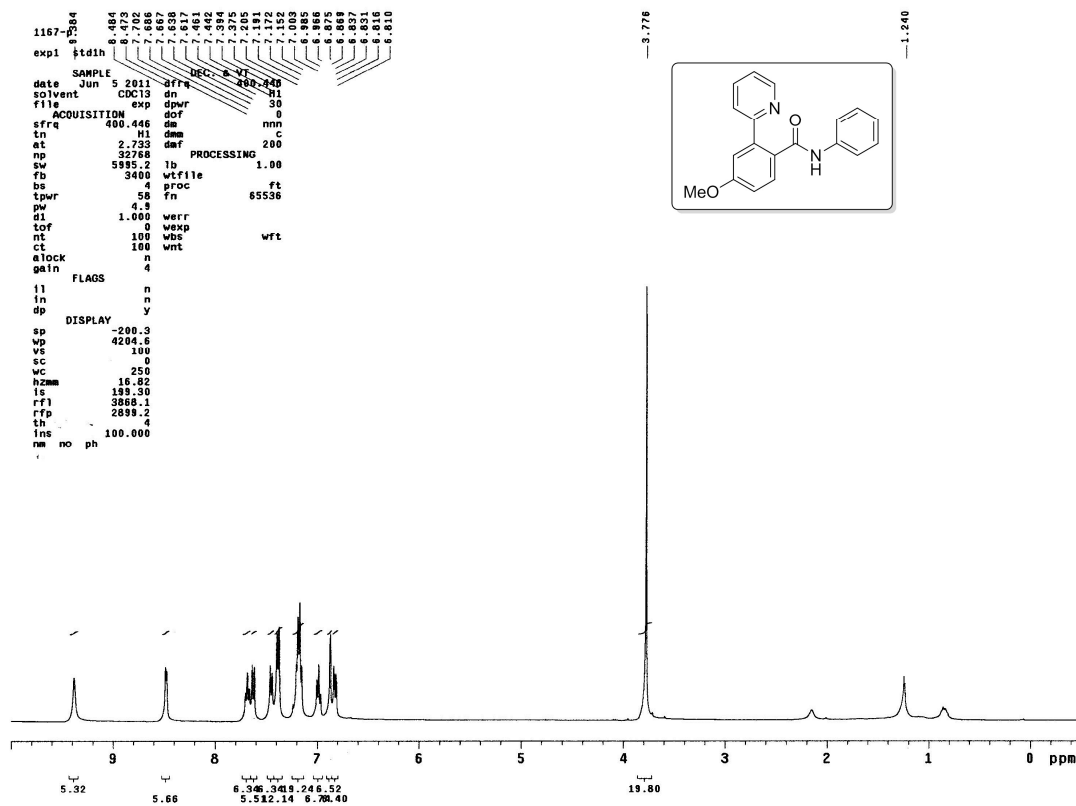
<sup>1</sup>H and <sup>13</sup>C NMR spectra of compound **3h**.



$^1\text{H}$  and  $^{13}\text{C}$  NMR spectra of compound **3i**.

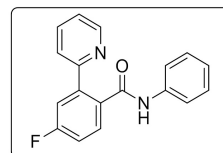
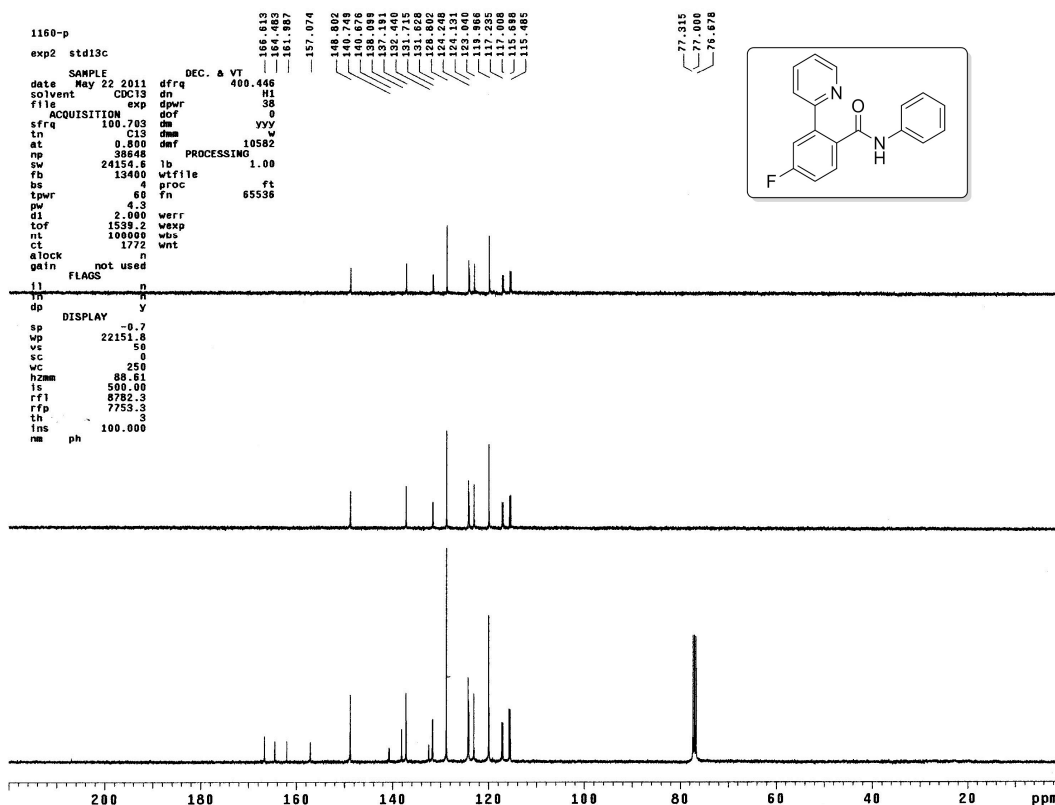
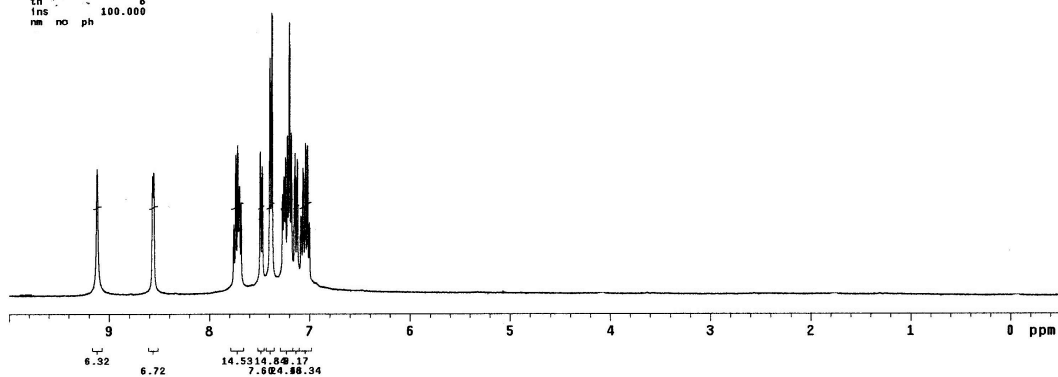
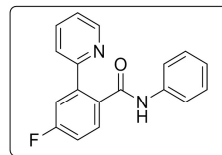


$^1\text{H}$  and  $^{13}\text{C}$  NMR spectra of compound **3j**.



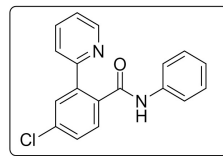
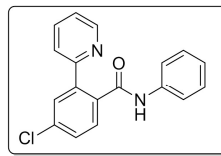
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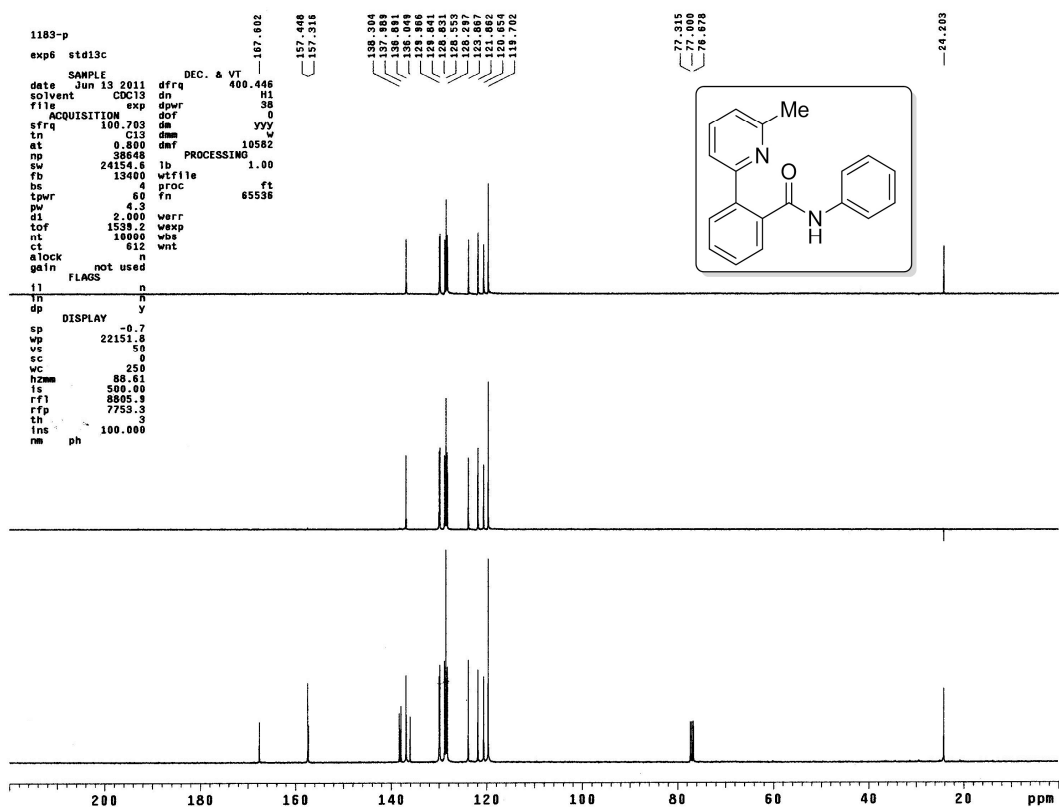
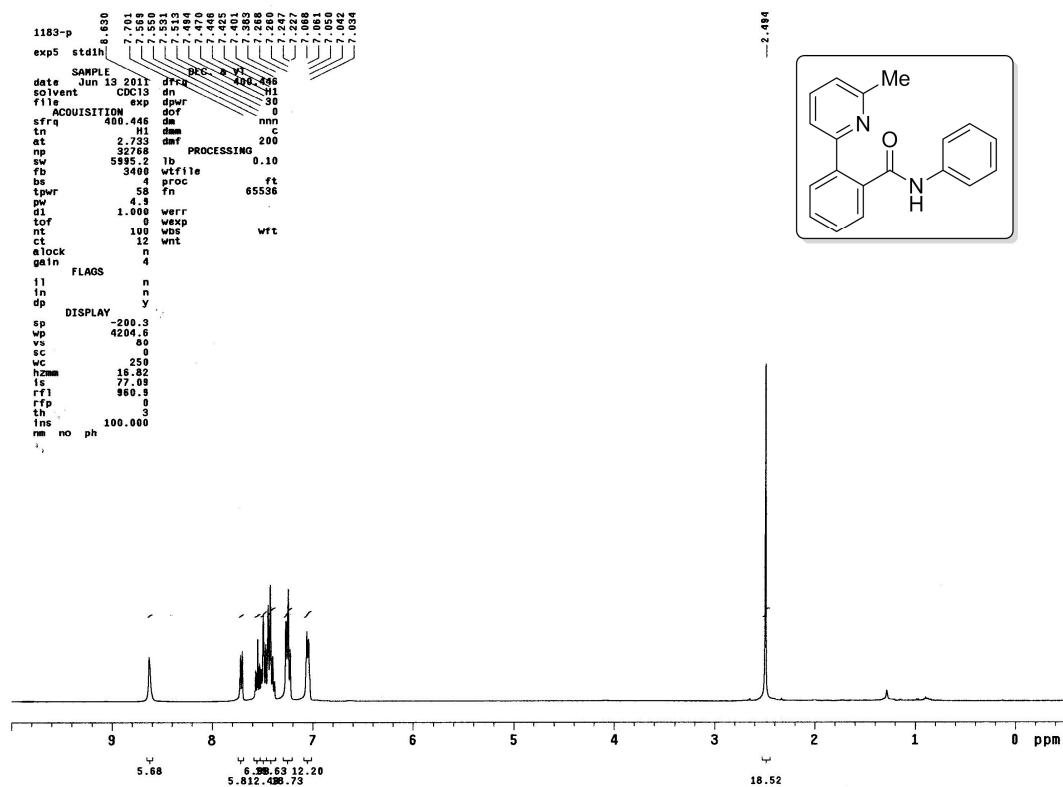


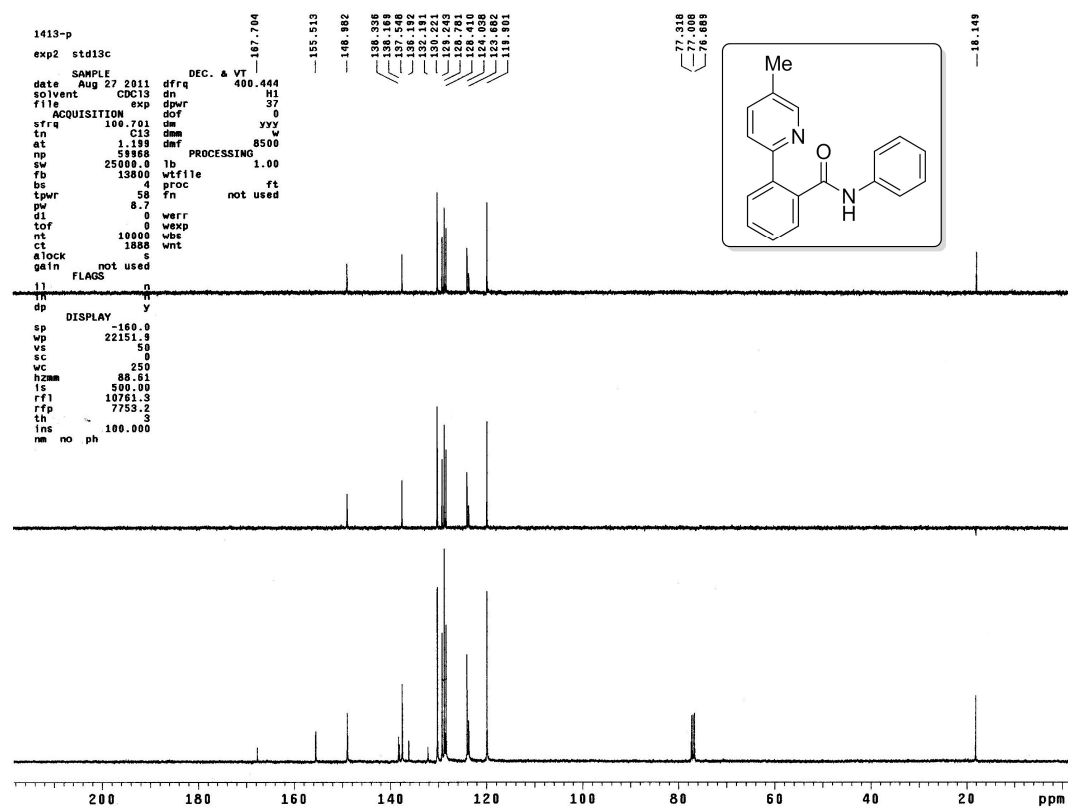
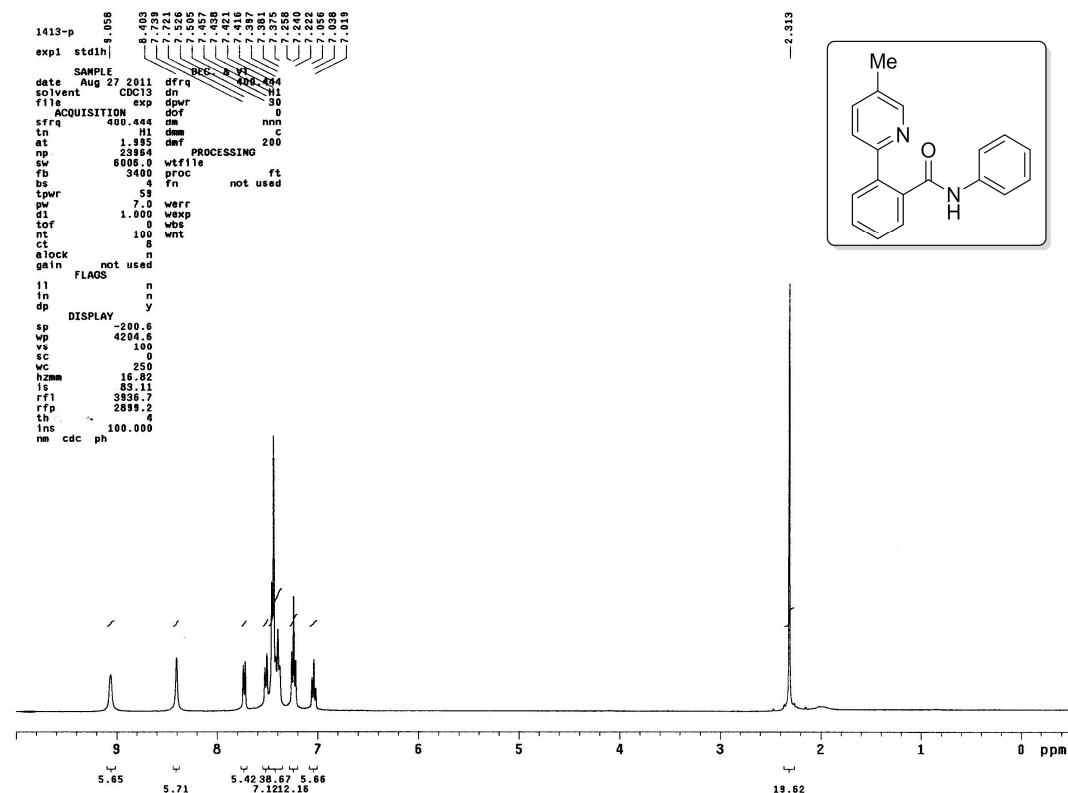


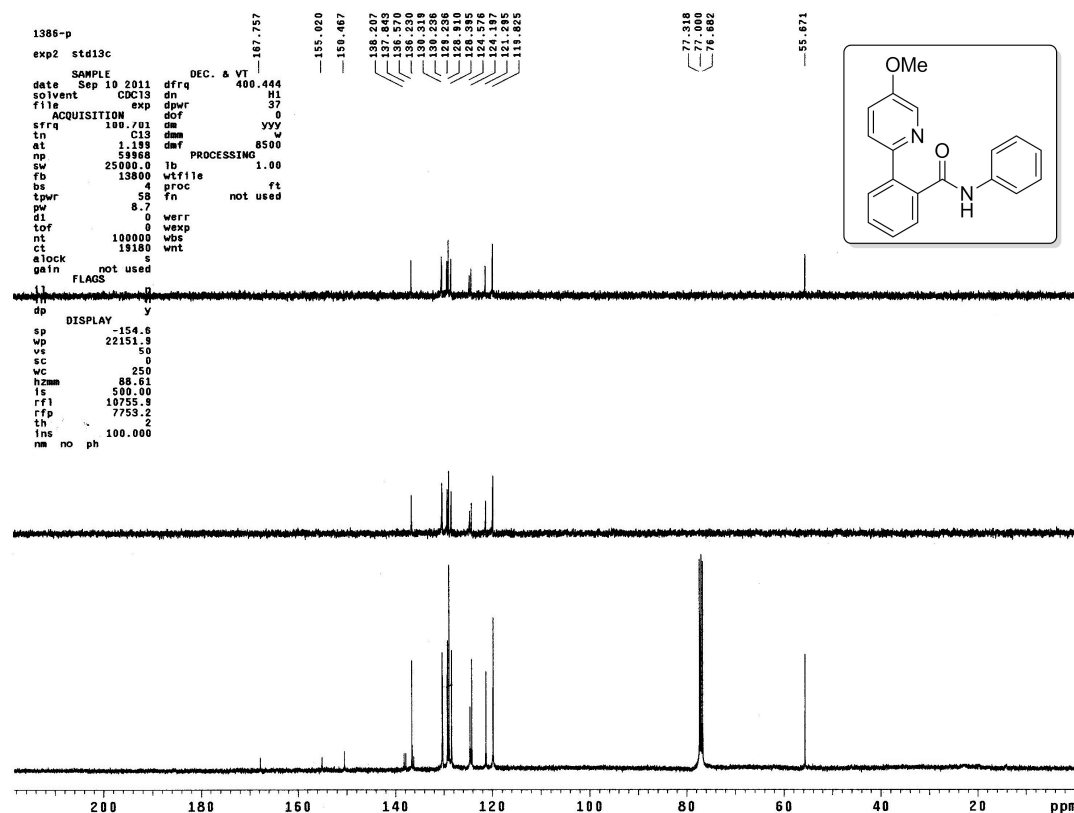
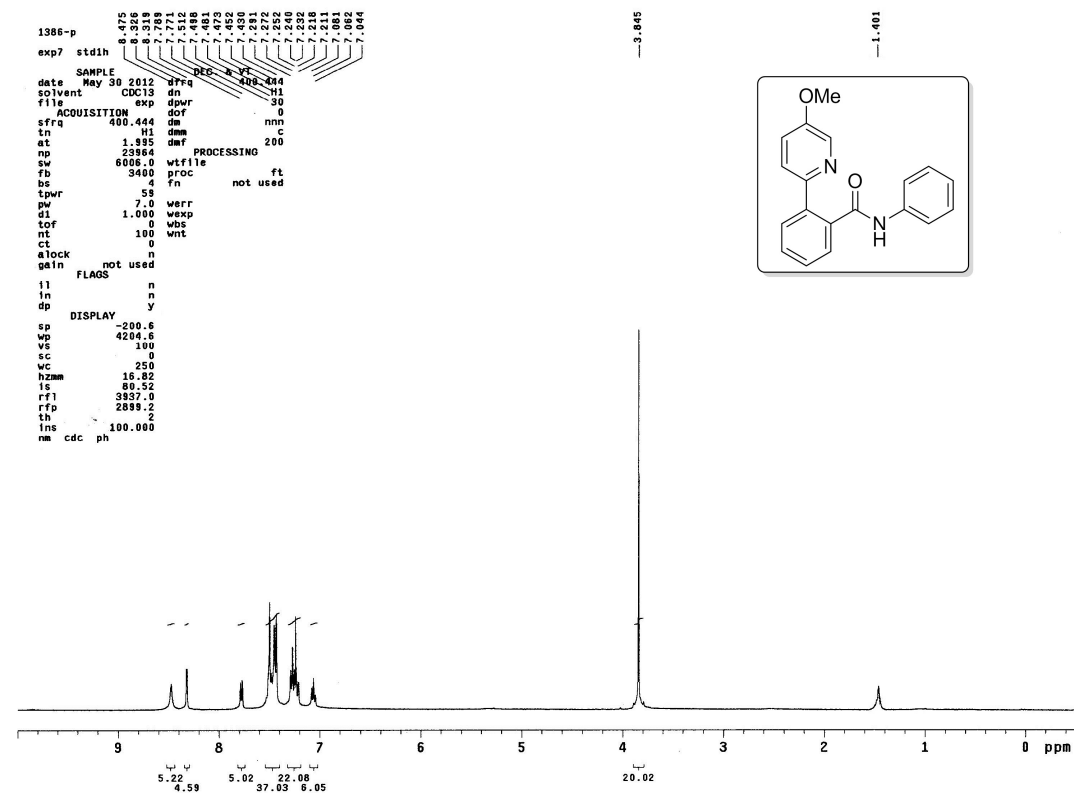


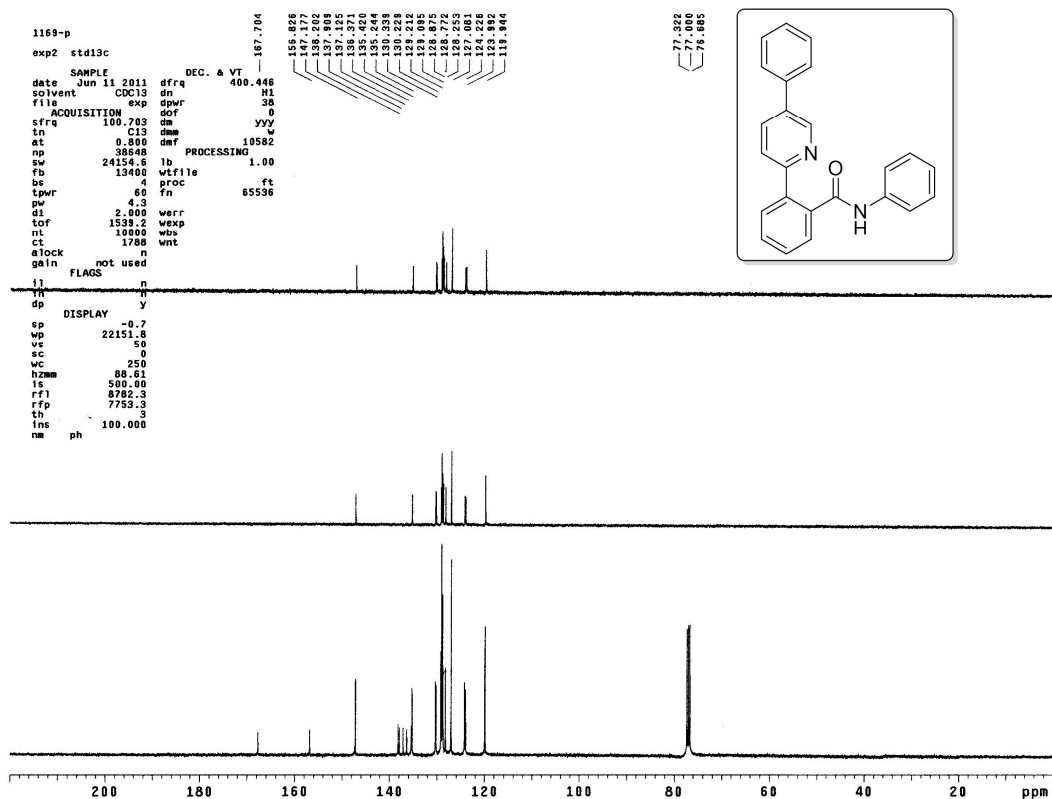
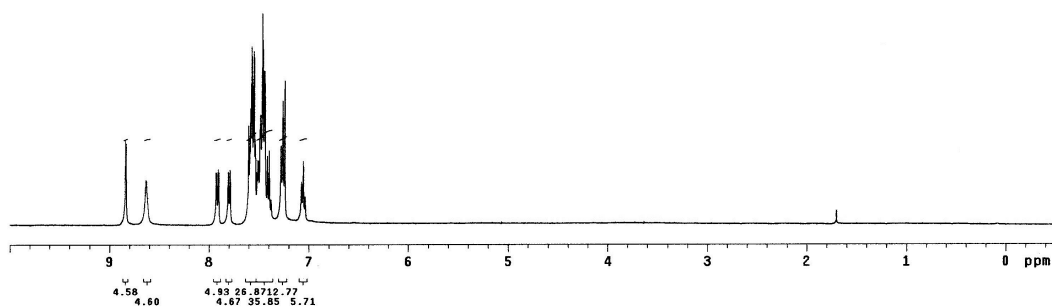
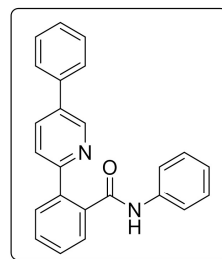
<sup>1</sup>H and <sup>13</sup>C NMR spectra of compound **3l**.

$^1\text{H}$  and  $^{13}\text{C}$  NMR spectra of compound **3m**.

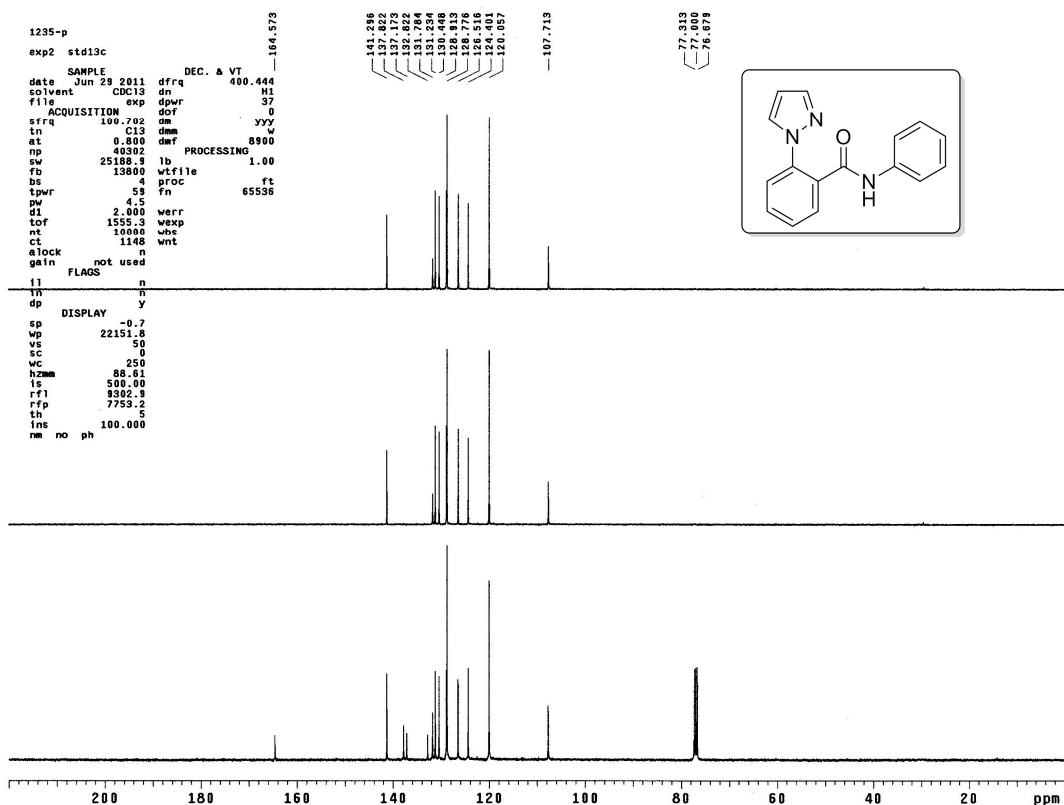
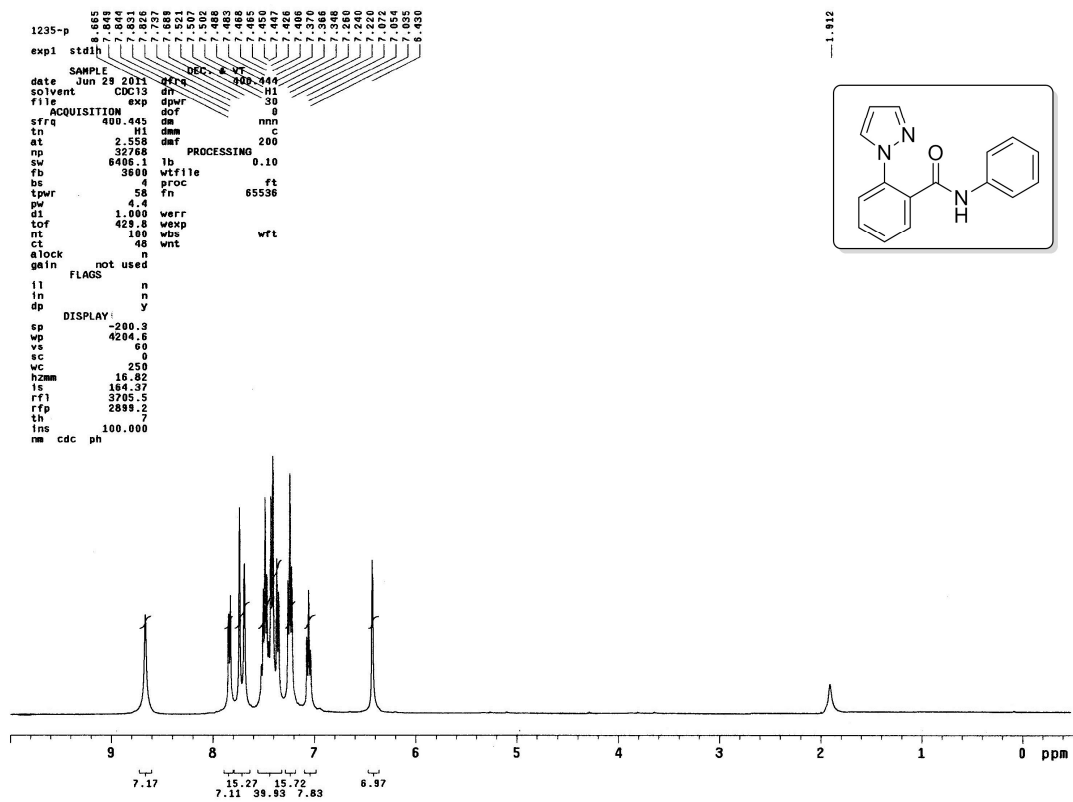


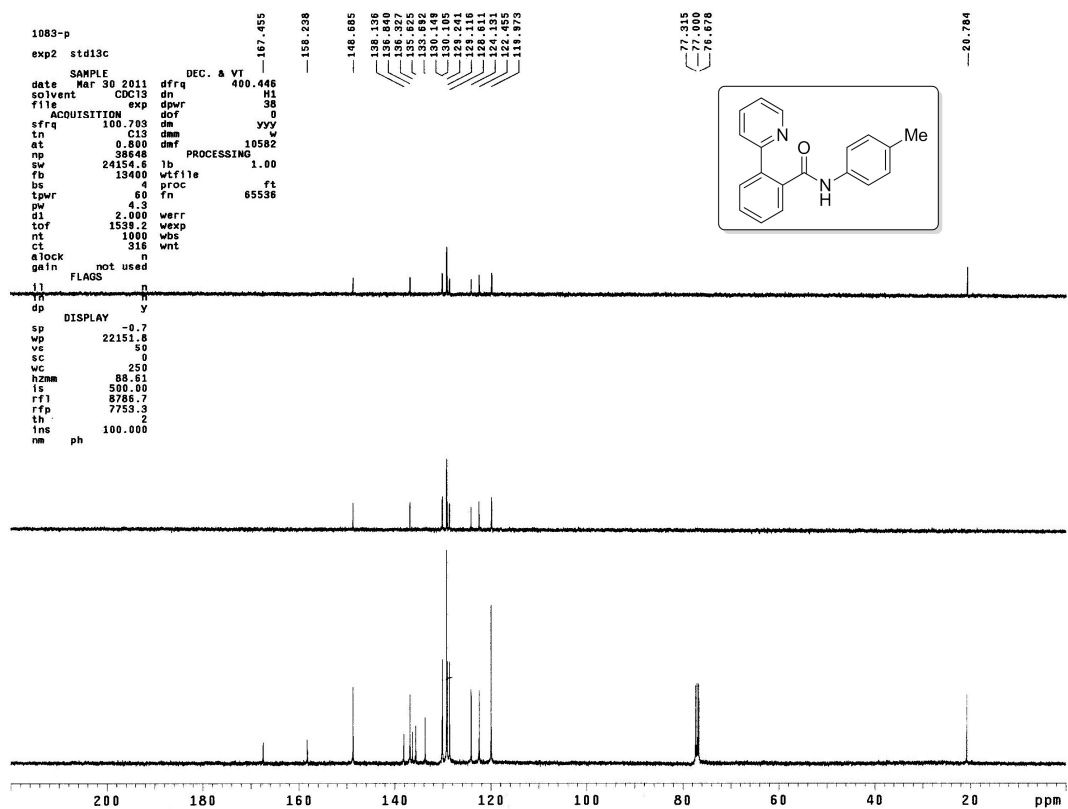
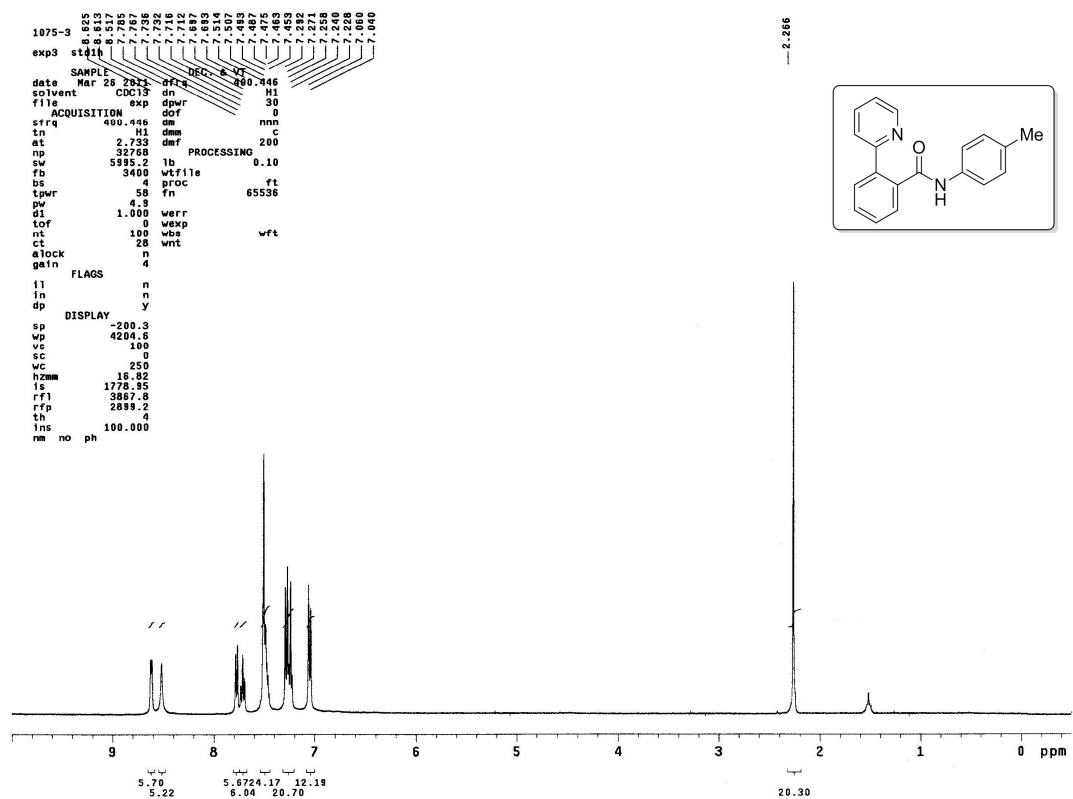




[illegible]

$^1\text{H}$  and  $^{13}\text{C}$  NMR spectra of compound **3q**.

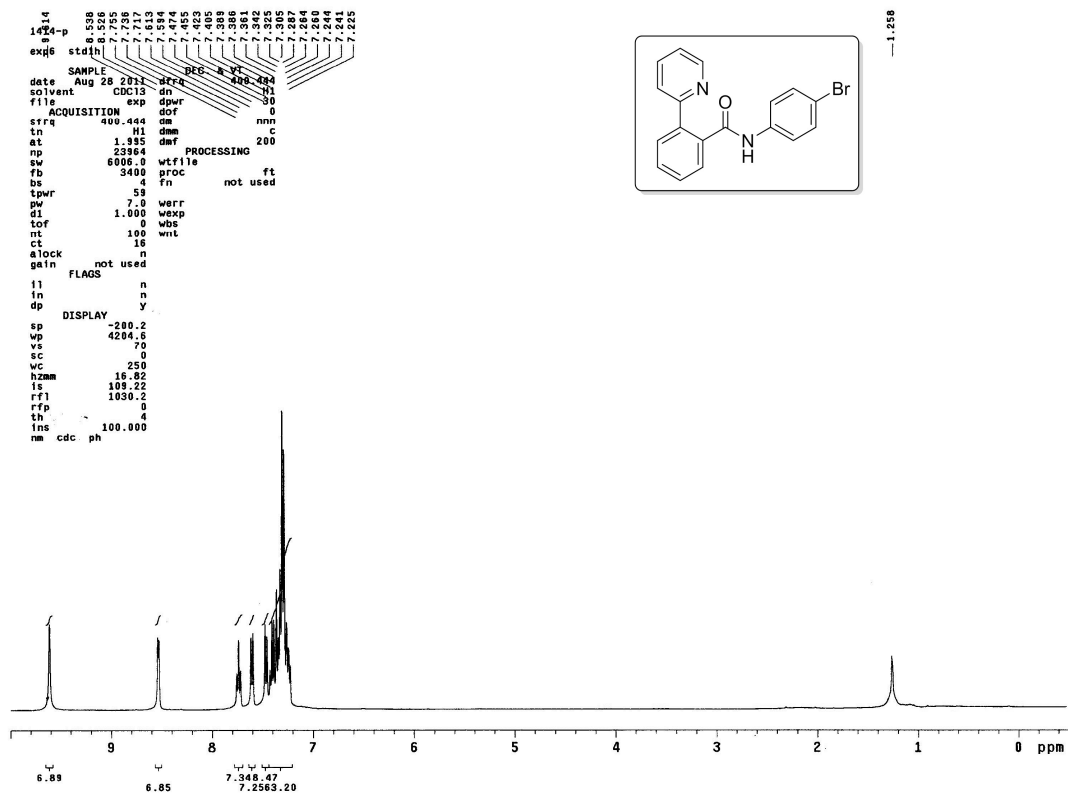






[illegible]

$^1\text{H}$  and  $^{13}\text{C}$  NMR spectra of compound **3t**.



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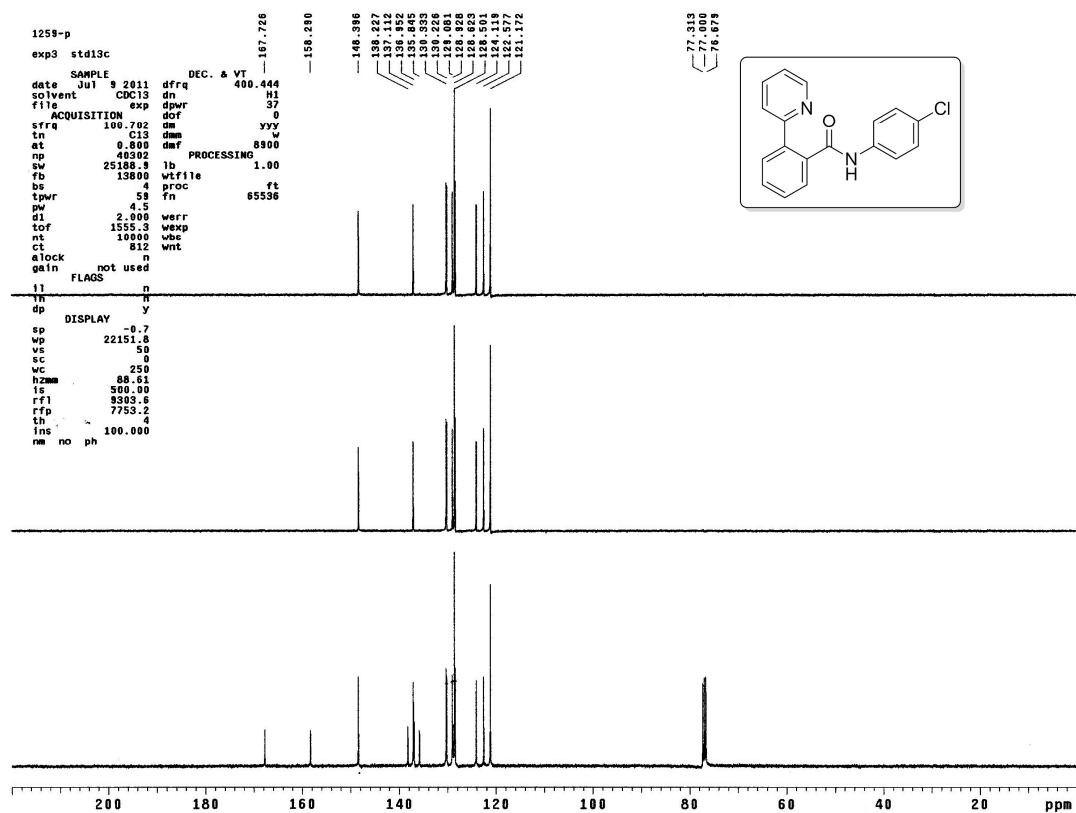
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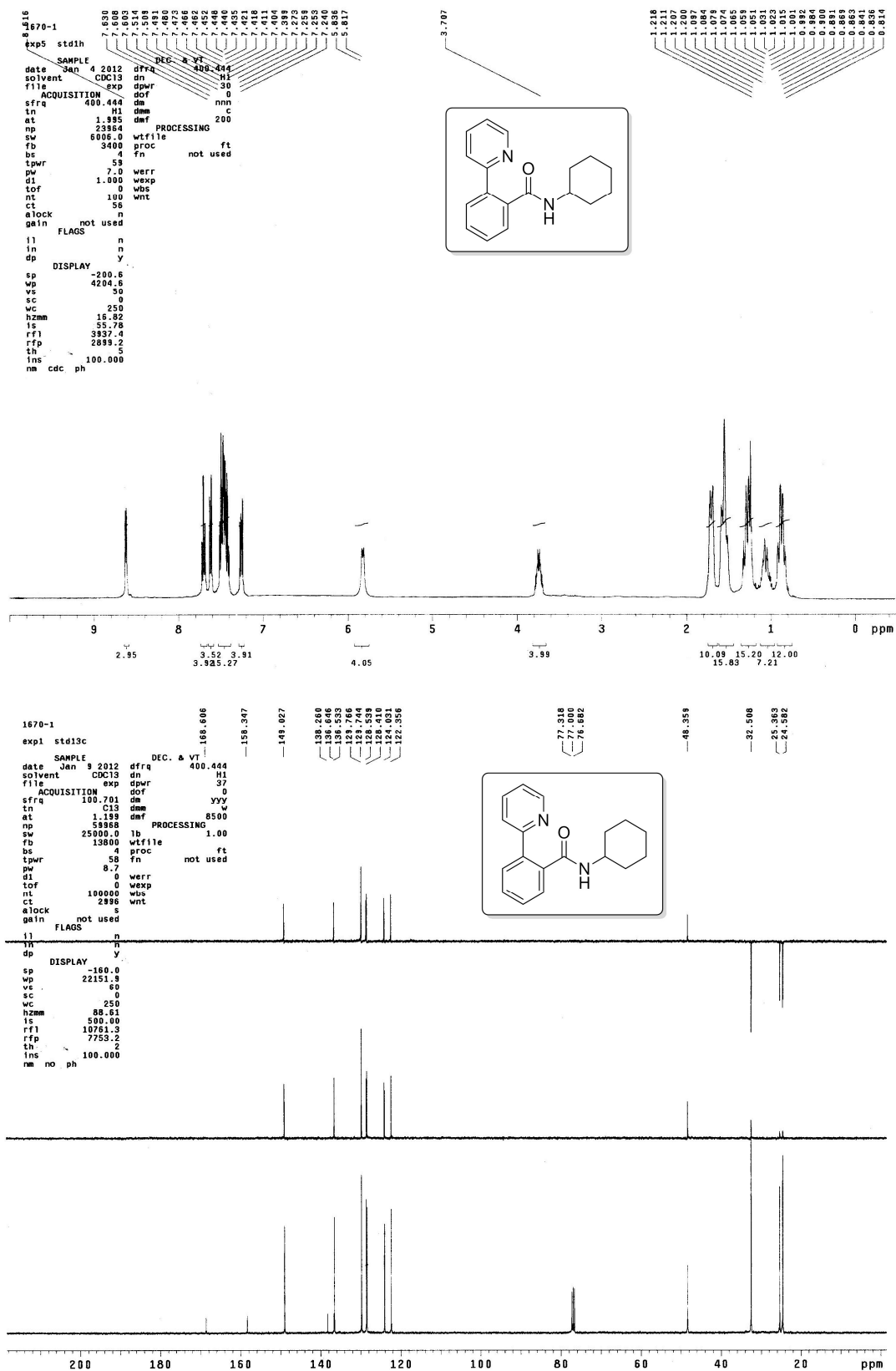
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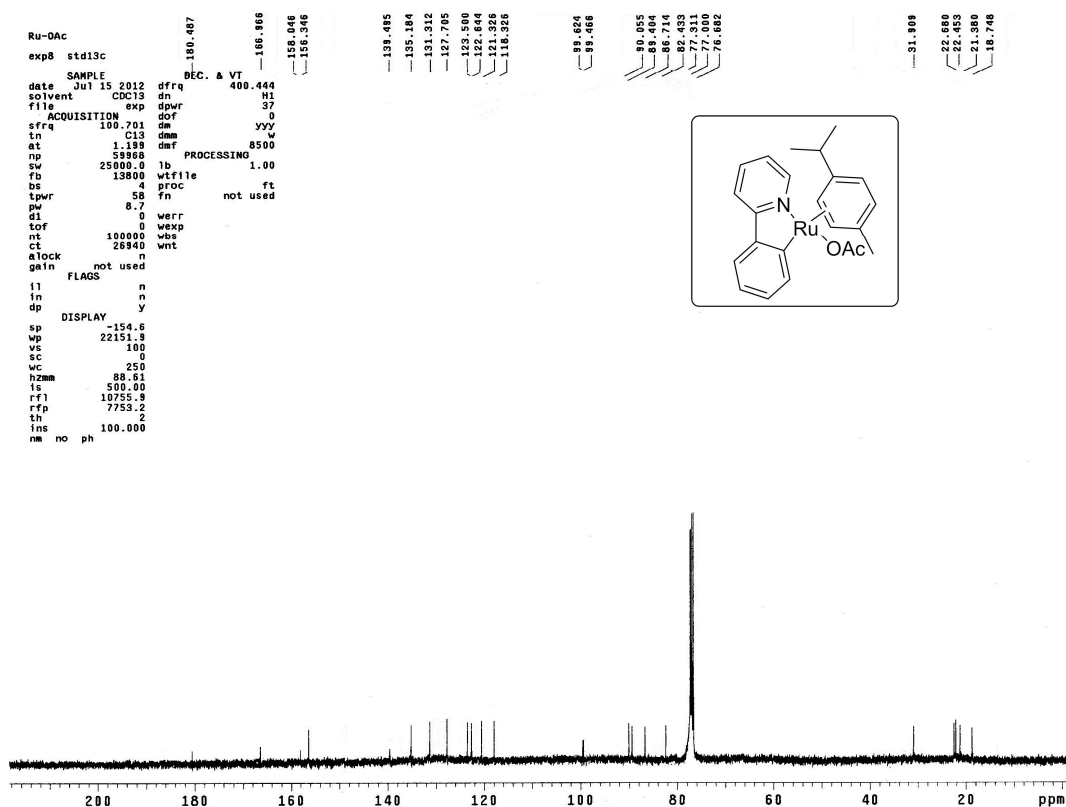
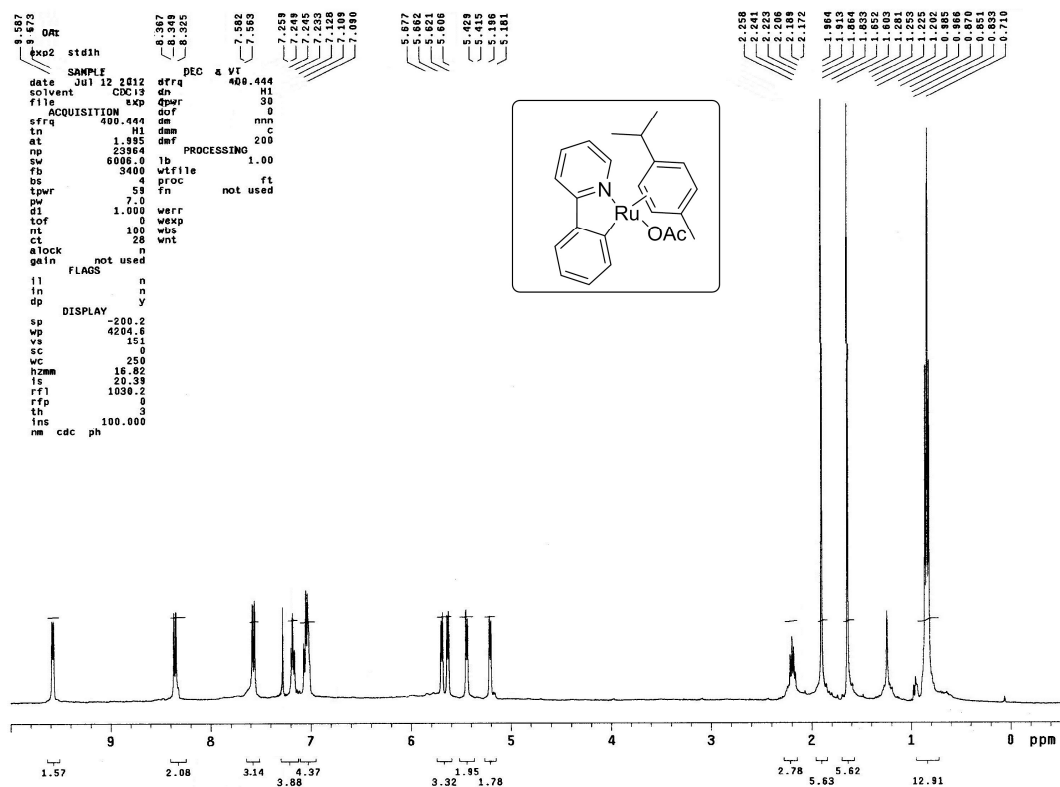
Chemical structure: c1ccc(cc1)C(=O)Nc2ccc(cc2)Cl

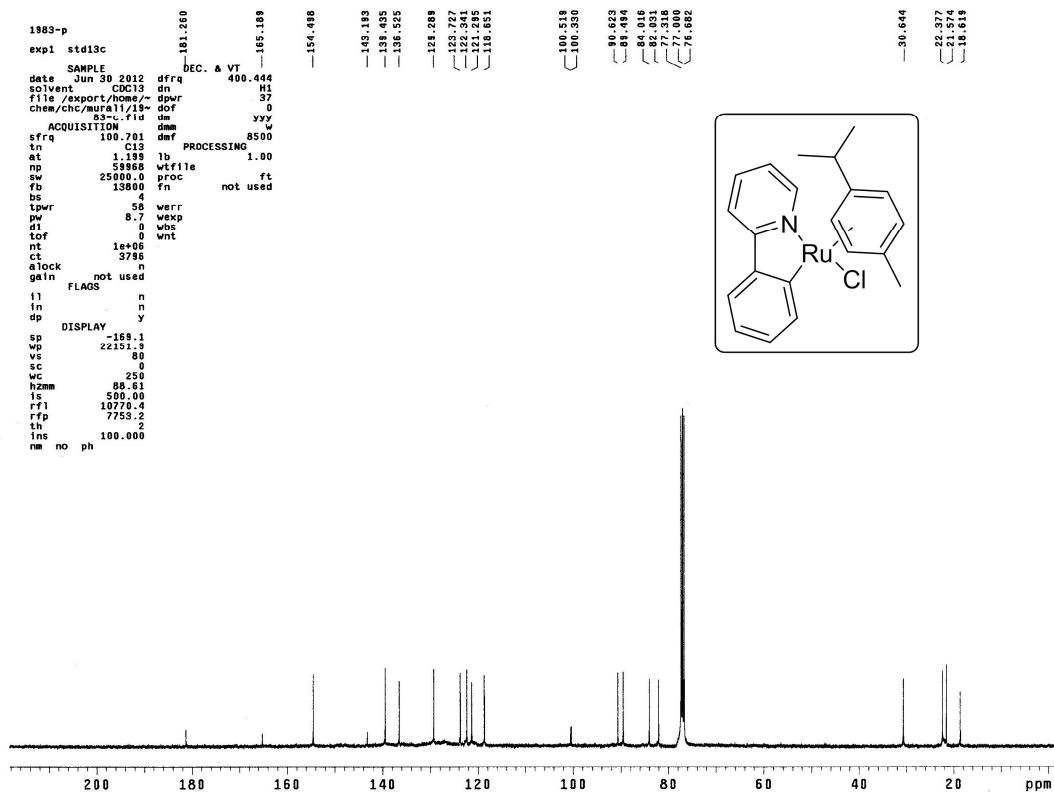
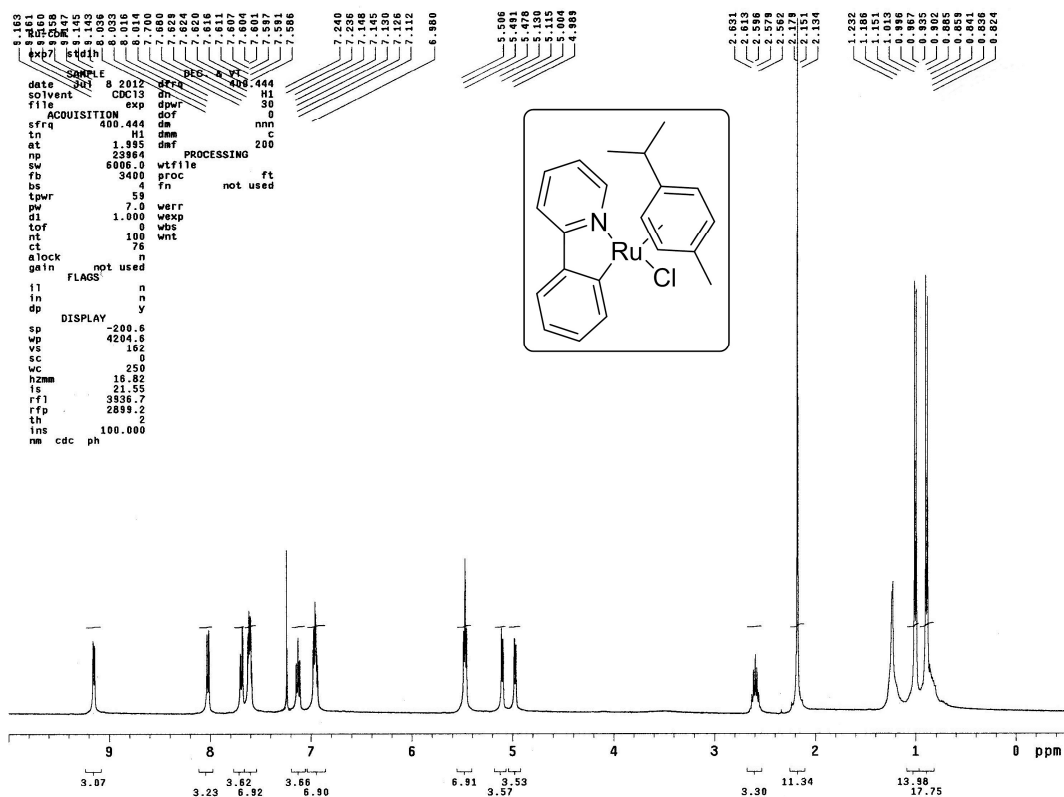


$^1\text{H}$  and  $^{13}\text{C}$  NMR spectra of compound **3v**.



# <sup>1</sup>H and <sup>13</sup>C NMR spectra of complex A-OAc



$^1\text{H}$  and  $^{13}\text{C}$  NMR spectra of complex **A-Cl**

ORTEP diagram of compound **3r**:

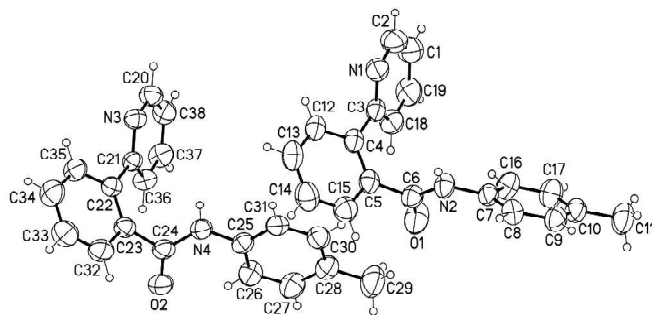
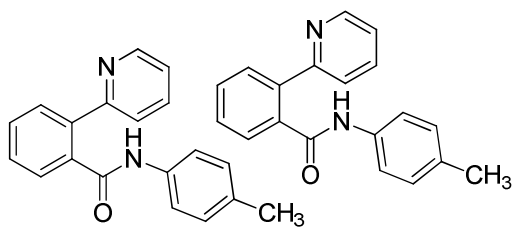


Table 1. Crystal data and structure refinement for **3r**.

Identification code	110406_0m	
Empirical formula	C38 H32 N4 O2	
Formula weight	576.68	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P -1	
Unit cell dimensions	a = 9.1206(3) Å	$\alpha = 100.4450(10)^\circ$ .
	b = 10.6882(3) Å	$\beta = 99.3990(10)^\circ$ .
	c = 16.4955(5) Å	$\gamma = 93.992(2)^\circ$ .
Volume	1551.97(8) Å <sup>3</sup>	
Z	2	
Density (calculated)	1.234 Mg/m <sup>3</sup>	
Absorption coefficient	0.077 mm <sup>-1</sup>	
F(000)	608	
Crystal size	0.30 x 0.30 x 0.17 mm <sup>3</sup>	
Theta range for data collection	1.28 to 26.41°.	
Index ranges	-11 ≤ h ≤ 11, -13 ≤ k ≤ 13, -20 ≤ l ≤ 20	
Reflections collected	23578	
Independent reflections	6329 [R(int) = 0.0292]	
Completeness to theta = 26.41°	99.2 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9486 and 0.9165	
Refinement method	Full-matrix least-squares on F <sup>2</sup>	
Data / restraints / parameters	6329 / 0 / 399	
Goodness-of-fit on F <sup>2</sup>	1.066	
Final R indices [I > 2σ(I)]	R1 = 0.0466, wR2 = 0.1506	
R indices (all data)	R1 = 0.0776, wR2 = 0.1968	
Largest diff. peak and hole	0.500 and -0.344 e.Å <sup>-3</sup>	