

## Supporting Information

# Stabilized Pyrrolyl Iodonium Salts and Metal-Free Oxidative Cross-Coupling

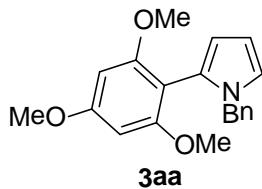
*Koji Morimoto,<sup>a</sup> Yusuke Ohnishi,<sup>a</sup> Daichi Koseki,<sup>a</sup> Akira Nakamura,<sup>b</sup> Toshifumi Dohi,<sup>a</sup> Yasuyuki Kita*

<sup>a</sup> Ritsumeikan University College of Pharmaceutical Sciences 1-1-1 Nojihigashi, Kusatsu, Shiga 525-8577, Japan

<sup>b</sup> Ritsumeikan University Research Organization of Science and Technology, 1-1-1 Nojihigashi, Kusatsu, Shiga, 525-8577, Japan

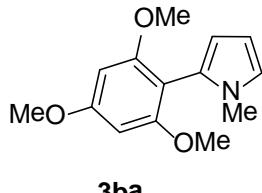
## Characterization data of the products 3

### 1-Benzyl-2-[(2,4,6-trimethoxy)phenyl]-1*H*-pyrrole (3aa)



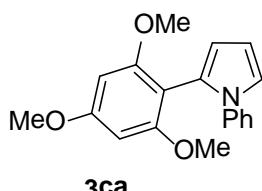
White solid; mp: 87-89 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  3.54 (s, 6H), 3.75 (s, 3H), 4.72 (s, 2H), 6.16 (dd, 1H,  $J$  = 3.4, 1.0 Hz), 6.05 (s, 2H), 6.22 (d, 1H,  $J$  = 2.4 Hz), 6.65 (t, 1H,  $J$  = 2.0 Hz), 6.93 (d, 2H,  $J$  = 6.8 Hz), 7.08-7.16 (m, 3H) ppm;  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  50.6, 55.3, 55.6, 90.4, 103.3, 107.9, 109.3, 120.7, 125.5, 126.8, 127.1, 128.1, 138.9, 160.1, 161.4 ppm; IR (KBr): 3942 w, 3053 w, 2987 w, 2945 w, 2685 w, 2305 w, 1610 w, 1421 w, 1263 s, 1155 w, 1126 w, 895 w, 814 w, 748 s, 706 m  $\text{cm}^{-1}$ ; HRFABMS calcd for  $\text{C}_{20}\text{H}_{21}\text{NO}_3$  [M] $^+$  323.1521, found 323.1537.

### 1-Methyl-2-[(2,4,6-trimethoxy)phenyl]-1*H*-pyrrole (3ba)



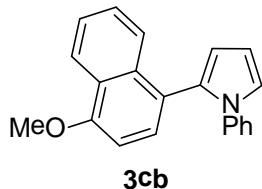
White solid; mp: 87-89 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  3.37 (s, 3H), 3.72 (s, 6H), 3.84 (s, 3H), 6.04 (dd, 1H,  $J$  = 3.7, 1.8 Hz), 6.18 (s, 2H), 6.21 (t, 1H,  $J$  = 3.0 Hz), 6.70 (t, 1H,  $J$  = 2.0 Hz) ppm;  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  34.0, 55.3, 55.8, 90.5, 103.5, 107.1, 109.4, 121.4, 125.2, 159.9, 161.2 ppm; IR (KBr): 2930 m, 1611 s, 1583 s, 1552 m, 1496 w, 1454 m, 1413 m, 1334 m, 1307 w, 1224 m, 1203 s, 1155 s, 1124 s, 1030 w, 812 w, 711 m  $\text{cm}^{-1}$ ; HRFABMS calcd for  $\text{C}_{14}\text{H}_{17}\text{NO}_3$  [M] $^+$  247.1208, found 247.1204.

### 1-Phenyl-2-[(2,4,6-trimethoxy)phenyl]-1*H*-pyrrole (3ca)



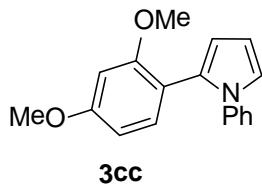
White solid; mp: 87-89 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  3.49 (s, 6H), 3.78 (s, 3H), 6.02 (s, 2H), 6.26-6.27 (m, 1H), 6.39 (t, 1H,  $J$  = 2.8 Hz), 6.97 (t, 1H,  $J$  = 2.8 Hz), 7.07-7.13 (m, 3H), 7.21 (t, 2H,  $J$  = 7.6 Hz) ppm;  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  55.1, 55.4, 90.3, 103.9, 108.8, 111.6, 121.4, 124.1, 124.9, 125.6, 128.2, 141.3, 159.3, 161.1 ppm; IR (KBr): 3944 w, 3053 w, 2986 w, 2941 w, 2305 w, 1502 w, 1417 w, 1261 s, 1226 w, 1157 w, 1128 m, 895 w, 814 w, 763 s, 748 s, 704 m  $\text{cm}^{-1}$ ; HRFABMS calcd for  $\text{C}_{19}\text{H}_{19}\text{NO}_3$  [M] $^+$  309.1365, found 309.1357.

**2-[(4-Methoxy)naphthalen-1-yl]-1-phenyl-1*H*-pyrrole (3cb)**



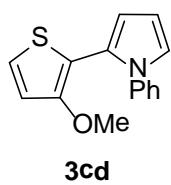
Brown oil;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  3.96 (s, 3H), 6.43 (q, 1H,  $J = 1.7$  Hz), 6.45 (t, 1H,  $J = 3.2$  Hz), 6.69 (d, 1H,  $J = 7.8$  Hz), 7.03-7.16 (m, 5H), 7.35-7.42 (m, 3H), 7.87-7.90 (m, 2H), 8.22-8.26 (m, 1H) ppm;  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  55.4, 103.2, 108.8, 112.4, 121.8, 122.5, 124.7, 125.0, 125.9, 126.0, 126.5, 128.7, 129.1 ppm; IR (KBr): 2925, 2853, 2362, 2252, 1723, 1586, 1498, 1459, 1384, 1237, 1096, 913, 744  $\text{cm}^{-1}$ ; MALDI-TOFMS calcd for  $\text{C}_{21}\text{H}_{17}\text{NO} [\text{M}]^+$  299.1310, found 299.1305.

**2-[(2,4-Dimethoxy)phenyl]-1-phenyl-1*H*-pyrrole (3cc)**



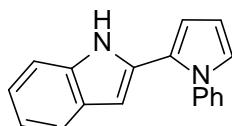
Colorless oil;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  3.19 (s, 3H), 3.69 (s, 3H), 6.14-6.15 (m, 1H), 6.20-6.23 (m, 2H), 6.38 (dd, 1H,  $J = 8.3, 2.4$  Hz), 6.86-6.87 (m, 1H), 6.99-7.02 (m, 2H), 7.08-7.11 (m, 2H), 7.16-7.19 (m, 2H) ppm;  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  54.7, 55.3, 98.6, 104.2, 108.8, 110.7, 115.5, 122.3, 124.1, 125.7, 128.5, 130.3, 132.0, 141.5, 157.6, 160.5 ppm; IR (KBr): 2932, 1614, 1502, 1465, 1305, 1209, 1161, 1034, 765  $\text{cm}^{-1}$ ; HRFABMS calcd for  $\text{C}_{18}\text{H}_{17}\text{NO}_2 [\text{M}]^+$  279.1259, found 279.1254.

**2-[(3-Methoxy)thiophen-2-yl]-1-phenyl-1*H*-pyrrole (3cd)**



Colorless oil;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  3.47 (s, 3H), 6.36 (t, 1H,  $J = 3.3$  Hz), 6.48-6.50 (m, 1H), 6.72 (d, 1H,  $J = 5.7$  Hz), 6.94-6.95 (m, 1H), 7.08 (d, 1H,  $J = 5.4$  Hz), 7.22-7.35 (m, 5H) ppm;  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  58.2, 109.1, 111.5, 112.2, 116.3, 123.2, 123.3, 124.0, 125.3, 126.6, 128.6, 140.6, 154.0 ppm; IR (KBr): 3105, 3051, 2958, 2931, 2850, 1597, 1523, 1500, 1427, 1379, 1321, 1259, 1236, 1103, 1070, 790, 763, 750, 717, 696  $\text{cm}^{-1}$ . MALDI-TOFMS calcd for  $\text{C}_{15}\text{H}_{13}\text{NOS} [\text{M}]^+$  255.0718, found 255.0713.

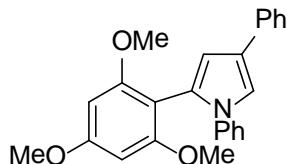
**2-(1-Phenyl-1*H*-pyrrol-2-yl)-1*H*-indole (3ce)**



**3ce**

White solid; mp: 144-145 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 6.41 (t, 1H, *J* = 2.8 Hz), 6.52 (d, 1H, *J* = 3.6 Hz), 6.60 (d, 1H, *J* = 2.4 Hz), 6.94 (s, 1H), 7.07 (td, 1H, *J* = 8.0, 1.2 Hz), 7.16 (td, 1H, *J* = 6.8, 1.2 Hz), 7.20-7.27 (m, 5H), 7.31 (d, 1H, *J* = 8.0 Hz), 7.63 (d, 1H, *J* = 8.0 Hz), 7.99 (s, 1H) ppm; <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 109.1, 109.3, 110.0, 110.9, 120.0, 120.2, 122.2, 122.7, 122.8, 125.8, 126.5, 126.6, 127.3, 128.8, 135.6, 140.8 ppm; IR (KBr): 3404, 3058, 2926, 1597, 1498, 1456, 1434, 1331, 1242, 1199, 1097, 81, 791, 741, 697 cm<sup>-1</sup>; MALDI-TOFMS calcd for C<sub>18</sub>H<sub>14</sub>N<sub>2</sub> [M+H]<sup>+</sup> 258.1157, found 258.1152.

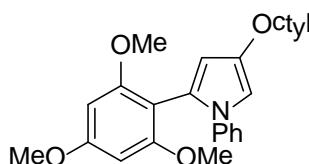
**1,4-Diphenyl-2-[(2,4,6-trimethoxy)phenyl]-1*H*-pyrrole (3da)**



**3da**

White solid; mp: 59-61 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 3.51 (s, 6H), 3.79 (s, 3H), 6.03 (s, 2H), 6.61 (s, 1H), 7.12-7.32 (m, 9H), 7.58 (dd, 2H, *J* = 8.4, 1.2 Hz) ppm; <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 55.2, 55.5, 90.5, 103.7, 110.1, 118.2, 124.0, 125.0, 125.1, 125.2, 125.8, 125.9, 128.3, 128.4, 135.8, 141.2, 159.3, 161.3 ppm; IR (KBr): 2936, 2837, 1583, 1500, 1454, 1415, 1336, 1227, 1204, 1155, 1126, 911, 812, 748, 695 cm<sup>-1</sup>; MALDI-TOFMS calcd for C<sub>25</sub>H<sub>24</sub>NO<sub>3</sub> [M+H]<sup>+</sup> 386.1756, found 386.1751.

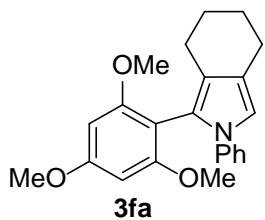
**4-Octyl-1-phenyl-2-[(2,4,6-trimethoxy)phenyl]-1*H*-pyrrole (3ea)**



**3ea**

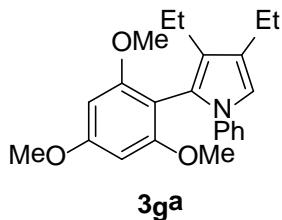
Brown oil; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 0.88 (m, 3H), 1.22-1.45 (m, 10H), 1.69-1.65 (m, 2H), 2.55 (t, 2H, *J* = 7.8 Hz), 3.48 (s, 6H), 3.78 (s, 3H), 6.02 (s, 2H), 6.16 (s, 1H), 6.76 (s, 1H), 7.11-7.05 (m, 3H), 7.19 (t, 2H, *J* = 7.3Hz) ppm; <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 14.1, 22.7, 27.3, 29.3, 29.6, 29.9, 30.4, 31.9, 55.2, 55.4, 90.2, 90.4, 104.3, 112.5, 118.6, 123.7, 124.0, 125.1, 128.2, 141.6, 159.1, 160.9 ppm; IR (KBr): 2999, 2925, 2853, 2359, 1610, 1584, 1500, 1415, 1127, 812, 765, 695 cm<sup>-1</sup>; MALDI-TOFMS calcd for C<sub>27</sub>H<sub>36</sub>NO<sub>3</sub> [M+H]<sup>+</sup> 422.1365, found 422.2690.

**2-Phenyl-1-[(2,4,6-trimethoxy)phenyl]-4,5,6,7-tetrahydro-2*H*-isoindole (3fa)**



White solid; mp: 117-119 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  1.72-1.79 (m, 4H), 2.39 (t, 2H,  $J = 5.8$  Hz), 2.48 (t, 2H,  $J = 5.9$  Hz), 3.48 (s, 6H), 3.78 (s, 3H), 6.02 (s, 2H), 6.71 (s, 1H), 7.02-7.07 (m, 3H), 7.17 (t, 2H,  $J = 7.6$  Hz) ppm;  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  22.1, 22.2, 23.9, 24.1, 55.2, 55.4, 90.4, 103.4, 116.9, 119.7, 120.1, 121.7, 123.6, 124.9, 128.2, 141.7, 159.2, 161.0 ppm; IR (KBr): 2998, 2926, 2837, 1585, 1502, 1335, 1204, 1127, 813, 764, 696  $\text{cm}^{-1}$ ; MALDI-TOFMS calcd for  $\text{C}_{23}\text{H}_{25}\text{NO}_3$   $[\text{M}+\text{H}]^+$  363.1834, found 363.1830.

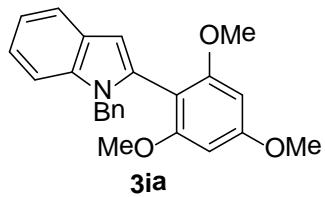
**3,4-Diethyl-1-phenyl-2-[(2,4,6-trimethoxy)phenyl]-1*H*-pyrrole (3ga)**



**3ga**

White solid; mp: 97-99 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  1.02 (t, 3H,  $J = 7.5$  Hz), 1.28 (t, 3H,  $J = 7.5$  Hz), 2.30 (q, 2H,  $J = 7.5$  Hz), 2.59 (q, 2H,  $J = 7.5$  Hz), 3.50 (s, 6H), 3.78 (s, 3H), 6.02 (s, 2H), 6.74 (s, 1H), 7.01-7.06 (m, 3H), 7.14 (t, 2H,  $J = 7.8$  Hz) ppm;  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  12.4, 14.7, 18.6, 18.8, 55.2, 55.4, 90.3, 103.7, 117.8, 121.3, 123.8, 124.8, 125.0, 125.1, 128.1, 141.4, 159.7, 161.2 ppm; IR (KBr): 2960, 2927, 2852, 1583, 1501, 1462, 1412, 1204, 1128, 764, 697  $\text{cm}^{-1}$ ; MALDI-TOFMS calcd for  $\text{C}_{23}\text{H}_{28}\text{NO}_3$   $[\text{M}+\text{H}]^+$  366.2069, found 366.2064.

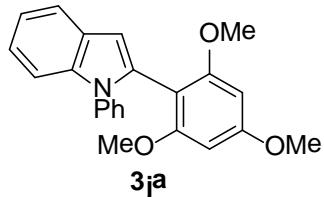
**1-Benzyl-2-[(2,4,6-trimethoxy)phenyl]-1*H*-indole (3ha)**



Yellow solid; mp: 153-155 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  3.58 (s, 6H), 3.84 (s, 3H), 5.07 (s, 2H), 6.15 (s, 2H), 6.49 (s, 1H), 6.99 (d, 2H,  $J = 7.8$  Hz), 7.06-7.09 (m, 2H), 7.13-7.19 (m, 4H), 7.63-7.65 (m, 1H) ppm;  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  47.5, 55.4, 55.6, 90.4, 102.8, 103.1, 110.1, 119.1, 120.4, 120.8, 126.6, 128.1,

128.5, 133.4, 136.8, 138.5, 160.1, 162.1 ppm ; IR (KBr): 3003, 2925, 2850, 1611, 1583, 1461, 1415, 1338, 1226, 1126, 812, 731 cm<sup>-1</sup>; MALDI-TOFMS calcd for C<sub>24</sub>H<sub>24</sub>NO<sub>3</sub> [M+H]<sup>+</sup> 374.1756, found 374.1751.

### **1-Phenyl-2-[(2,4,6-trimethoxy)phenyl]-1*H*-indole (3ia)**

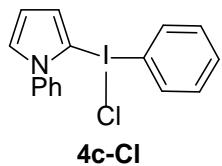


White solid; mp: 168-170 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 3.49 (s, 6H), 3.70 (s, 3H), 5.97 (s, 2H), 6.44 (s, 1H), 7.02-7.03 (m, 2H), 7.11-7.27 (m, 4H), 7.22-7.26 (m, 3H), 7.52-7.54 (m, 1H) ppm; <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 55.3, 55.5, 90.3, 103.5, 104.7, 110.3, 119.8, 120.5, 121.3, 126.3, 126.8, 128.3, 128.5, 133.1, 137.4, 138.8, 159.6, 161.7 ppm; IR (KBr): 3002, 2937, 2838, 2364, 1612, 1585, 1499, 1454, 1313, 1204, 1128, 1039, 741 cm<sup>-1</sup>; MALDI-TOFMS calcd for C<sub>23</sub>H<sub>22</sub>NO<sub>3</sub> [M+H]<sup>+</sup> 360.1600, found 360.1594.

### **Synthesis of iodonium salt 4c-X (Scheme 3, Eq. 1)**

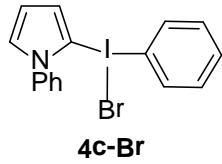
Iodine(III) reagent (1 equiv.) and TMSX (1 equiv.) were added to a solution of pyrrole **1c** (1 equiv.) in HFIP (0.1 M) at room temperature and the mixture was stirred under an inert atmosphere for 1 h. Then H<sub>2</sub>O (20 mL) was added and then stirred for 3 min. The aqueous phase was extracted with CH<sub>2</sub>Cl<sub>2</sub> and the combined organic phases were dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>. Removal of the drying agent by filtration afforded a solution of pure iodonium(III) salts **4c-X**.

### **Phenyl-(1-phenyl-1*H*-pyrrole-2-yl)-iodonium chloride (4c-Cl)**



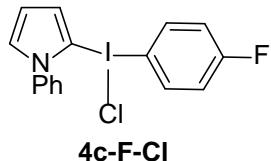
White solid; mp: 191-193 °C ; <sup>1</sup>H NMR (400 MHz, DMSO-d<sup>6</sup>): δ 6.46 (t, 1H, J = 2.9 Hz), 7.24-7.25 (m, 1H), 7.35-7.38 (m, 5H), 7.49-7.61 (m, 6H) ppm; <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 100.7, 112.3, 122.2, 123.6, 126.2, 128.5, 128.7, 129.5, 130.7, 131.0, 133.1, 138.8 ppm; IR (KBr): 3087, 1595, 1496, 1433, 1379, 1319, 1275, 1072, 1038, 992, 747 cm<sup>-1</sup>; MALDI-TOFMS calcd for C<sub>16</sub>H<sub>13</sub>IN [M-Cl]<sup>+</sup> 346.19, found 346.02.

### **Phenyl-(1-phenyl-1*H*-pyrrole-2-yl)-iodonium bromide (4c-Br)**



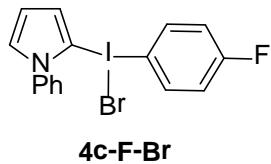
White solid; mp: 182-184 °C; <sup>1</sup>H NMR (400 MHz, DMSO-d<sup>6</sup>): δ 6.49 (t, 1H, *J* = 3.4 Hz), 7.30 (dd, 1H, *J* = 3.9, 1.0 Hz), 7.34-7.41 (m, 5H), 7.53-7.62 (m, 6H) ppm; <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 98.6, 112.4, 120.1, 121.2, 123.9, 126.3, 128.9, 129.6, 131.0, 131.2, 133.3, 138.7 ppm; IR (KBr): 3087, 1595, 1565, 1496, 1432, 1320, 1276, 992, 749 cm<sup>-1</sup>; MALDI-TOFMS calcd for C<sub>16</sub>H<sub>13</sub>IN [M-Br]<sup>+</sup> 346.19, found 346.17.

#### **4-Fruolophenyl -(1-phenyl-1*H*-pyrrole-2-yl)-iodonium chloride (4c-F-Cl)**



White solid; mp: 204-206 °C; <sup>1</sup>H NMR (400 MHz, DMSO-d<sup>6</sup>): δ 6.48 (t, 1H, *J* = 2.9 Hz), 7.27 (t, 2H, *J* = 8.8 Hz), 7.29-7.30 (m, 1H), 7.34-7.36 (m, 2H), 7.39 (dd, 1H, *J* = 2.9, 1.9 Hz), 7.55-7.58 (m, 3H), 7.65-7.57 (m, 2H) ppm; <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 101.5, 112.3, 116.8, 116.9, 118.2, 118.4, 123.5, 126.2, 128.4, 128.8, 129.6, 135.7, 135.8, 138.8, 161.7, 164.2 ppm; IR (KBr): 3090, 3050, 1891, 1763, 1634, 1594, 1753, 1494, 1480, 1433, 1376, 1319, 1229, 1161, 1035, 1002, 942, 923, 824, 772, 727, 700, 503 cm<sup>-1</sup>; MALDI-TOFMS calcd for C<sub>16</sub>H<sub>12</sub>FIN [M-Cl]<sup>+</sup> 363.99, found 363.98.

#### **4-Fruolophenyl -(1-phenyl-1*H*-pyrrole-2-yl)-iodonium bromide (4c-F-Br)**



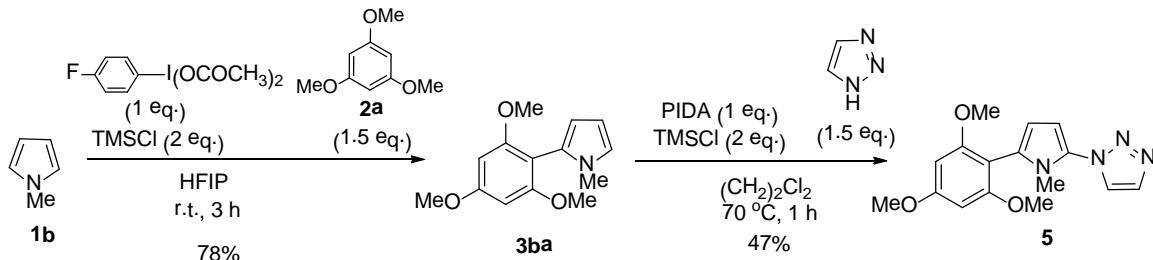
White solid; mp: 198-200 °C; <sup>1</sup>H NMR (400 MHz, DMSO-d<sup>6</sup>): δ 6.49 (t, 1H, *J* = 3.8 Hz), 7.28 (t, 2H, *J* = 8.8 Hz), 7.31-7.35 (m, 3H), 7.40-7.41 (m, 1H), 7.57 (m, 3H), 7.64-7.68 (m, 2H) ppm; <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 24.1, 100.9, 112.2, 116.5, 118.2, 118.4, 120.0, 123.4, 126.2, 128.3, 128.8, 129.6, 129.9, 135.9, 136.0, 138.8, 161.7, 164.2, 174.5 ppm; IR (KBr): 3089, 3048, 1573, 1496, 1480, 1433, 1320, 1230, 1160, 1036, 825, 750 cm<sup>-1</sup>; MALDI-TOFMS calcd for C<sub>16</sub>H<sub>12</sub>FIN [M-Cl]<sup>+</sup> 363.99, found 363.98.

#### **Reaction of iodonium(III) salt 4c-X with 1,3,5-trimethoxybenzene 2a (Scheme 3, Eq. 2)**

1,3,5-Trimethoxybenzene **2a** (1.5 equiv.) and TMSX (1 equiv.) were added to a solution of pyrrole iodonium salt **4c-X** (1 equiv.) in HFIP (0.1 M) at room temperature, and the mixture was stirred under an inert atmosphere for 1 h. Saturated aqueous sodium hydrogen carbonate was added to the mixture when the reaction completed. The aqueous phase was extracted with CH<sub>2</sub>Cl<sub>2</sub>. The extract was dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and then evaporated to dryness. The crude residue was purified by column chromatography on

silica-gel (eluent: *n*-hexane/ethylacetate) to give the pure product **3ca**. The structure of the resulting product **3ca** was confirmed by comparing its NMR spectra with authentic sample (see Table 2).

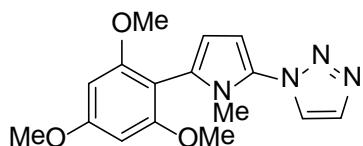
### General procedure for synthesis pyrrole trimer **5**



To a stirred solution of pyrrole **1b** (0.4 mmol 1 equiv.) in 1,1,1,3,3,3-hexafluoroisopropanol (HFIP, 0.1 M), 4-F-PIDA (0.4 mmol 1 equiv.) was added at room temperature. After 15 min, to the mixture, arene **2a** (0.6 mmol 1.5 equiv.) and TMSCl (0.8 mmol 2 equiv.) was added and then stirred for 3 h under the same conditions, while the reaction progress was checked by TLC. Saturated aqueous sodium hydrogen carbonate was added to the mixture when the reaction completed. The aqueous phase was extracted with  $\text{CH}_2\text{Cl}_2$ . The extract was dried over anhydrous  $\text{Na}_2\text{SO}_4$  and then evaporated to dryness. The crude residue was purified by column chromatography on silica-gel (eluent: *n*-hexane/ethylacetate) to give the pure **3ba**.

Next, PIDA (0.4 mmol 1 equiv.) and TMSCl (0.8 mmol 2 equiv.) were added to a solution of pyrrole dimer **3ba** (0.4 mmol 1 equiv.) in  $(\text{CH}_2)_2\text{Cl}_2$  (0.1 M) at ambient temperature and the mixture was stirred under an inert atmosphere at 70 °C . After 1 h, saturated aqueous sodium hydrogen carbonate was added to the mixture at room temperature when the reaction completed. The aqueous phase was extracted with  $\text{CH}_2\text{Cl}_2$ . The extract was dried over anhydrous  $\text{Na}_2\text{SO}_4$  and then evaporated to dryness. The crude residue was purified by column chromatography on silica-gel (eluent: *n*-hexane/ethylacetate) to give the pure **5**.

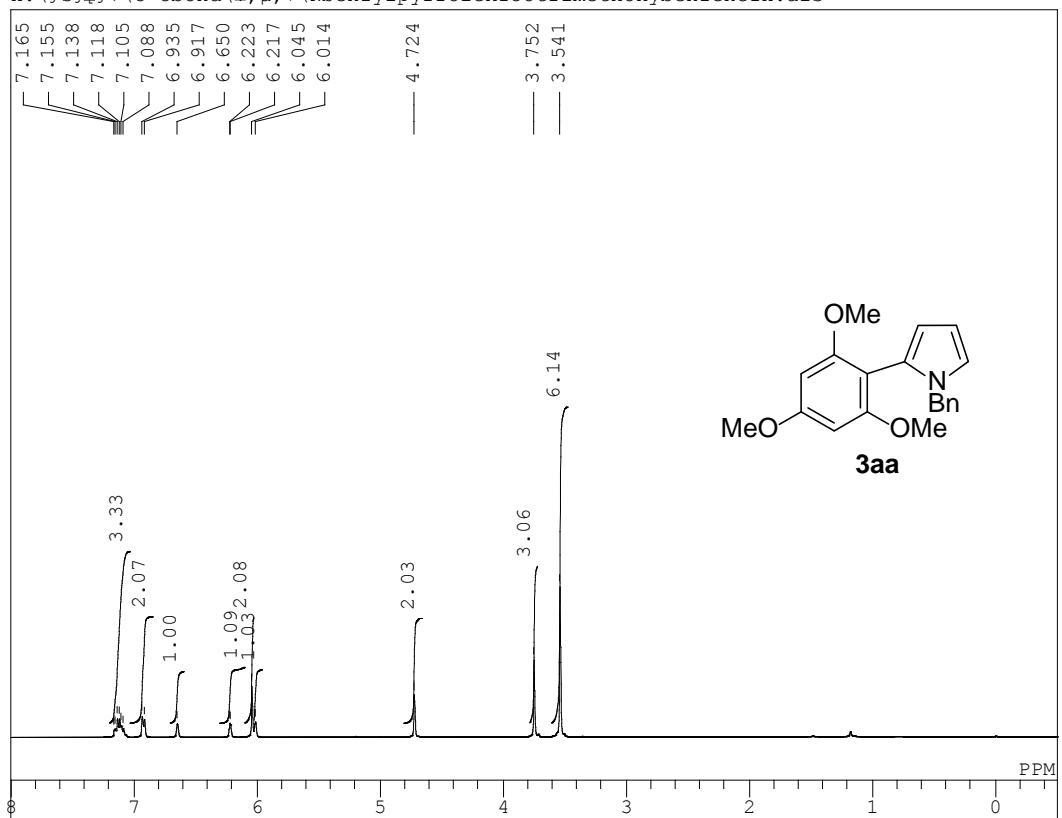
**1-(1-methyl-5-(2,4,6-trimethoxyphenyl)-1*H*-pyrrol-2-yl)-1*H*-1,2,3-triazole (5)**



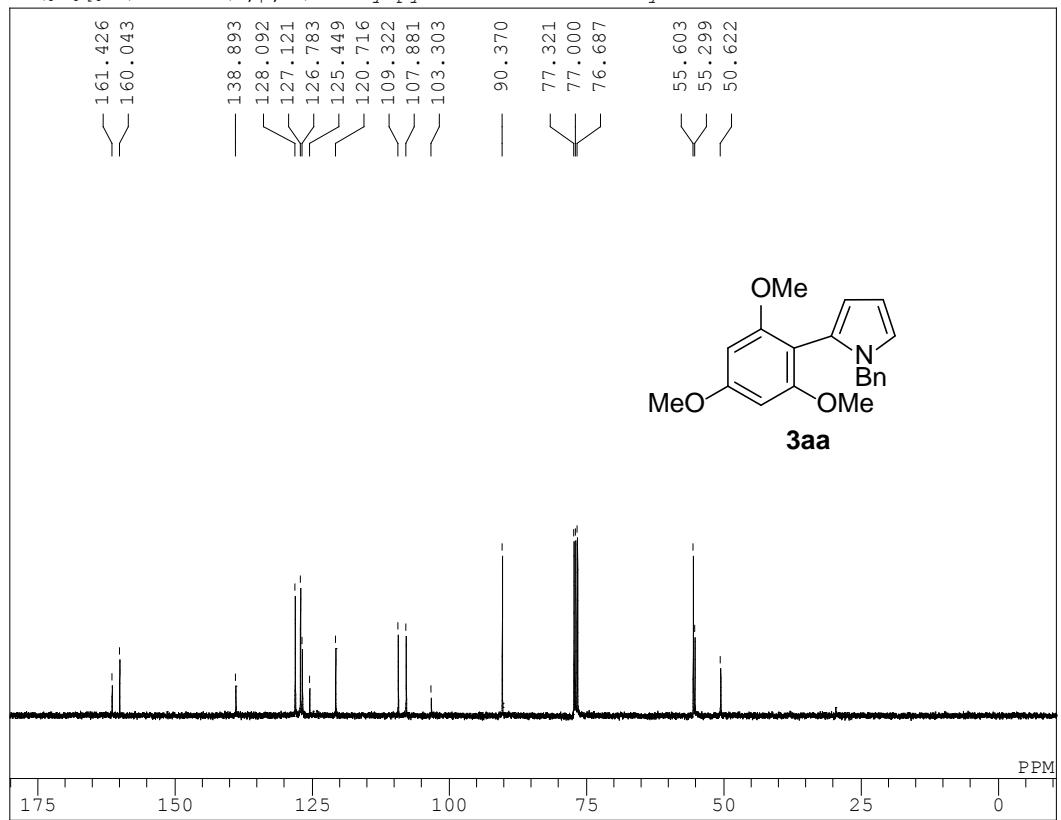
**5**

Brown oil;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  3.18 (s, 3H), 3.75 (s, 6H), 3.85 (s, 3H), 6.12 (d, 1H,  $J$  = 3.4 Hz), 6.19 (s, 2H), 6.34 (d, 1H,  $J$  = 3.9 Hz), 7.79-7.81 (m, 2H) ppm;  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  31.0, 55.4, 55.8, 90.5, 102.3, 104.1, 108.4, 124.7, 126.2, 126.6, 132.4, 133.0, 160.0, 161.8 ppm; IR (KBr): 3122, 2922, 2850, 1710, 1610, 1584, 1537, 1464, 1415, 1337, 1276, 1228, 1205, 1155, 1125, 1035, 1011, 947, 816, 750, 647  $\text{cm}^{-1}$ ; MALDI-TOFMS calcd for  $\text{C}_{16}\text{H}_{19}\text{N}_4\text{O}_3$   $[\text{M}+\text{H}]^+$  315.1457, found 315.1452.

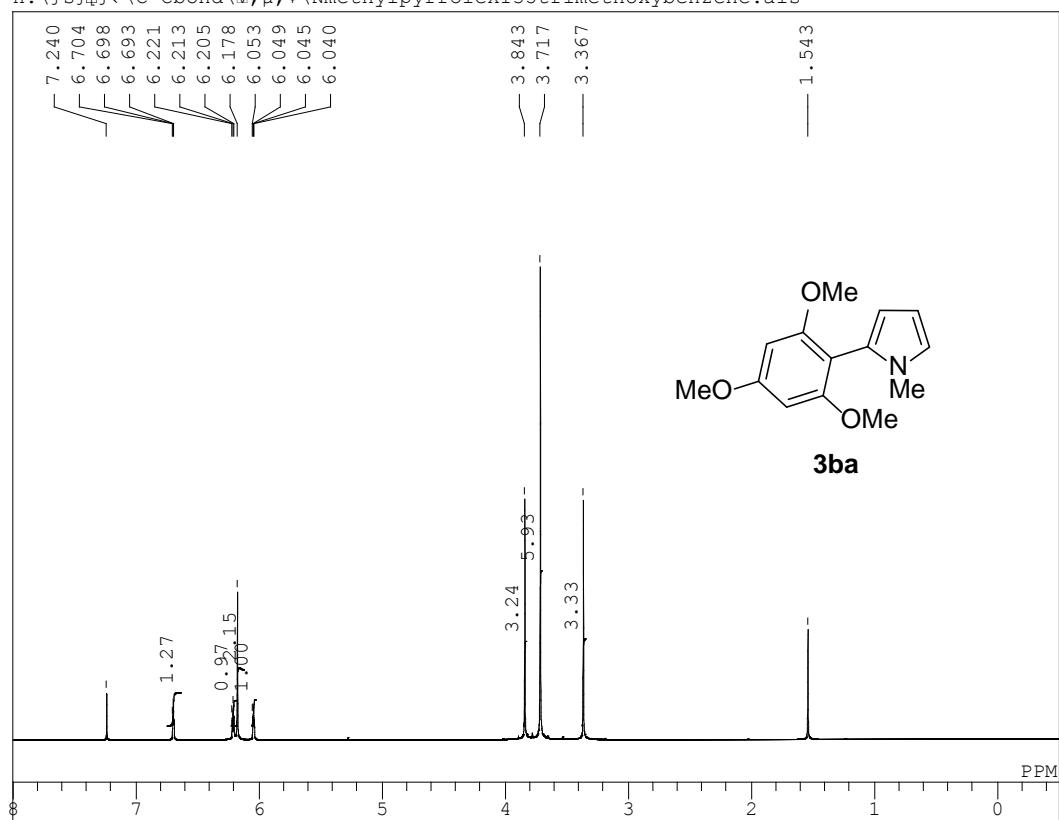
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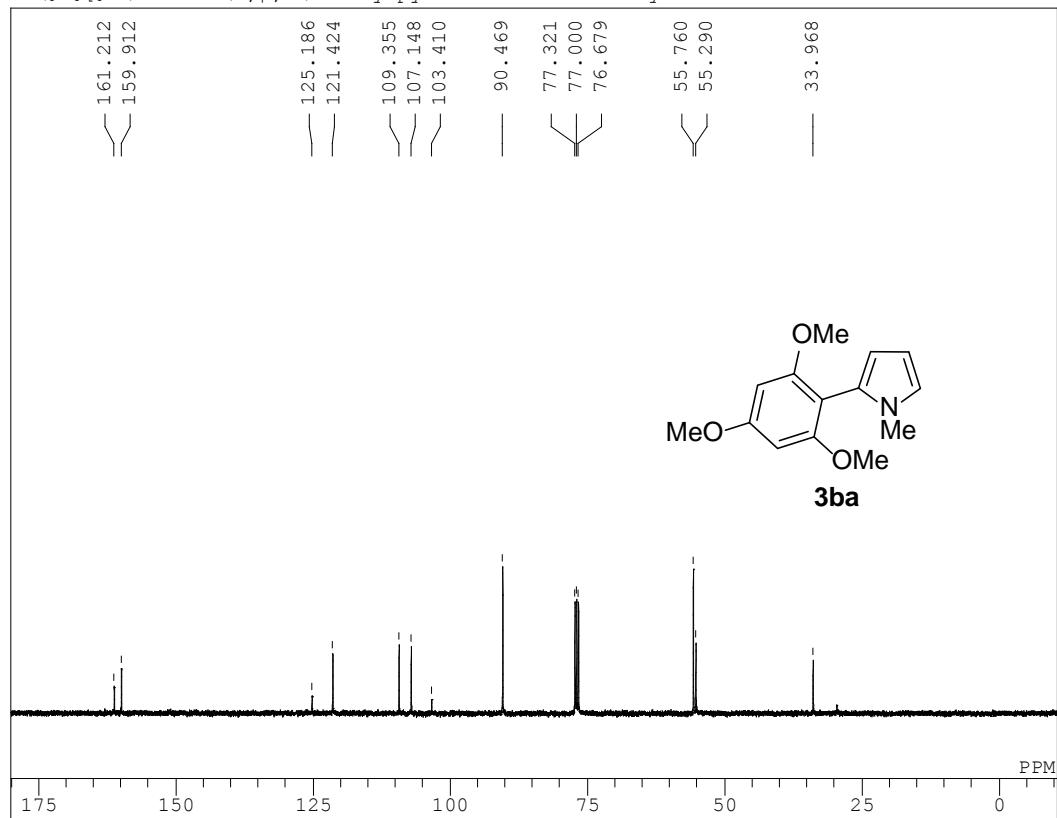
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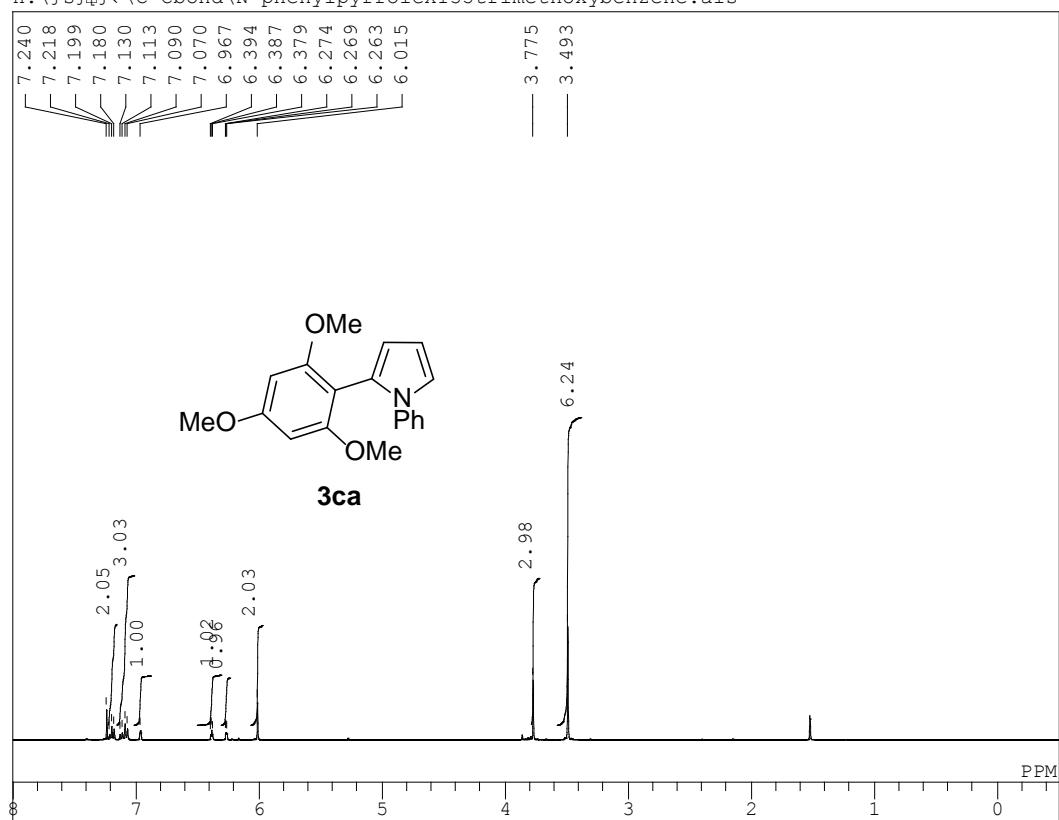
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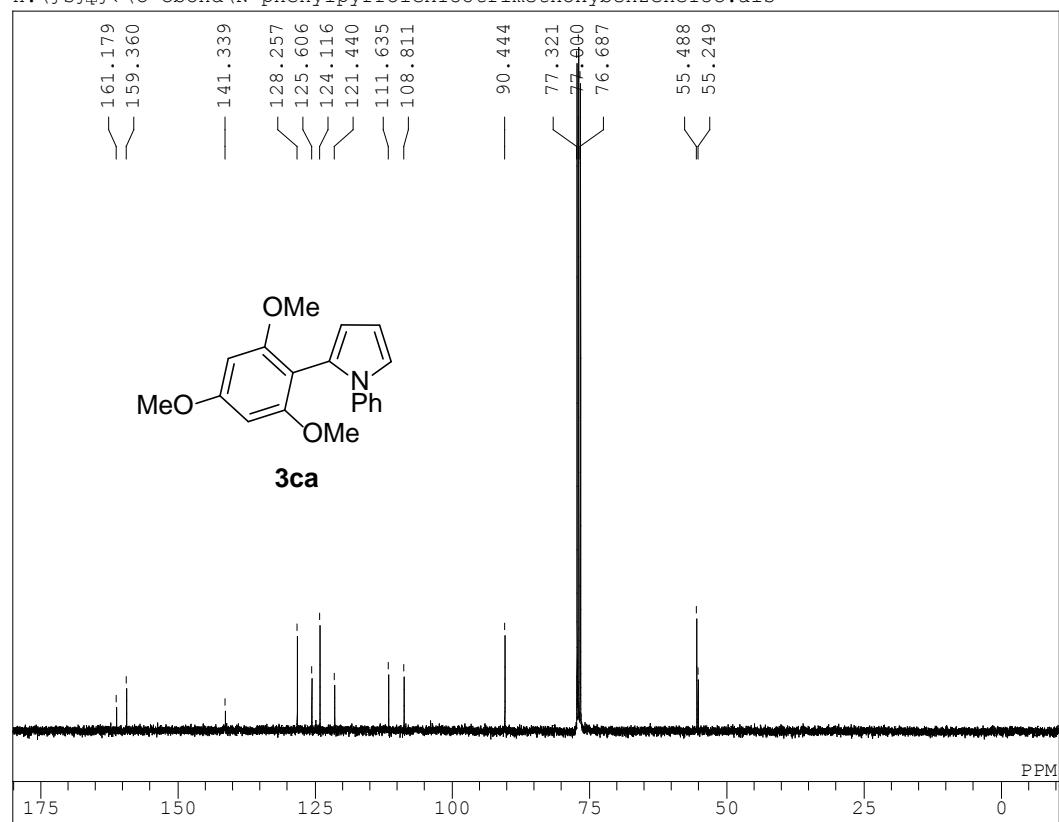
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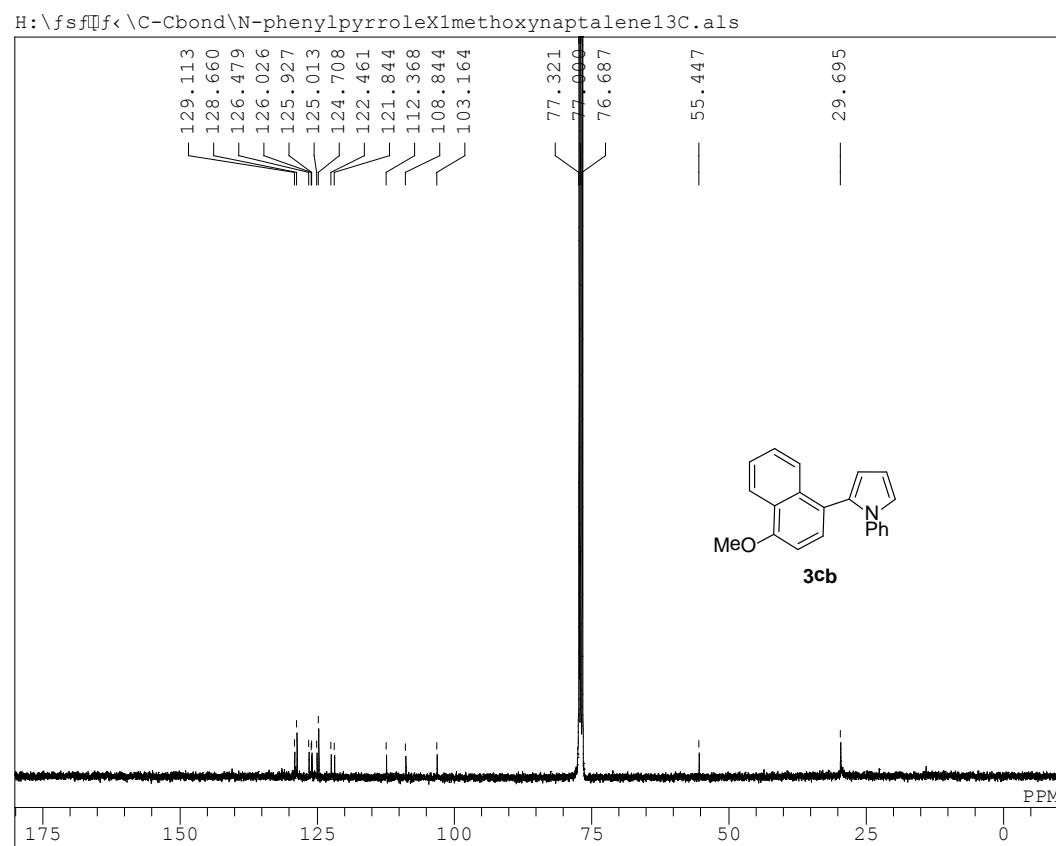
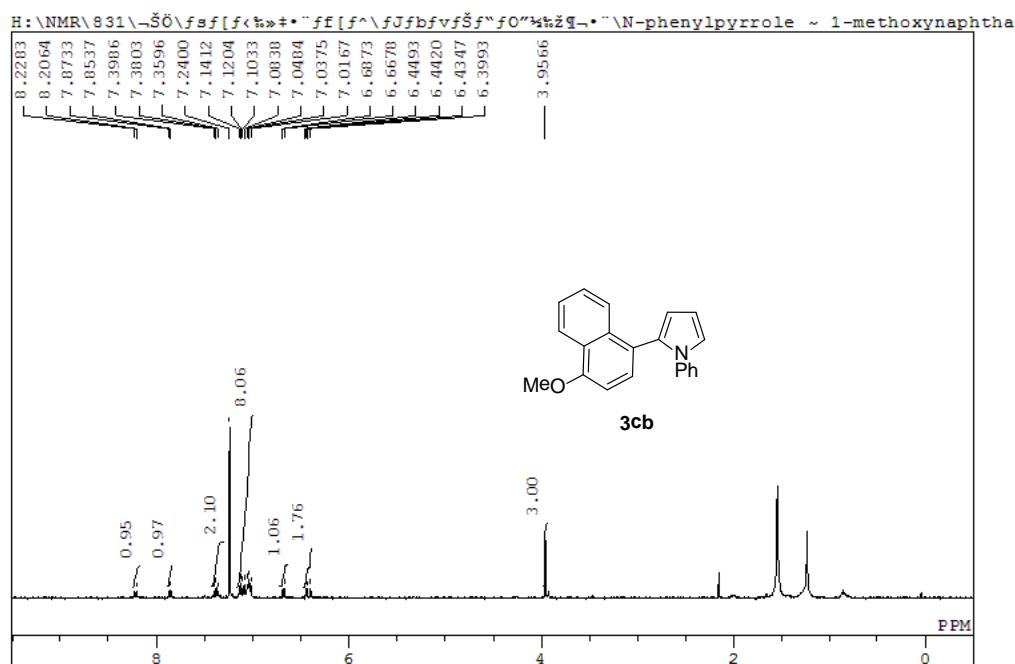


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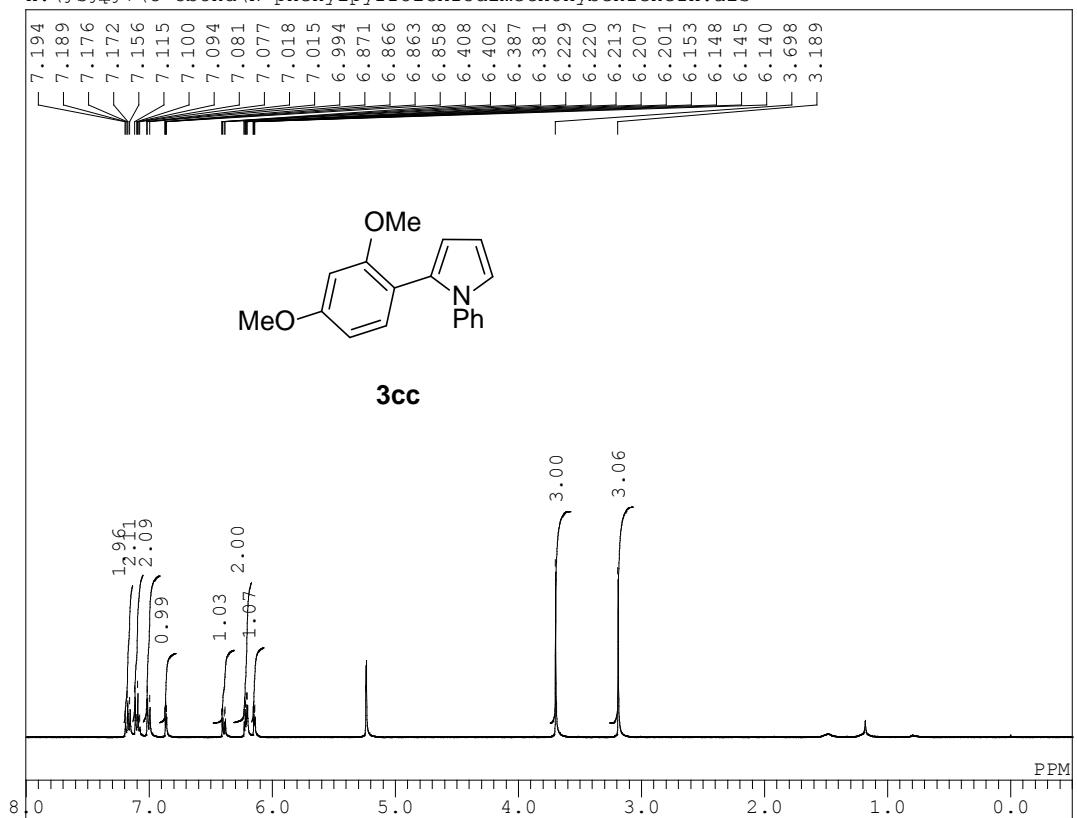


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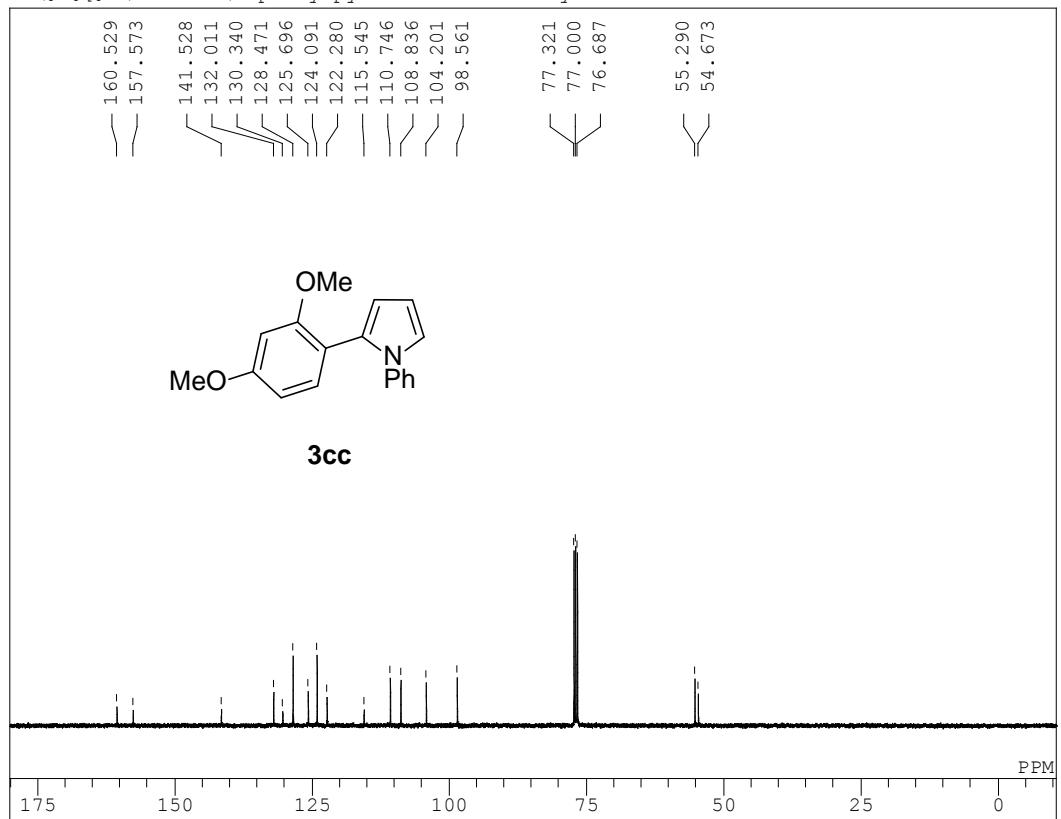


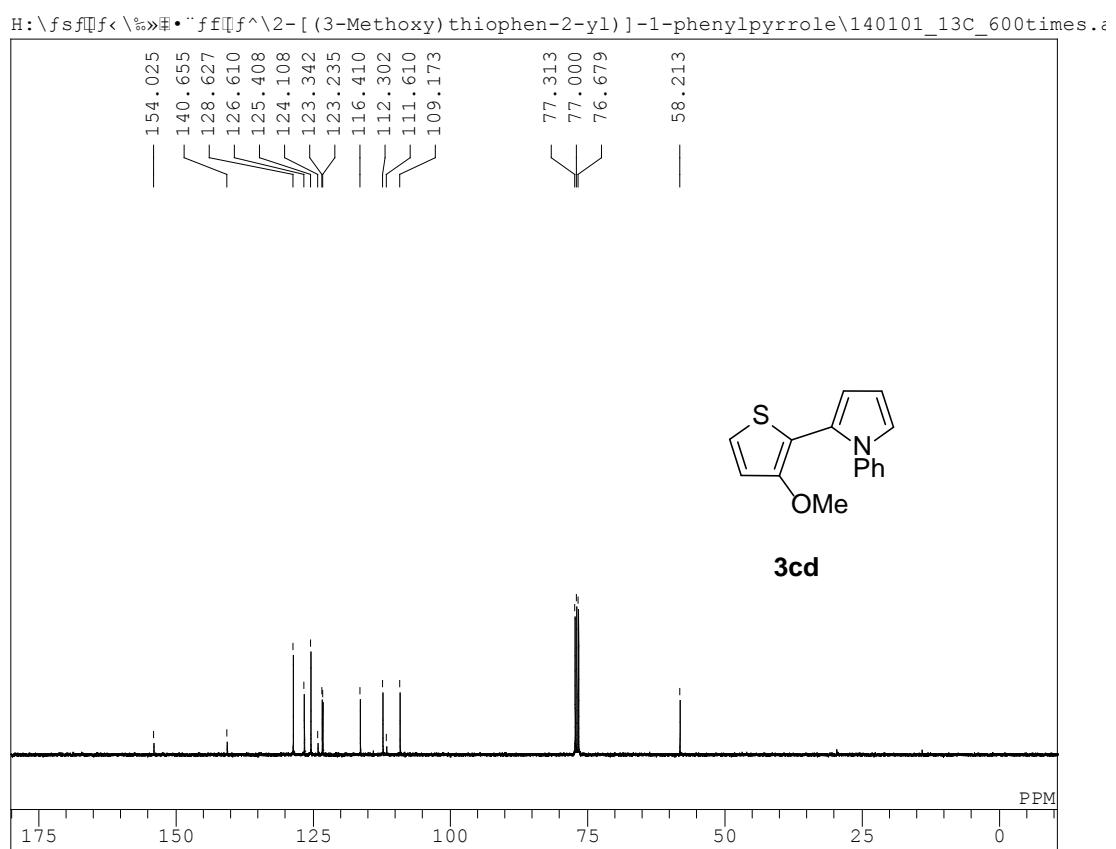
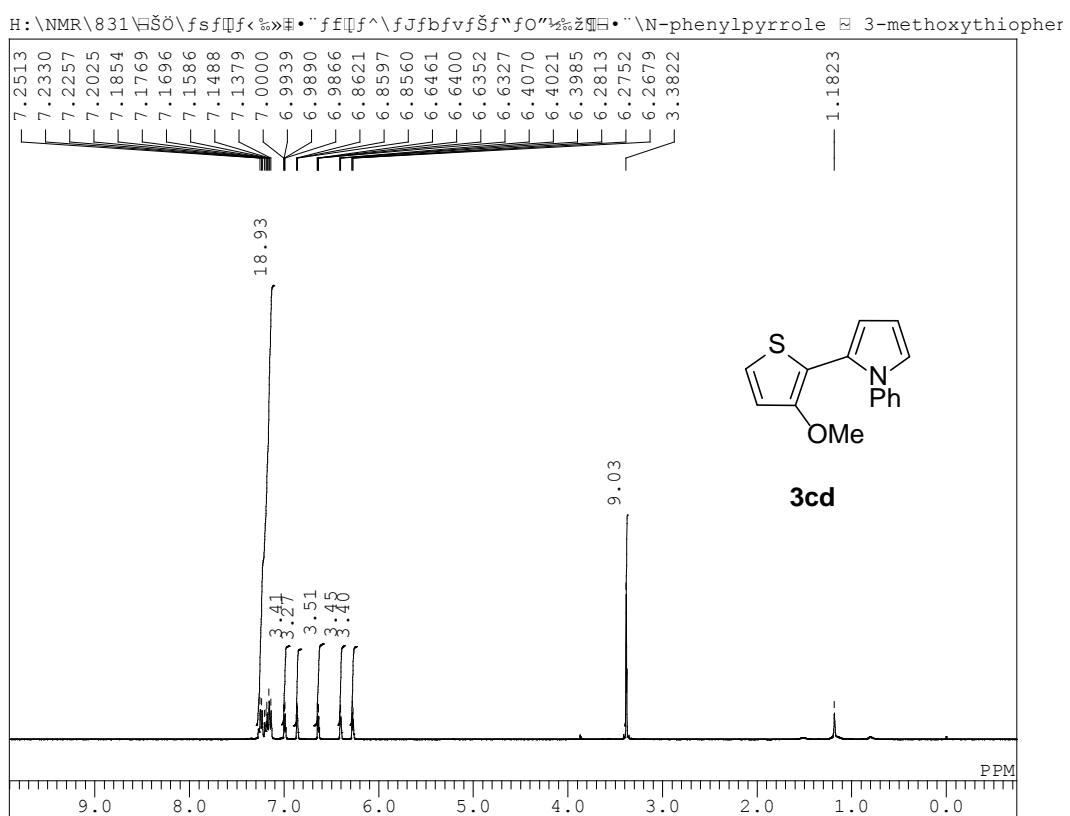


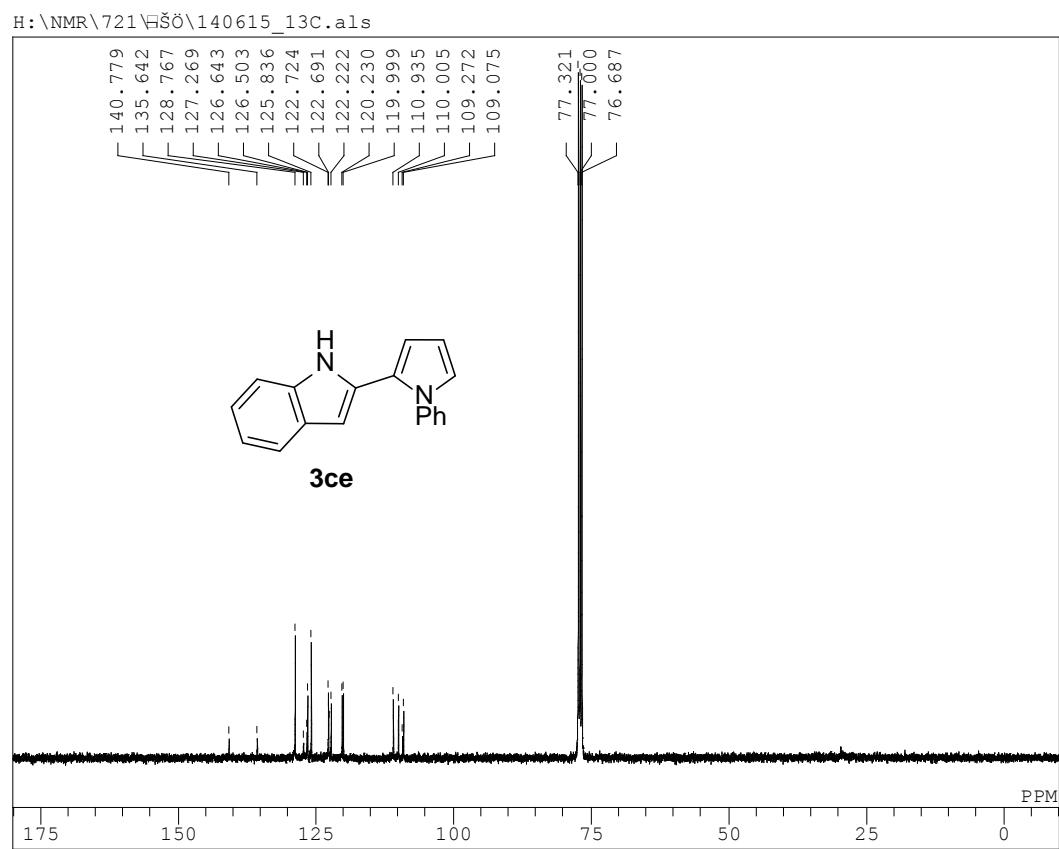
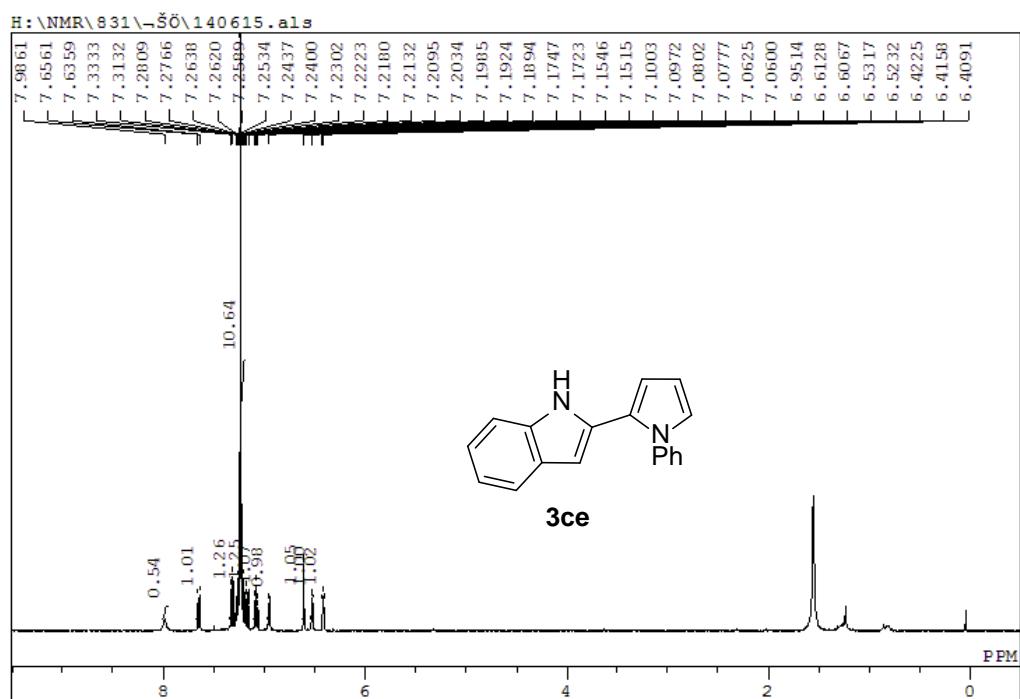
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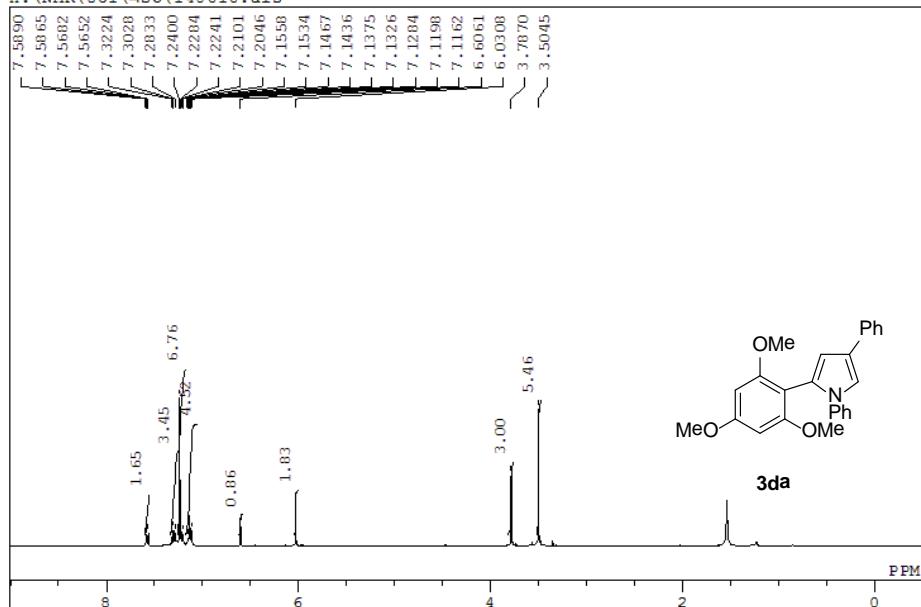
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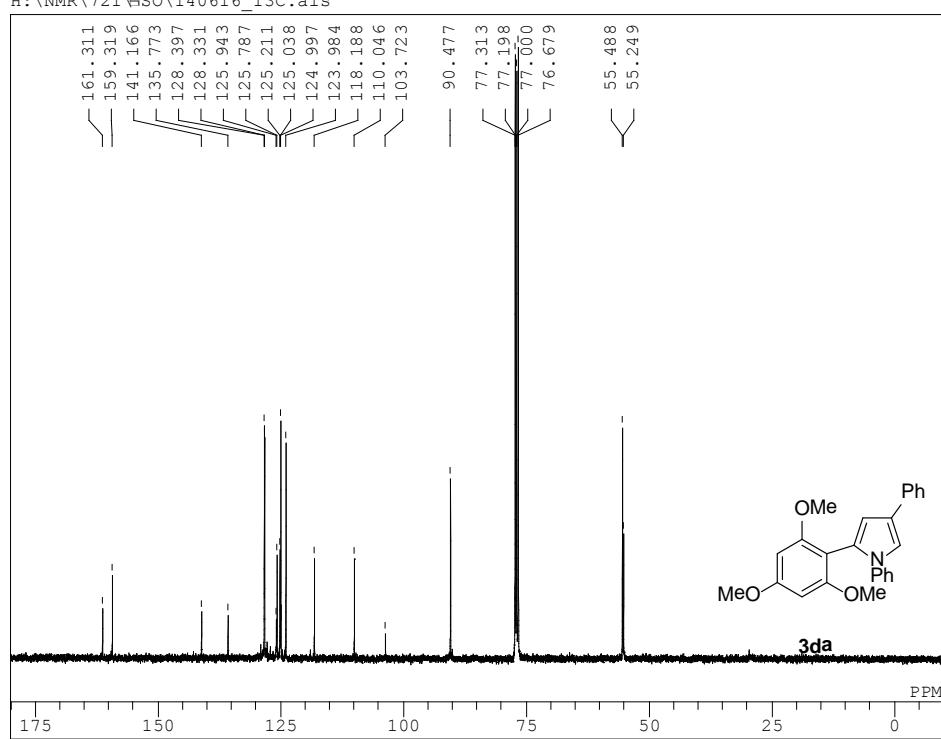


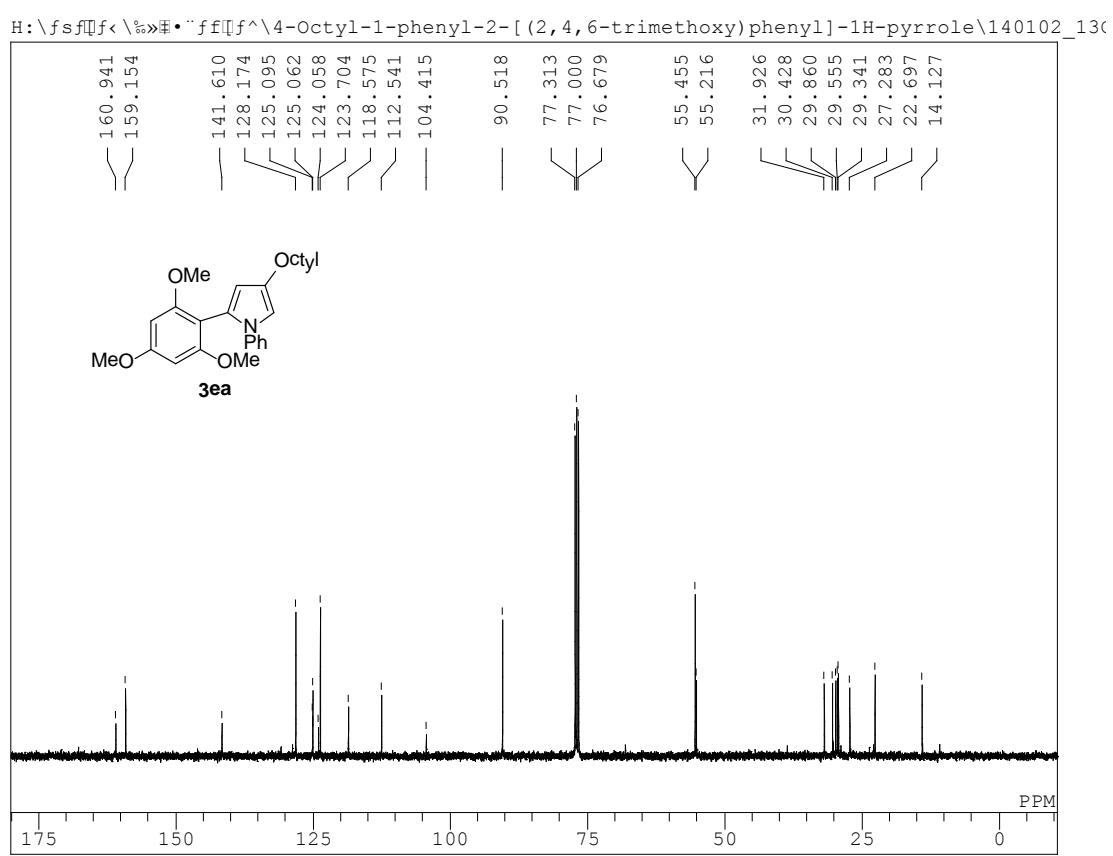
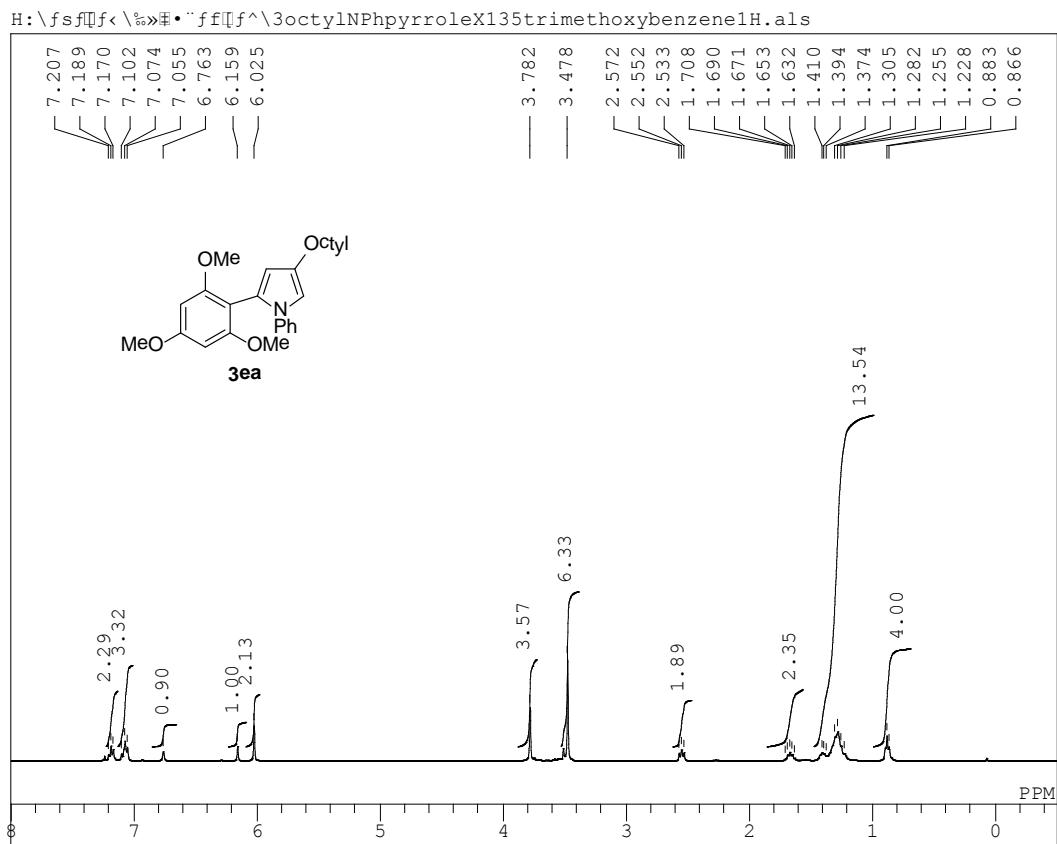


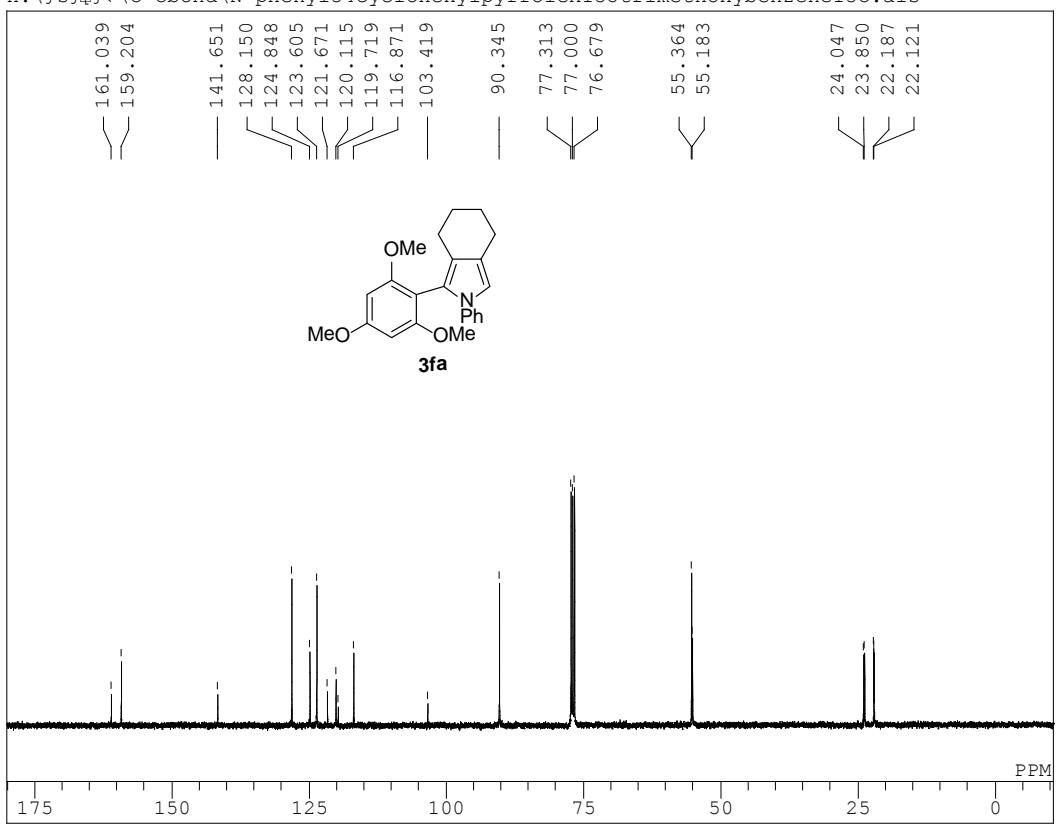
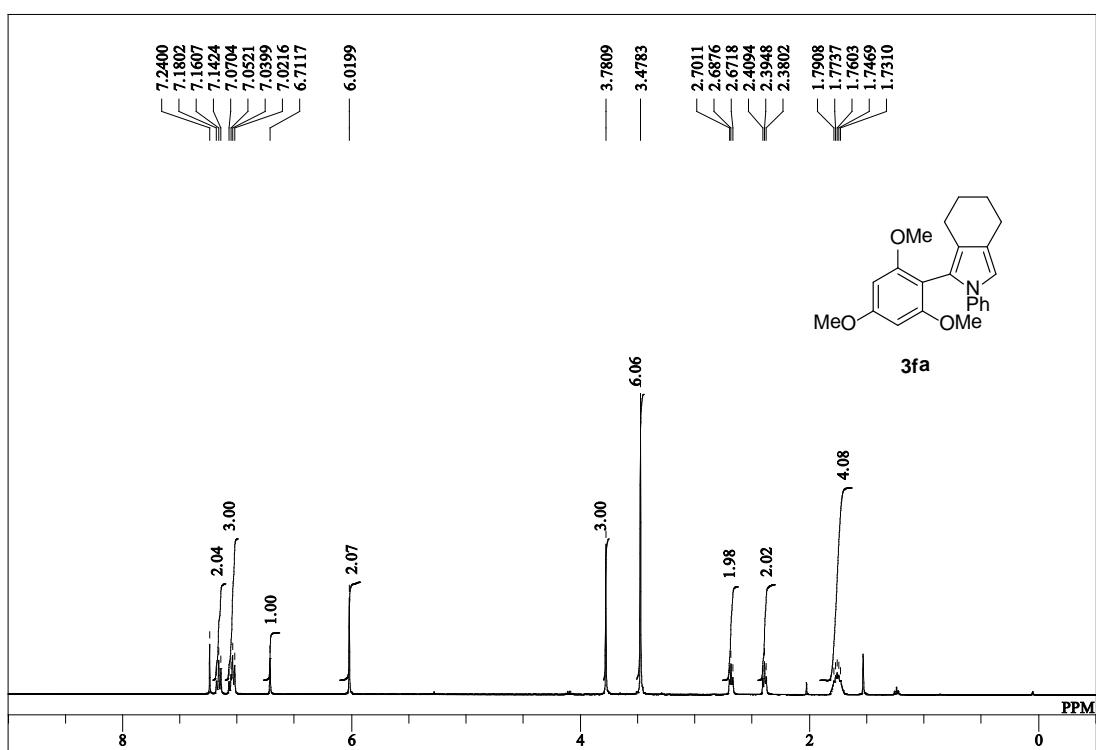
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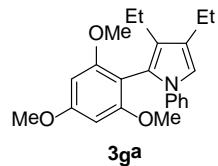
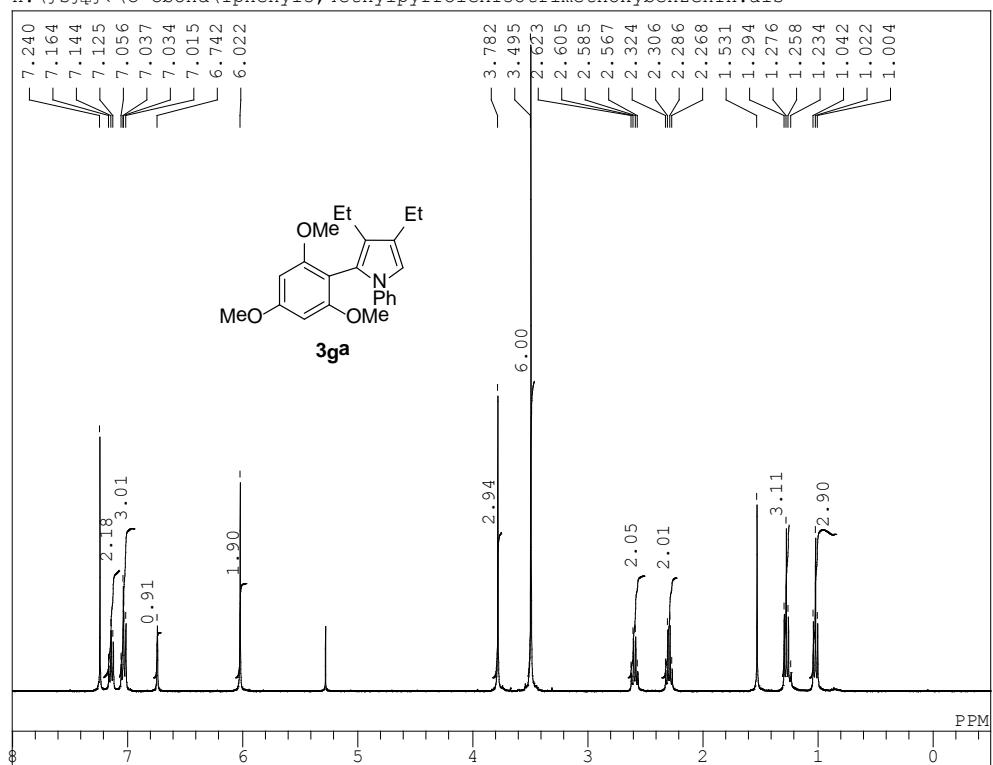
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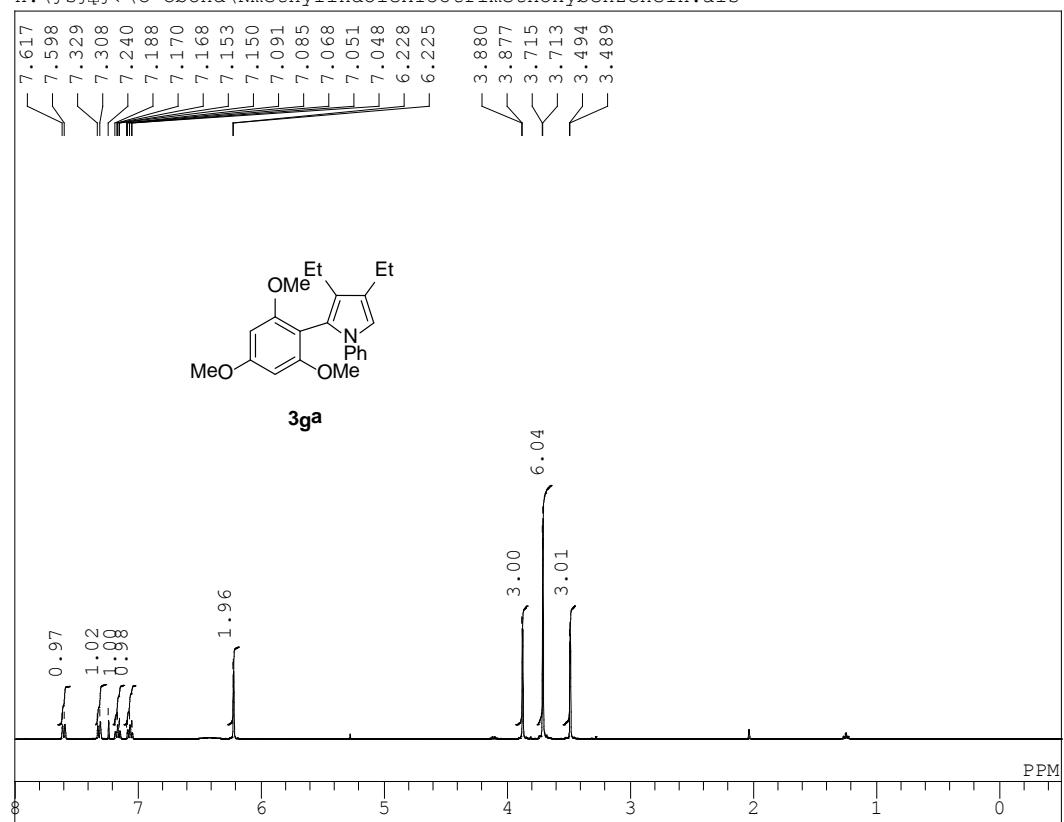


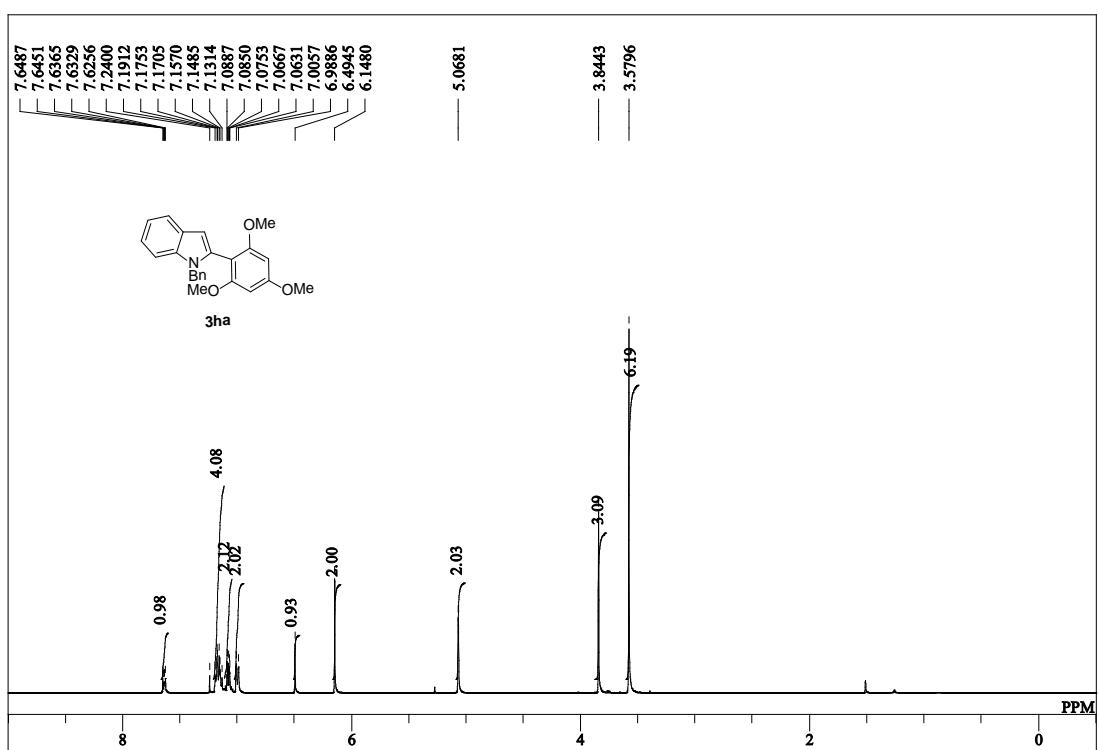


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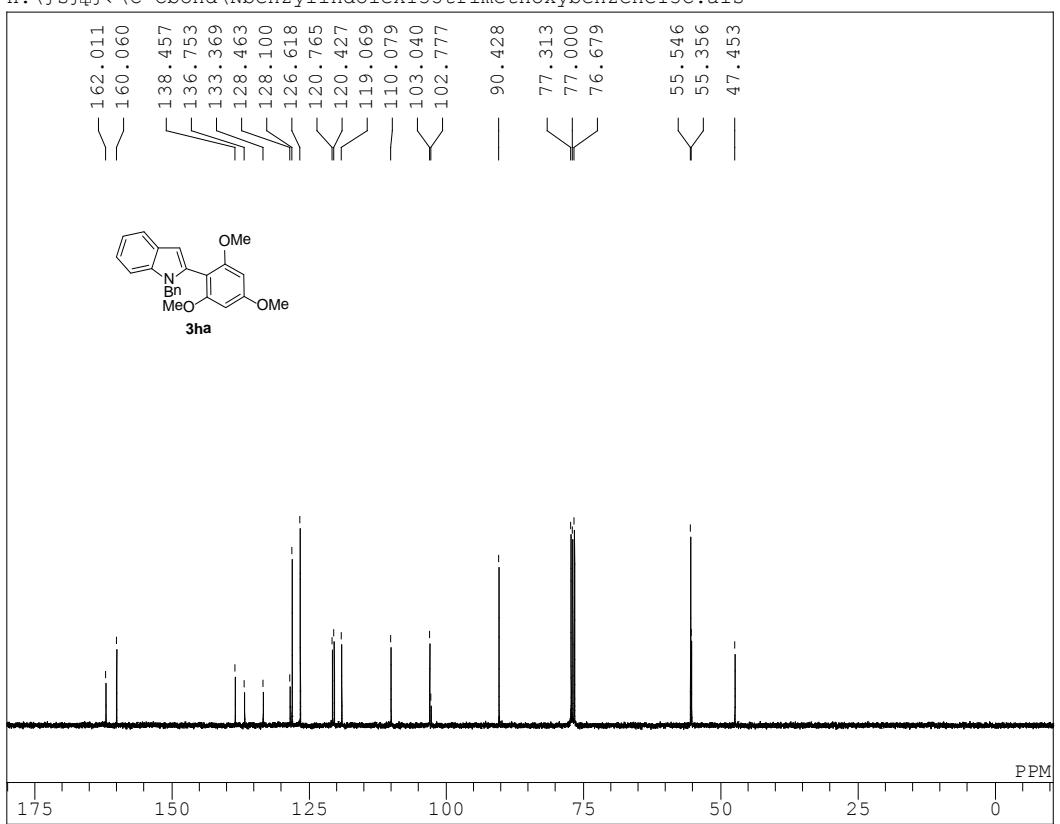


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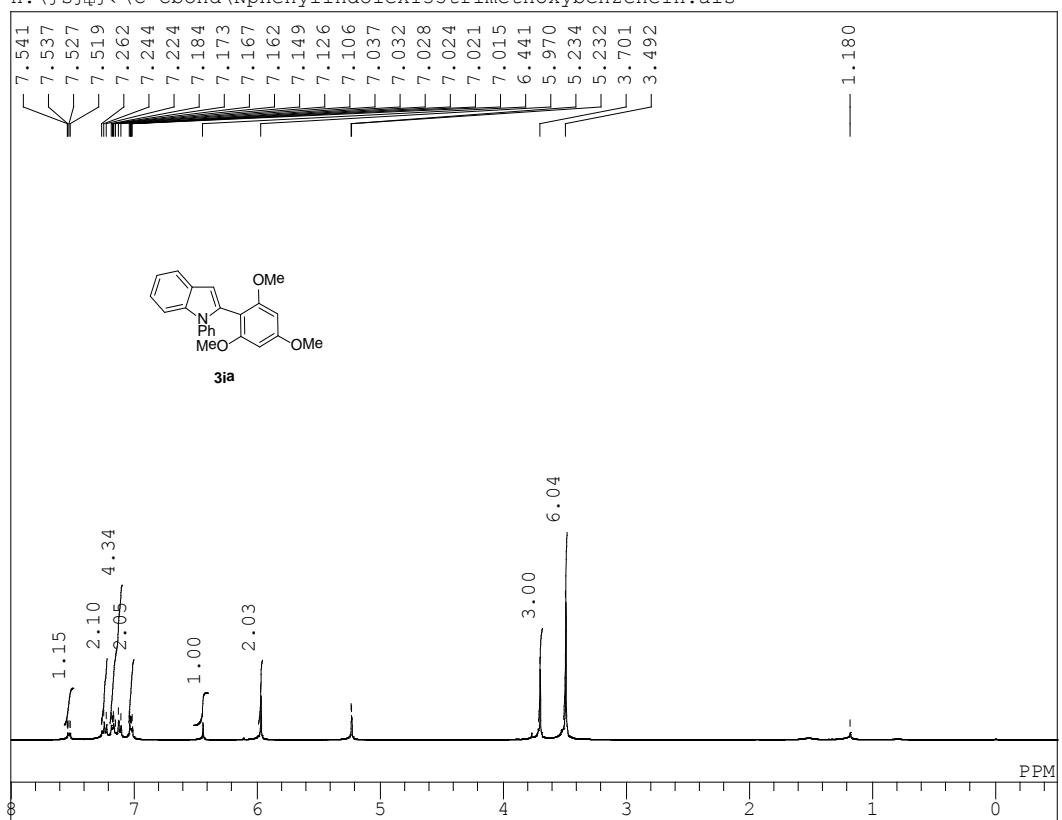




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