

## Supporting Information

### Photochemically Induced Coupling Reaction of C(sp<sup>3</sup>)–H Bonds and 4-Cyanopyridine

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#### Contents

General Information	S1
Procedure for Photochemically-Induced Coupling Reaction with 4-Cyanopyridine	S1
Procedure for Photochemically-Induced Coupling Reaction on Gram Scale	S2
Analytical Data	S2
NMR Charts	S12

#### General Information

All reactions sensitive to air or moisture were carried out under argon atmosphere and anhydrous conditions unless otherwise noted. Reagents were used as supplied unless otherwise stated. Analytical thin-layer chromatography (TLC) was performed using E. Merck Silica gel 60 F254 pre-coated plates. Flash column chromatography was generally performed using 40-50 µm Silicagel 60N (Kanto) or 75 µm Activated Alumina (Wako). <sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded on a JEOL JNM-ECX-500 (500 MHz), JNM-ECA-500 (500 MHz), or a JNM-ECS-400 (400 MHz) spectrometer. Chemical shifts are reported in δ (ppm) with reference to residual solvent signals [<sup>1</sup>H-NMR: CDCl<sub>3</sub> (7.26); <sup>13</sup>C-NMR: CDCl<sub>3</sub> (77.0)]. Signal patterns are indicated as s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; br, broad. IR spectra were recorded on a JASCO FT/IR-4100 spectrometer. ESI-TOF mass spectra were recorded on a BRUKER DALTONICS micrOTOF II or JEOL JMS-T100LP instrument (HRMS). UV irradiation was carried out by using a Riko 100 W medium-pressure mercury lamp.

#### Procedure for Photochemically-Induced Coupling Reaction with 4-Cyanopyridine

To a MeCN/H<sub>2</sub>O (2:1, 0.04 M) solution of 4-cyanopyridine **2** (16.3 mg, 157 µmol) in a test tube were added benzophenone (14.3 mg, 78.5 µmol) and cumene **1a** (44 µL, 313 µmol) at room temperature. The mixture was degassed by freeze-thaw procedure for 3 times. The test tube was placed at 5 cm distance from a UV-lamp and irradiated with a Riko 100 W medium-pressure mercury lamp at room temperature for 12 h. Then, the reaction mixture was treated with saturated aqueous sodium bicarbonate. The mixture was extracted with AcOEt (20 mL x 3), washed with brine, dried over Na<sub>2</sub>SO<sub>4</sub>, and concentrated. The analysis of crude's <sup>1</sup>H NMR chart indicated that the desired product **3a** was formed in 92% yield along with the formation of α,α-diphenyl-4-pyridylmethanol **4** in 7% yield. The residue was purified with flash column chromatography (hexane/AcOEt 5:1) to give the

pyridine derivative **3a** in 90% yield (27.7 mg).

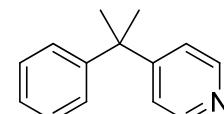
### **Procedure for Photochemically-Induced Coupling Reaction on a Gram Scale**

To a MeCN/H<sub>2</sub>O (2:1, 0.4 M) solution of 4-cyanopyridine **2** (1.03 g, 9.89 mmol) in a 50 mL flask were added benzophenone (180 mg, 0.989 mmol) and cumene **1a** (2.76 mL, 19.8 mmol) at room temperature. The mixture was degassed by freeze-thaw procedure for 3 times. The flask was placed at 5 cm distance from a UV-lamp and irradiated with a Riko 100 W medium-pressure mercury lamp at room temperature for 160 h. Then, the reaction mixture was treated with saturated aqueous sodium bicarbonate. The mixture was extracted with AcOEt (40 mL x 3), washed with brine, dried over Na<sub>2</sub>SO<sub>4</sub>, and concentrated. The residue was purified with flash column chromatography (hexane/AcOEt 5:1) to give the pyridine derivative **3a** in 78% yield (1.52 g) along with the recovery of 4-cyanopyridine **2** in 10% yield (100 mg).

### **Analytical Data**

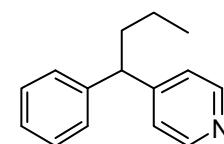
#### ***4-(2-phenylpropan-2-yl)pyridine (3a):***

90% yield (27.7 mg); colorless oil; IR (neat) 1596, 1491, 1410, 824, 765, 700 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 1.68 (6H, s), 7.13 (2H, dd, *J* = 1.4, 4.6 Hz), 7.18-7.25 (3H, m), 7.29 (2H, m), 8.48 (2H, d, *J* = 6.0 Hz); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 29.9, 42.8, 122.1, 126.2, 126.6, 128.2, 148.6, 149.5, 159.6; HRMS (ESI-TOF) calcd for C<sub>14</sub>H<sub>16</sub>N (M+H)<sup>+</sup> 198.1277, found 198.1301.



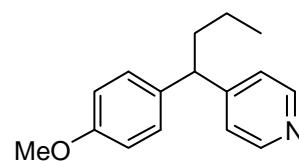
#### ***4-(1-phenylbutyl)pyridine (3b):***

72% yield (23.8 mg); colorless oil; IR (neat) 1596, 1494, 1452, 1414, 815, 743, 700 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 0.93 (3H, t, *J* = 7.4 Hz), 1.28 (2H, m), 2.01 (2H, m), 3.88 (1H, t, *J* = 7.8 Hz), 7.15 (2H, dd, *J* = 1.4, 4.6 Hz), 7.18-7.24 (3H, m), 7.30 (2H, m), 8.47 (2H, d, *J* = 6.0 Hz); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 13.9, 20.9, 37.1, 50.5, 123.3, 126.6, 127.9, 128.6, 143.3, 149.7, 154.2; HRMS (ESI-TOF) calcd for C<sub>15</sub>H<sub>18</sub>N (M+H)<sup>+</sup> 212.1434, found 212.1435.



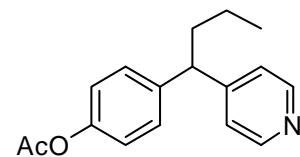
#### ***4-(1-(4-methoxyphenyl)butyl)pyridine (3c):***

83% yield (29.4 mg); colorless oil; IR (neat) 1598, 1511, 1462, 1415, 1249, 1035, 824 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 0.92 (3H, t, *J* = 7.3 Hz), 1.26 (2H, m), 1.98 (2H, m), 3.77 (3H, s), 3.83 (1H, t, *J* = 7.8 Hz), 6.83 (2H, dd, *J* = 2.3, 6.4 Hz), 7.10-7.15 (4H, m), 8.46 (2H, dd, *J* = 1.6, 4.6 Hz); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 13.9, 20.9, 37.2, 49.6, 55.2, 113.9, 123.1, 128.7, 135.3, 149.6, 154.7, 158.2; HRMS (ESI-TOF) calcd for C<sub>16</sub>H<sub>20</sub>NO (M+H)<sup>+</sup> 242.1539, found 242.1537.



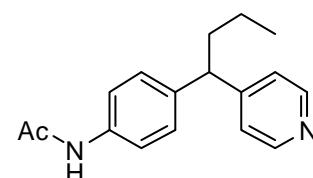
**4-(1-(pyridin-4-yl)butyl)phenyl acetate (3d):**

71% yield (35.7 mg); colorless oil; IR (neat) 1761, 1597, 1504, 1415, 1201, 824 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 0.91 (3H, t, *J* = 7.3 Hz), 1.26 (2H, m), 1.98 (2H, q, *J* = 7.8 Hz), 2.27 (3H, s), 3.87 (1H, t, *J* = 7.8 Hz), 7.01 (2H, dd, *J* = 2.3, 6.9 Hz), 7.13 (2H, dd, *J* = 1.4, 4.6 Hz), 7.20 (2H, dd, *J* = 2.3, 6.9 Hz), 8.48 (2H, dd, *J* = 1.4, 4.6 Hz); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 13.9, 20.8, 21.1, 37.1, 49.8, 121.6, 123.2, 128.7, 140.8, 149.2, 149.8, 153.8, 169.4; HRMS (ESI-TOF) calcd for C<sub>17</sub>H<sub>20</sub>NO<sub>2</sub> (M+H)<sup>+</sup> 270.1489, found 270.1484.



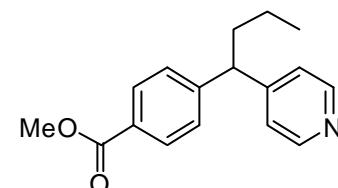
**N-(4-(1-(pyridin-4-yl)butyl)phenyl)acetamide (3e):**

63% yield (28.1 mg); colorless oil; IR (neat) 3254, 1668, 1602, 1538, 1513, 1413, 826 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 0.92 (3H, t, *J* = 7.3 Hz), 1.26 (2H, m), 1.98 (2H, q, *J* = 7.8 Hz), 2.15 (3H, s), 3.86 (1H, t, *J* = 7.8 Hz), 7.10-7.20 (4H, m), 7.35 (1H, brs), 7.43 (2H, d, *J* = 8.2 Hz), 8.48 (2H, d, *J* = 5.0 Hz); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 13.9, 20.8, 24.4, 37.0, 49.8, 120.2, 123.3, 128.3, 136.6, 138.9, 149.4, 154.5, 168.5; HRMS (ESI-TOF) calcd for C<sub>17</sub>H<sub>21</sub>N<sub>2</sub>O (M+H)<sup>+</sup> 269.1648, found 269.1633.



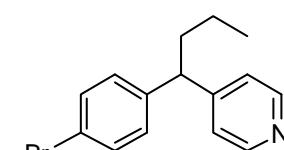
**methyl 4-(1-(pyridin-4-yl)butyl)benzoate (3f):**

65% yield (27.6 mg); colorless oil; IR (neat) 1721, 1596, 1436, 1415, 1281, 1112 cm<sup>-1</sup>; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 0.93 (3H, t, *J* = 7.8 Hz), 1.27 (2H, sextet, *J* = 7.8 Hz), 2.02 (2H, q, *J* = 7.8 Hz), 3.89 (3H, s), 3.94 (1H, t, *J* = 7.8 Hz), 7.13 (2H, d, *J* = 5.8 Hz), 7.27 (2H, d, *J* = 8.0 Hz), 7.26 (2H, d, *J* = 8.0 Hz), 8.49 (2H, d, *J* = 5.8 Hz); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 13.9, 20.9, 36.9, 50.5, 52.1, 123.2, 127.9, 128.7, 130.0, 148.5, 149.8, 153.3, 166.8; HRMS (ESI-TOF) calcd for C<sub>17</sub>H<sub>20</sub>NO<sub>2</sub> (M+H)<sup>+</sup> 270.1489, found 270.1481.



**4-(1-(4-bromophenyl)butyl)pyridine (3g):**

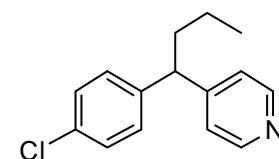
68% yield (29.7 mg); yellow oil; IR (neat) 1596, 1485, 1412, 1072, 811 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 0.92 (3H, t, *J* = 7.8 Hz), 1.26 (2H, m), 1.97 (2H, m), 3.84 (1H, t, *J* = 7.8 Hz), 7.07 (2H, d, *J* = 8.7 Hz), 7.11 (2H, d, *J* = 6.0 Hz), 7.41 (2H, d, *J* = 8.7 Hz), 8.48 (2H, d, *J* = 6.0 Hz); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 13.9, 20.8, 36.9, 49.8, 120.5, 123.1, 129.6, 131.7, 142.3, 149.8, 153.5; HRMS (ESI-TOF) calcd for C<sub>15</sub>H<sub>17</sub>NBr (M+H)<sup>+</sup> 290.0539, found 290.0532.



**4-(1-(4-chlorophenyl)butyl)pyridine (3h):**

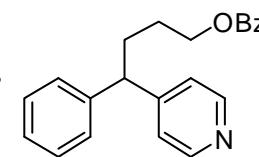
70% yield (25.1 mg); yellow oil; IR (neat) 1596, 1490, 1462, 1412, 1092, 814 cm<sup>-1</sup>; <sup>1</sup>H NMR

(400 MHz, CDCl<sub>3</sub>) δ 0.92 (3H, t, *J* = 7.3 Hz), 1.26 (2H, m), 1.98 (2H, m), 3.85 (1H, t, *J* = 7.8 Hz), 7.10-7.15 (4H, m), 7.27 (2H, m), 8.48 (2H, d, *J* = 1.4, 4.6 Hz); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 13.9, 20.8, 37.0, 49.8, 123.1, 128.7, 129.2, 132.4, 141.7, 149.8, 153.6; HRMS (ESI-TOF) calcd for C<sub>15</sub>H<sub>17</sub>NCl (M+H)<sup>+</sup> 246.1044, found 246.1035.



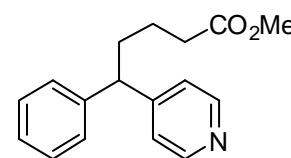
**4-phenyl-4-(pyridin-4-yl)butyl benzoate (3i):**

79% yield (38.8 mg); colorless oil; IR (neat) 1715, 1597, 1494, 1452, 1415, 1275, 1117, 713 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 1.75 (2H, m), 1.27 (2H, m), 3.95 (1H, t, *J* = 7.8 Hz), 4.33 (2H, dt, *J* = 0.9, 6.0 Hz), 7.20 (2H, dd, *J* = 1.4, 4.6 Hz), 7.23 (3H, m), 7.32 (2H, m), 7.44 (2H, t, *J* = 7.3 Hz), 7.56 (1H, t, *J* = 7.3 Hz), 8.01 (2H, d, *J* = 7.3 Hz), 8.50 (2H, dd, *J* = 1.4, 4.6 Hz); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 27.1, 31.3, 50.3, 64.4, 123.1, 126.9, 127.8, 128.3, 128.8, 129.5, 130.2, 132.9, 142.5, 149.8, 153.6, 166.5; HRMS (ESI-TOF) calcd for C<sub>22</sub>H<sub>22</sub>NO<sub>2</sub> (M+H)<sup>+</sup> 332.1645, found 332.1629.



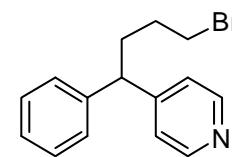
**methyl 5-phenyl-5-(pyridin-4-yl)pentanoate (3j):**

83% yield (28.4 mg); colorless oil; IR (neat) 1735, 1596, 1494, 1452, 1415, 1200, 744, 701 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 1.59 (2H, m), 2.07 (2H, m), 2.34 (2H, t, *J* = 7.3 Hz), 3.65 (3H, s), 3.88 (1H, t, *J* = 7.8 Hz), 7.15 (2H, d, *J* = 1.8, 4.6 Hz), 7.21 (3H, m), 7.30 (2H, m), 8.47 (2H, d, *J* = 1.8, 4.6 Hz); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 23.2, 33.7, 34.2, 50.6, 51.5, 123.1, 126.8, 127.8, 128.7, 142.7, 149.8, 153.6, 173.6; HRMS (ESI-TOF) calcd for C<sub>17</sub>H<sub>20</sub>NO<sub>2</sub> (M+H)<sup>+</sup> 270.1489, found 270.1476.



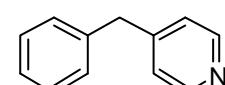
**4-(1-(4-bromophenyl)butyl)pyridine (3k):**

62% yield (27.8 mg); yellow oil; IR (neat) 1595, 1494, 1452, 1414, 743, 701 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 1.82 (2H, m), 2.22 (2H, m), 3.40 (2H, t, *J* = 6.4 Hz), 3.89 (1H, t, *J* = 7.8 Hz), 7.16 (2H, dd, *J* = 1.8, 4.6 Hz), 7.22 (3H, m), 7.31 (2H, m), 8.50 (2H, d, *J* = 1.8, 4.6 Hz); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 30.8, 33.3, 33.4, 50.0, 123.0, 126.9, 127.8, 128.8, 142.4, 149.9, 153.4; HRMS (ESI-TOF) calcd for C<sub>15</sub>H<sub>17</sub>NBr (M+H)<sup>+</sup> 290.0539, found 290.0525.



**4-benzylpyridine (3l): [CAS: 2116-65-6]:<sup>1</sup>**

51% yield (17.0 mg); colorless oil; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 3.97 (2H, s), 7.10 (2H, dd, *J* = 1.8, 4.6 Hz), 7.17 (2H, d, *J* = 7.3 Hz), 7.25 (1H, t,

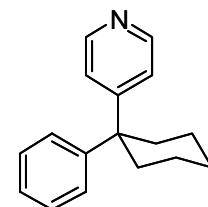


(1) Wu, G. G.; Chen, F. X.; LaFrance, D.; Liu, Z.; Greene, S. G.; Wong, Y.-S.; Xie, J. *Org. Lett.* **2011**, 13, 5220.

$J = 7.8$  Hz), 7.32 (2H, m), 8.49 (2H, dd,  $J = 1.8, 4.6$  Hz);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  41.2, 124.2, 126.7, 128.7, 129.0, 138.8, 149.7, 150.0.

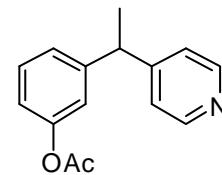
**4-(1-phenylcyclohexyl)pyridine (3m):**

50% yield (23.5 mg); colorless oil; IR (neat) 1593, 1494, 1449, 1411, 810, 759, 699  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  1.45-1.65 (6H, m), 2.20-2.35 (4H, m), 7.15-7.20 (3H, m), 7.25-7.35 (4H, m), 8.47 (2H, d,  $J = 6.4$  Hz);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  22.6, 26.1, 36.3, 46.1, 122.4, 126.0, 127.0, 128.5, 146.5, 149.7, 157.8; HRMS (ESI-TOF) calcd for  $\text{C}_{17}\text{H}_{20}\text{N}$  ( $\text{M}+\text{H}$ ) $^+$  238.1596, found 238.1608.



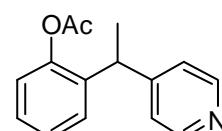
**3-(1-(pyridin-4-yl)ethyl)phenyl acetate (3n):**

83% yield (27.3 mg); colorless oil; IR (neat) 1765, 1595, 1486, 1442, 1414, 1203, 826, 801  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  1.62 (3H, d,  $J = 7.3$  Hz), 2.28 (3H, s), 4.12 (1H, q,  $J = 7.3$  Hz), 6.90 (1H, s), 6.96 (1H, d,  $J = 8.0$  Hz), 7.05 (1H, d,  $J = 8.0$  Hz), 7.12 (2H, d,  $J = 6.0$  Hz), 7.31, (1H, t,  $J = 8.0$  Hz), 8.49 (2H, d,  $J = 6.0$  Hz);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  20.9, 21.1, 43.9, 119.8, 120.7, 122.9, 125.1, 129.5, 146.0, 149.8, 150.8, 154.5, 169.4; HRMS (ESI-TOF) calcd for  $\text{C}_{15}\text{H}_{15}\text{NO}_2\text{Na}$  ( $\text{M}+\text{Na}$ ) $^+$  264.0995, found 264.0999.



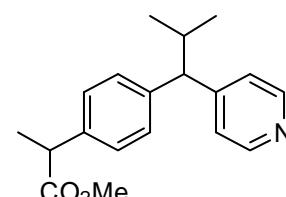
**2-(1-(pyridin-4-yl)ethyl)phenyl acetate (3o):**

68% yield (21.9 mg); colorless oil; IR (neat) 1764, 1597, 1488, 1451, 1414, 1201, 829, 755  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  1.59 (3H, d,  $J = 7.3$  Hz), 2.16 (3H, s), 4.23 (1H, q,  $J = 7.3$  Hz), 7.04 (1H, dd,  $J = 1.4, 7.8$  Hz), 7.09 (2H, dd,  $J = 1.8, 4.6$  Hz), 7.20-7.30 (3H, m), 8.47 (2H, dd,  $J = 1.8, 4.6$  Hz);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  20.2, 20.8, 38.1, 122.7, 122.8, 126.3, 127.8, 128.3, 135.9, 148.4, 149.6, 154.4, 169.0; HRMS (ESI-TOF) calcd for  $\text{C}_{15}\text{H}_{16}\text{NO}_2$  ( $\text{M}+\text{H}$ ) $^+$  242.1176, found 242.1175.



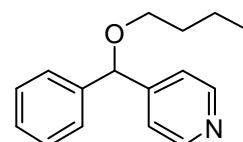
**methyl 2-(4-(2-methyl-1-(pyridin-4-yl)propyl)phenyl)propanoate (3p):**

75% yield based on the NMR analysis using  $\text{CH}_2\text{Br}_2$  as an internal standard, 9.2 mg (20%) was isolated; colorless oil; IR (neat) 1736, 1595, 1459, 1415, 1210, 1166, 809  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  0.85 (3H, d,  $J = 6.4$  Hz), 0.87 (3H, d,  $J = 6.4$  Hz), 1.45 (3H, d,  $J = 7.3$  Hz), 2.46 (1H, m), 3.37 (1H, d,  $J = 10.5$  Hz), 3.64 (3H, s), 3.67 (1H, q,  $J = 7.3$  Hz), 7.20 (6H, m), 8.46 (2H, dd,  $J = 1.4, 4.6$  Hz);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  18.5, 21.5, 21.6, 31.4, 44.9, 52.0, 59.9, 123.5, 127.7, 128.2, 138.7, 141.7, 149.6, 153.8, 175.0; HRMS (ESI-TOF) calcd for  $\text{C}_{19}\text{H}_{23}\text{NO}_2\text{Na}$  ( $\text{M}+\text{Na}$ ) $^+$  320.1621, found 320.1607.



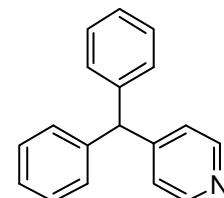
**4-(butoxy(phenyl)methyl)pyridine (3q):**

66% yield (28.6 mg); colorless oil; IR (neat) 1596, 1493, 1453, 1412, 1100, 790, 740, 700  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  0.92 (3H, t,  $J = 7.3$  Hz), 1.42 (2H, m), 1.63 (2H, m), 3.45 (2H, m), 5.29 (1H, s), 7.25-7.35 (7H, m), 8.53 (2H, dd,  $J = 1.4, 4.6$  Hz);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  13.9, 19.4, 31.9, 69.1, 82.3, 121.6, 127.1, 128.0, 128.6, 141.0, 149.7, 151.6; HRMS (ESI-TOF) calcd for  $\text{C}_{16}\text{H}_{20}\text{NO}$  ( $\text{M}+\text{H}$ ) $^+$  242.1539, found 242.1525.



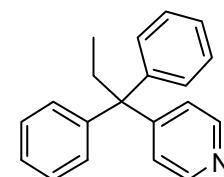
**4-benzhydrylpyridine (3r) [CAS: 3678-72-6]:<sup>2</sup>**

87% yield (38.5 mg); colorless solid;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  5.51 (1H, s), 7.05 (2H, dd,  $J = 1.4, 4.6$  Hz), 7.09 (4H, m), 7.20-7.35 (6H, m), 8.51 (2H, dd,  $J = 1.4, 4.6$  Hz);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  56.2, 124.7, 126.9, 128.6, 129.3, 142.0, 149.6, 152.9.



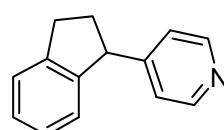
**4-(1,1-diphenylpropyl)pyridine (3s):**

74% yield (31.0 mg); colorless oil; IR (neat) 1592, 1493, 1445, 1410, 815, 757, 702  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  0.78 (3H, t,  $J = 7.3$  Hz), 2.62 (2H, q,  $J = 7.3$  Hz), 7.20-7.35 (12H, m), 8.49 (2H, dd,  $J = 1.8, 5.0$  Hz);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  10.1, 32.1, 56.8, 124.4, 126.3, 128.0, 129.1, 145.5, 149.3, 156.6; HRMS (ESI-TOF) calcd for  $\text{C}_{20}\text{H}_{20}\text{N}$  ( $\text{M}+\text{H}$ ) $^+$  274.1590, found 274.1576.



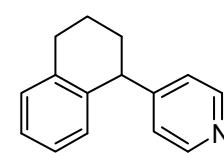
**4-(2,3-dihydro-1*H*-inden-1-yl)pyridine (3t):**

89% yield (23.0 mg); colorless oil; IR (neat) 1597, 1478, 1458, 1414, 819, 747  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  2.04 (1H, ddt,  $J = 8.2, 8.7, 12.8$  Hz), 2.61 (1H, dddd,  $J = 4.6, 8.2, 8.7, 12.8$  Hz), 2.95-3.15 (2H, m), 4.32 (1H, t,  $J = 8.2$  Hz), 6.94 (1H, d,  $J = 7.3$  Hz), 7.11 (2H, dd,  $J = 1.6, 4.6$  Hz), 7.15 (1H, t,  $J = 7.3$  Hz), 7.22 (1H, t,  $J = 7.3$  Hz), 7.31 (1H, d,  $J = 7.3$  Hz), 8.51 (2H, dd,  $J = 1.6, 4.6$  Hz);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  31.8, 35.8, 50.8, 123.3, 124.6, 124.7, 126.6, 127.1, 144.3, 144.9, 149.8, 154.3; HRMS (ESI-TOF) calcd for  $\text{C}_{14}\text{H}_{14}\text{N}$  ( $\text{M}+\text{H}$ ) $^+$  196.1121, found 196.1127.



**4-(1,2,3,4-tetrahydronaphthalen-1-yl)pyridine (3u):**

70% yield (28.4 mg); colorless oil; IR (neat) 1596, 1492, 1450, 1412, 815, 743  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  1.70-1.90 (3H, m), 2.17 (1H, m),

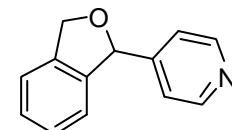


(2) Niwa, T.; Yorimitsu, H.; Oshima, K. *Org. Lett.* **2007**, 9, 2373.

2.80-3.00 (2H, m), 4.12 (1H, dd,  $J = 6.0, 6.9$  Hz), 6.78 (1H, d,  $J = 7.8$  Hz), 7.02 (2H, dd,  $J = 1.8, 4.6$  Hz), 7.06 (1H, dd,  $J = 7.3, 7.8$  Hz), 7.16 (2H, d,  $J = 7.3$  Hz), 8.49 (2H, dd,  $J = 1.8, 4.6$  Hz);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  20.5, 29.5, 32.5, 44.9, 124.1, 125.9, 126.4, 129.2, 130.0, 137.2, 137.6, 149.6, 156.3; HRMS (ESI-TOF) calcd for  $\text{C}_{15}\text{H}_{16}\text{N} (\text{M}+\text{H})^+$  210.1277, found 210.1273.

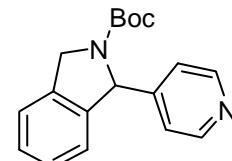
**4-(1,3-dihydroisobenzofuran-1-yl)pyridine (3v):**

75% yield (30.3 mg); yellow oil; IR (neat) 1599, 1462, 1414, 1044, 802, 748  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  5.25 (1H, dd,  $J = 1.8, 12.4$  Hz), 5.37 (1H, dd,  $J = 2.8, 12.4$  Hz), 6.14 (1H, brs), 7.07 (1H, d,  $J = 7.8$  Hz), 7.20-7.35 (5H, m), 8.58 (2H, dd,  $J = 1.4, 4.6$  Hz);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  73.7, 84.6, 121.16, 121.24, 121.9, 127.7, 128.1, 138.7, 140.4, 150.0, 151.1; HRMS (ESI-TOF) calcd for  $\text{C}_{13}\text{H}_{12}\text{NO} (\text{M}+\text{H})^+$  198.0913, found 198.0908.



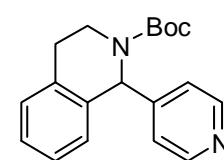
**tert-butyl 1-(pyridin-4-yl)isoindoline-2-carboxylate (3w):**

77% yield (32.0 mg), mixture of two rotamers; amorphous; IR (neat) 1699, 1598, 1475, 1391, 1170, 747  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  1.24 (45/8H, s), 1.47 (27/8H, s), 4.86 (6/8H, s), 4.91 (10/8H, s), 5.83 (5/8H, s), 5.96 (3/8H, s), 6.97 (5/8H, d,  $J = 7.8$  Hz), 7.02 (3/8H, d,  $J = 7.8$  Hz), 7.15-7.25 (3H, m), 7.29 (2H, m), 8.54 (2H, d,  $J = 5.5$  Hz); Detectable signals of  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  28.1, 28.4, 52.7, 53.0, 66.2, 66.5, 80.4, 121.4, 121.5, 122.7, 123.0, 123.2, 123.3, 127.8, 128.2, 135.7, 140.0, 149.8, 152.9, 154.0; HRMS (ESI-TOF) calcd for  $\text{C}_{18}\text{H}_{21}\text{N}_2\text{O}_2 (\text{M}+\text{H})^+$  297.1598, found 297.1584.



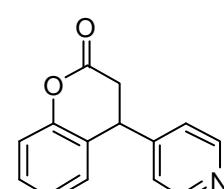
**tert-butyl 1-(pyridin-4-yl)-3,4-dihydroisoquinoline-2(1H)-carboxylate (3x):**

82% yield (40.3 mg), mixture of two rotamers; colorless oil; IR (neat) 1693, 1595, 1455, 1415, 1166, 750  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  1.48 (9H, s), 2.75 (1H, brs), 2.93 (1H, brs), 3.27 (1H, ddd,  $J = 4.6, 9.6, 13.2$  Hz), 3.89 (1/2H, brs), 4.02 (1/2H, brs), 6.12 (1/2H, brs), 6.34 (1/2H, brs), 7.08 (1H, brs), 7.14 (2H, d,  $J = 6.0$  Hz), 7.15-7.30 (3H, m), 8.51 (2H, d,  $J = 6.0$  Hz); Detectable signals of  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  28.35, 28.42, 38.4, 39.4, 56.3, 57.4, 80.5, 122.8, 126.3, 127.5, 128.2, 128.9, 134.1, 135.3, 149.6, 151.8, 154.9; HRMS (ESI-TOF) calcd for  $\text{C}_{19}\text{H}_{23}\text{N}_2\text{O}_2 (\text{M}+\text{H})^+$  311.1754, found 311.1750.



**4-(pyridin-4-yl)chroman-2-one (3y):**

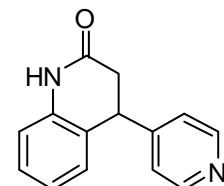
84% yield (31.3 mg); yellow oil; IR (neat) 1770, 1597, 1486, 1455, 1416, 1216, 824, 759  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  3.03 (1H, dd,  $J = 6.4, 16.0$  Hz), 3.11 (1H, dd,  $J = 6.4, 16.0$  Hz), 4.34 (1H, t,  $J = 6.4$  Hz), 7.00 (1H,



d,  $J = 6.8$  Hz), 7.05-7.20 (4H, m), 7.35 (1H, m), 8.57 (2H, dd,  $J = 1.4, 4.6$  Hz);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  36.1, 40.1, 117.5, 122.6, 123.7, 124.9, 128.1, 129.5, 149.2, 150.5, 151.7, 166.6; HRMS (ESI-TOF) calcd for  $\text{C}_{14}\text{H}_{12}\text{NO}_2$  ( $\text{M}+\text{H}$ ) $^+$  226.0863, found 226.0857.

**4-(pyridin-4-yl)-3,4-dihydroquinolin-2(1H)-one (3z):**

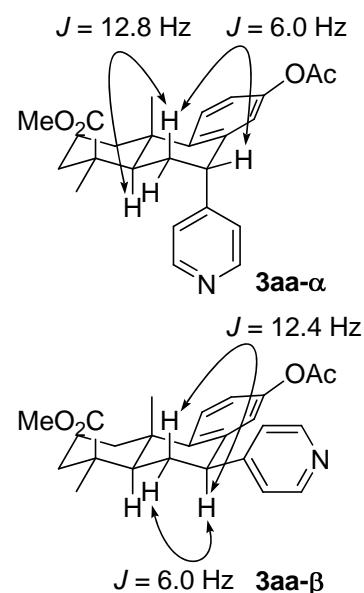
84% yield (29.2 mg); colorless solid; m.p. 210-213 °C; IR (neat) 3208, 1681, 1595, 1488, 1417, 825, 756  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  2.90 (1H, dd,  $J = 7.3, 16.5$  Hz), 3.00 (1H, dd,  $J = 6.4, 16.5$  Hz), 4.29 (1H, dd,  $J = 6.4, 7.3$  Hz), 6.90-7.05 (3H, m), 7.13 (2H, d,  $J = 6.0$  Hz), 7.25 (1H, t,  $J = 7.8$  Hz), 8.57 (2H, d,  $J = 6.0$  Hz), 9.11 (1H, brs);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  37.5, 41.3, 116.1, 122.9, 123.6, 124.4, 128.3, 128.7, 137.1, 150.1, 150.7, 170.0; HRMS (ESI-TOF) calcd for  $\text{C}_{14}\text{H}_{13}\text{N}_2\text{O}$  ( $\text{M}+\text{H}$ ) $^+$  225.1022, found 225.1041.



**Pyridinyl O-acetoxy podocarpic acid methyl ester (3aa):**

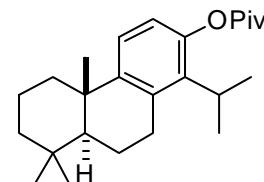
71% (45.3 mg); **3aa- $\alpha$**  / **3aa- $\beta$**  = 2:1; amorphous.

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  0.81 (2H, s), 0.86 (2/3H, dt,  $J = 4.6, 13.7$  Hz), 1.05 (2H, s), 1.10 (1/3H, dt,  $J = 4.1, 13.7$  Hz), 1.16 (1H, s), 1.20-1.50 (10/3H, m), 1.62 (2/3H, m), 1.73 (2/3H, dd,  $J = 1.4, 12.4$  Hz), 1.85-2.25 (10/3H, m), 2.27 (1H, s), 2.30 (2H, s), 2.38 (1/3H, dd,  $J = 6.0, 14.2$  Hz), 2.52 (2/3H, ddd,  $J = 6.0, 12.8, 12.8$  Hz), 3.63 (1H, s), 3.65 (2H, s), 3.99 (1/3H, dd,  $J = 6.0, 12.4$  Hz), 4.31 (2/3H, d,  $J = 6.0$  Hz), 6.66 (1/3H, d,  $J = 8.7$  Hz), 6.74 (1/3H, dd,  $J = 2.3, 8.7$  Hz), 6.80-7.00 (8/3H, m), 7.03 (1/3H, d,  $J = 2.3$  Hz), 7.07 (2/3H, d,  $J = 1.8$  Hz), 7.11 (2/3H, dd,  $J = 1.4, 4.6$  Hz), 8.48 (4/3H, d,  $J = 6.0$  Hz), 8.53 (2/3H, d,  $J = 6.0$  Hz); Detectable signals of  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  19.7, 19.8, 21.1, 21.2, 22.6, 23.4, 27.9, 28.4, 28.8, 31.5, 37.0, 37.4, 38.8, 38.9, 39.3, 43.1, 43.9, 44.5, 45.9, 48.3, 51.39, 51.44, 51.7, 118.3, 118.8, 119.1, 119.5, 124.07, 124.13, 130.6, 131.6, 131.7, 134.0, 149.4, 149.5, 149.7, 149.9, 150.3, 150.6, 156.2, 156.3, 169.5, 169.6, 177.3, 177.5; HRMS (ESI-TOF) calcd for  $\text{C}_{25}\text{H}_{30}\text{NO}_4$  ( $\text{M}+\text{H}$ ) $^+$  408.2169, found 408.2159.



**Preparation of *O*-pivaloyl totarol (1bb):**

To a solution of (+)-totarol (94.0 mg, 328  $\mu\text{mol}$ ) in pyridine (1.6 mL, 0.2 M) were added pivaloyl chloride (480  $\mu\text{L}$ , 3.90 mmol) and DMAP (2.0 mg, 16.4  $\mu\text{mol}$ ) at room temperature. The mixture was refluxed on 110 °C for three days, then the reaction was quenched with water. The mixture was extracted with  $\text{Et}_2\text{O}$  (20 mL x 3), washed with brine,



dried over  $\text{Na}_2\text{SO}_4$ , and concentrated. The residue was purified with flash column chromatography (hexane/AcOEt 40:1) to afford *O*-pivaloyl totarol **1aa** in 100% yield (122 mg): white solid; m.p. 113–115 °C; IR (neat) 1750, 1473, 1123, 733  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  0.93 (3H, s), 0.96 (3H, s), 1.21 (3H, s), 1.23–1.31 (9H, m), 1.39 (9H, s), 1.48 (1H, d,  $J$  = 13.8 Hz), 1.55–1.80 (3H, m), 1.93 (1H, dd,  $J$  = 7.8, 13.3 Hz), 2.25 (1H, d,  $J$  = 12.8 Hz), 2.78 (1H, m), 2.95 (1H, dd,  $J$  = 6.4, 17.4 Hz), 3.29 (1H, quintet,  $J$  = 7.3 Hz), 6.68 (1H, d,  $J$  = 8.7 Hz), 7.14 (1H, d,  $J$  = 8.7 Hz);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  19.2, 19.4, 20.69, 20.73, 21.6, 25.0, 27.0, 27.3, 28.8, 33.2, 33.3, 38.0, 39.1, 39.3, 41.4, 49.3, 120.5, 123.2, 134.0, 136.4, 147.6, 147.7, 177.5; HRMS (ESI-TOF) calcd for  $\text{C}_{25}\text{H}_{38}\text{O}_2\text{Na}$  ( $\text{M}+\text{Na}$ ) $^+$  393.2764, found 393.2772.

### *Pyridinyl O-pivaloyl totarol (3bb):*

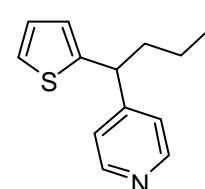
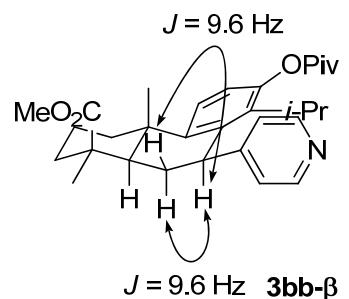
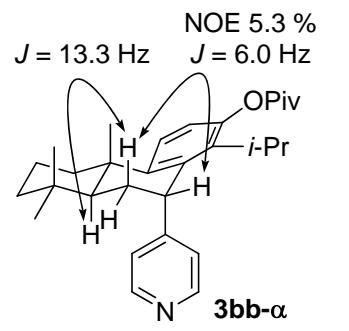
72% (52.8 mg); **3bb- $\alpha$**  / **3bb- $\beta$**  = 2:3; colorless oil.

**3bb- $\alpha$ :** IR (neat) 1748, 1595, 1471, 1122, 732  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  0.39 (3H, s), 0.69 (3H, d,  $J$  = 6.8 Hz), 0.84 (3H, s), 1.06 (1H, m), 1.14 (3H, d,  $J$  = 6.8 Hz), 1.23–1.40 (15H, m), 1.50–1.85 (3H, m), 2.19 (1H, ddd,  $J$  = 6.0, 13.3, 13.3 Hz), 2.30 (1H, d,  $J$  = 13.3 Hz), 2.78 (1H, quintet,  $J$  = 6.8 Hz), 4.41 (1H, d,  $J$  = 6.0 Hz), 6.81 (1H, d,  $J$  = 9.1 Hz), 6.91 (2H, brs), 7.26 (1H, d,  $J$  = 9.1 Hz), 8.0 Hz, 46 (2H, d,  $J$  = 4.1 Hz);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  19.4, 19.7, 20.9, 21.6, 25.5, 27.3, 27.9, 28.1, 32.3, 32.5, 38.9, 39.1, 39.3, 40.9, 43.2, 43.6, 122.6, 123.8, 124.2, 133.3, 137.1, 148.2, 148.8, 149.3, 156.7, 177.4; HRMS (ESI-TOF) calcd for  $\text{C}_{30}\text{H}_{42}\text{NO}_2$  ( $\text{M}+\text{H}$ ) $^+$  448.3210, found 448.3197.

**3bb- $\beta$ :** IR (neat) 1747, 1595, 1472, 1122, 732  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  0.55 (3H, d,  $J$  = 6.9 Hz), 0.83 (3H, s), 0.96 (3H, s), 1.08 (3H, d,  $J$  = 6.9 Hz), 1.15–1.90 (19H, m), 2.25–2.35 (2H, m), 2.86 (1H, quintet,  $J$  = 6.9 Hz), 4.37 (1H, dd,  $J$  = 9.6, 9.6 Hz), 6.78 (1H, d,  $J$  = 8.7 Hz), 6.94 (2H, brs), 7.24 (1H, d,  $J$  = 8.7 Hz), 8.47 (2H, d,  $J$  = 5.0 Hz);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  19.1, 19.2, 21.1, 21.3, 24.7, 27.3, 28.5, 31.5, 32.9, 33.3, 38.1, 39.1, 39.7, 41.4, 44.6, 48.8, 122.2, 122.5, 123.1, 134.4, 137.4, 148.9, 149.6, 149.8, 158.9, 177.4; HRMS (ESI-TOF) calcd for  $\text{C}_{30}\text{H}_{42}\text{NO}_2$  ( $\text{M}+\text{H}$ ) $^+$  448.3210, found 448.3190.

### **4-(1-(thiophen-2-yl)butyl)pyridine (3cc):**

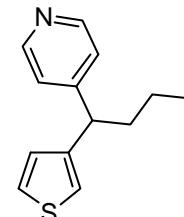
67% yield (22.9 mg); yellow oil; IR (neat) 1597, 1414, 822, 697  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  0.93 (3H, t,  $J$  = 7.5 Hz), 1.20–1.40 (2H, m), 1.90–2.10 (2H, m), 4.12 (1H, t,  $J$  = 7.5 Hz), 6.83 (1H, d,  $J$  = 3.4 Hz), 6.93 (1H, dd,  $J$  = 3.4, 5.2 Hz), 7.10–7.20 (3H, m), 8.51 (2H, d,  $J$  = 6.3 Hz);  $^{13}\text{C}$



NMR (125 MHz, CDCl<sub>3</sub>) δ 13.8, 20.9, 38.9, 45.9, 123.0, 123.9, 124.3, 126.7, 147.2, 149.8, 153.6; HRMS (ESI-TOF) calcd for C<sub>13</sub>H<sub>16</sub>NS (M+H)<sup>+</sup> 218.0998, found 218.1000.

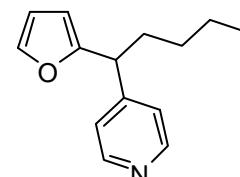
**4-(1-(thiophen-3-yl)butyl)pyridine (3dd):**

47% yield (19.3 mg); yellow oil; IR (neat) 1598, 1415, 824, 775 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 0.92 (3H, t, J = 7.8 Hz), 1.20-1.40 (2H, m), 1.90-2.10 (2H, m), 3.96 (1H, t, J = 7.8 Hz), 6.87 (1H, dd, J = 1.4, 5.0 Hz), 7.01 (1H, dd, J = 1.4, 2.7 Hz), 7.13 (2H, dd, J = 1.8, 4.6 Hz), 7.25 (1H, dd, J = 2.7, 5.0 Hz), 8.49 (2H, dd, J = 1.8, 4.6 Hz); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 13.9, 20.8, 37.6, 46.1, 120.6, 123.2, 125.8, 127.3, 144.1, 149.7, 153.9; HRMS (ESI-TOF) calcd for C<sub>13</sub>H<sub>16</sub>NS (M+H)<sup>+</sup> 218.0998, found 218.0991.



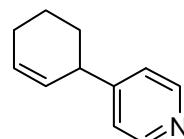
**4-(1-(furan-2-yl)pentyl)pyridine (3ee):**

51% yield (14.2 mg); colorless oil; IR (neat) 1597, 1560, 1503, 1460, 1415, 1010, 801, 732 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 0.87 (3H, t, J = 7.3 Hz), 1.10-1.40 (4H, m), 1.87 (1H, m), 2.08 (1H, m), 3.89 (1H, t, J = 7.8 Hz), 6.10 (1H, d, J = 3.2 Hz), 6.30 (1H, dd, J = 1.8, 3.2 Hz), 7.15 (2H, dd, J = 1.4, 4.6 Hz), 7.32 (1H, d, J = 1.8 Hz), 8.51 (2H, dd, J = 1.4, 4.6 Hz); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 13.9, 22.4, 29.6, 33.8, 44.7, 105.9, 110.1, 123.2, 141.7, 149.8, 151.9, 156.1; HRMS (ESI-TOF) calcd for C<sub>14</sub>H<sub>18</sub>NO (M+H)<sup>+</sup> 216.1383, found 216.1392.



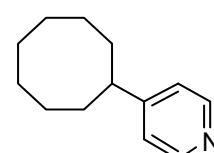
**4-(cyclohex-2-enyl)pyridine (3ff) [CAS: 78905-51-8]:<sup>3</sup>**

44% yield (9.8 mg); colorless oil; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 1.50-1.80 (3H, m), 1.95-2.15 (3H, m), 3.39 (1H, m), 5.66 (1H, dd, J = 1.8, 10.0 Hz), 5.95 (1H, ddd, J = 2.3, 3.6, 10.0 Hz), 7.15 (2H, d, J = 6.0 Hz), 8.50 (2H, d, J = 6.0 Hz); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 20.8, 24.8, 31.7, 41.1, 123.2, 128.1, 129.7, 149.6, 155.4.



**4-cyclooctylpyridine (3gg):**

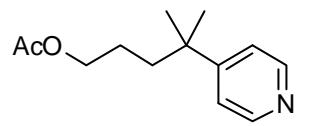
35% yield (11.1 mg); yellow oil; IR (neat) 1596, 1445, 1412, 832 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 1.50-1.90 (14H, m), 2.74 (1H, m), 7.10 (2H, dd, J = 1.8, 4.6 Hz), 8.46 (2H, dd, J = 1.8, 4.6 Hz); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 25.7, 26.2, 26.8, 33.6, 44.0, 122.5, 149.6, 158.8; HRMS (ESI-TOF) calcd for C<sub>13</sub>H<sub>20</sub>N (M+H)<sup>+</sup> 190.1590, found 190.1598.



(3) Bernardi, R.; Caronna, T.; Morrocchi, S.; Traldi, P.; Vittimberga, B. M. *J. Chem. Soc., Perkin Trans. 1* **1981**, 1607.

**4-methyl-4-(pyridin-4-yl)pentyl acetate (3hh):**

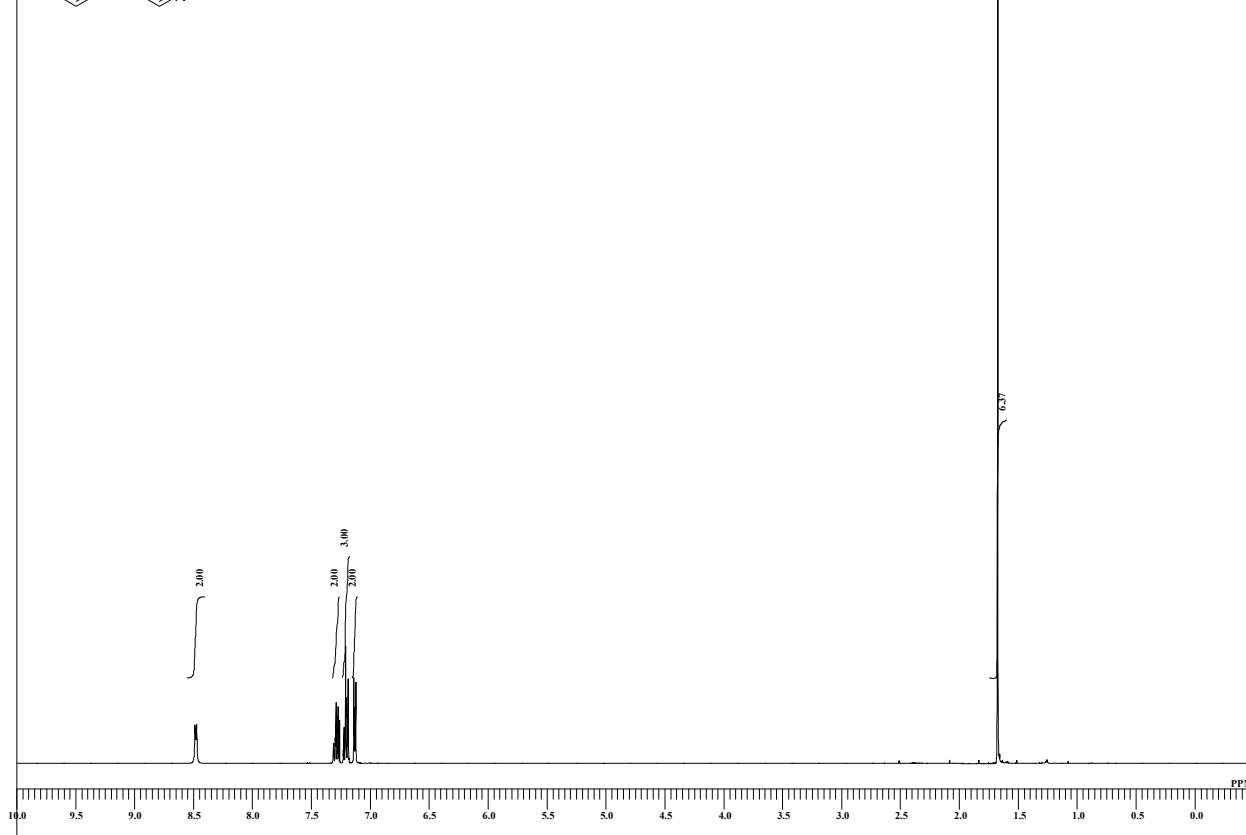
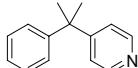
43% yield (14.5 mg); colorless oil; IR (neat) 1738, 1597, 1411, 1240, 823 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 1.31 (6H, s), 1.37 (2H, m), 1.65 (2H, m), 2.01 (3H, s), 3.96 (2H, t, *J* = 6.9 Hz), 7.23 (2H, dd, *J* = 1.8, 4.6 Hz), 8.52 (2H, dd, *J* = 1.8, 4.6 Hz); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 20.9, 24.0, 28.0, 37.5, 39.7, 64.6, 121.3, 149.4, 158.4, 171.1; HRMS (ESI-TOF) calcd for C<sub>13</sub>H<sub>20</sub>NO<sub>2</sub> (M+H)<sup>+</sup> 222.1489, found 222.1491.



TH-12-121-f21-31-2nd-H

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**3a**



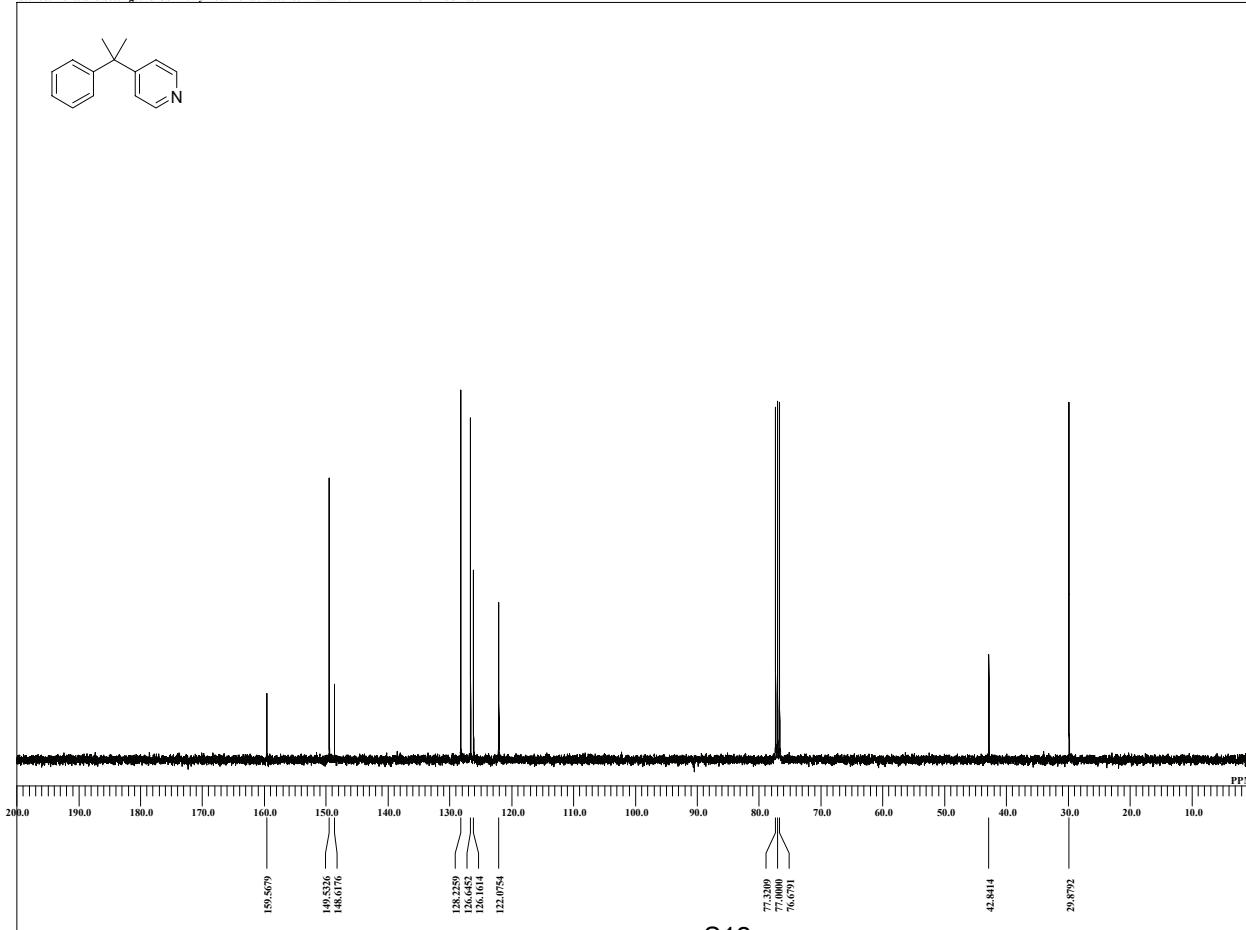
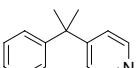
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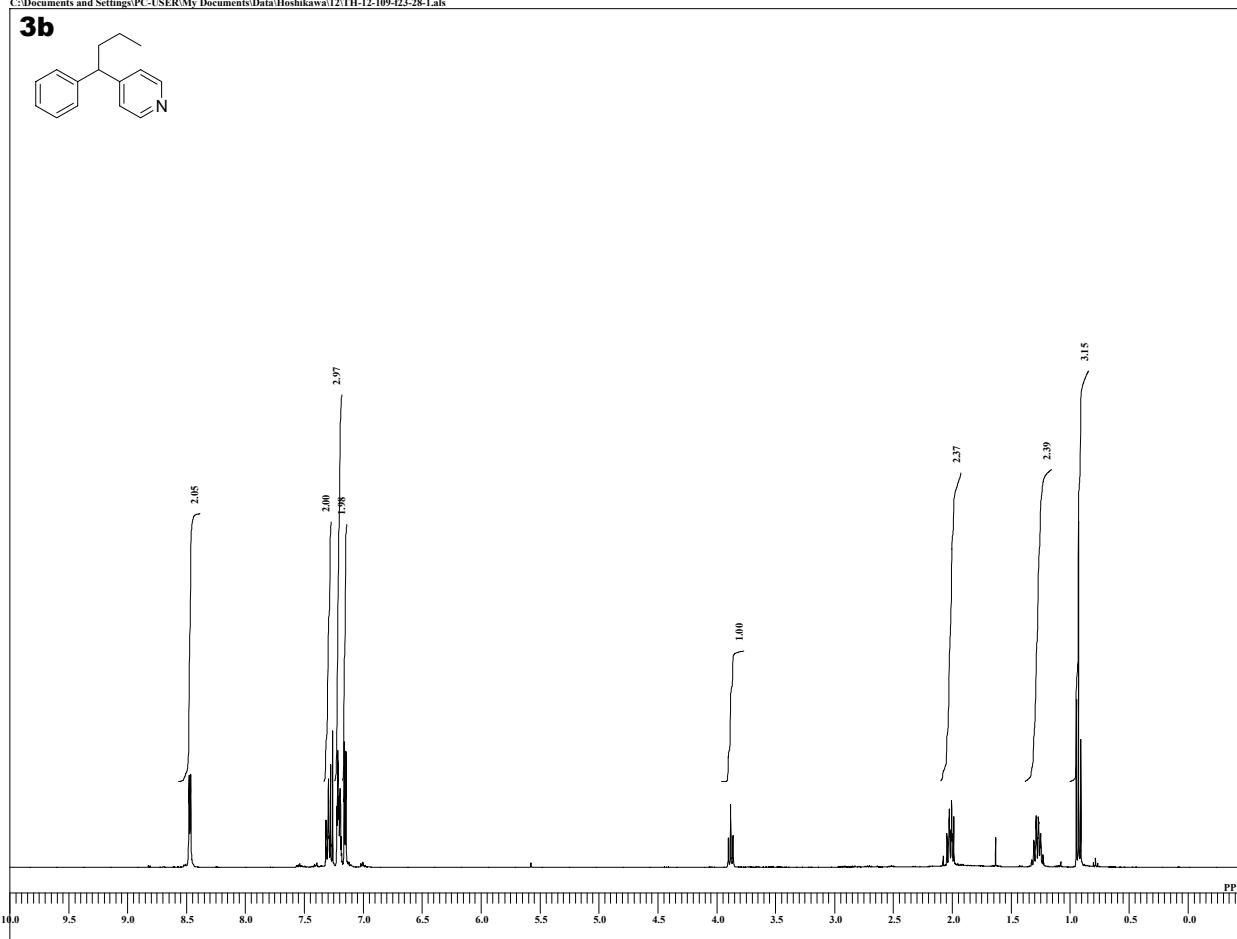
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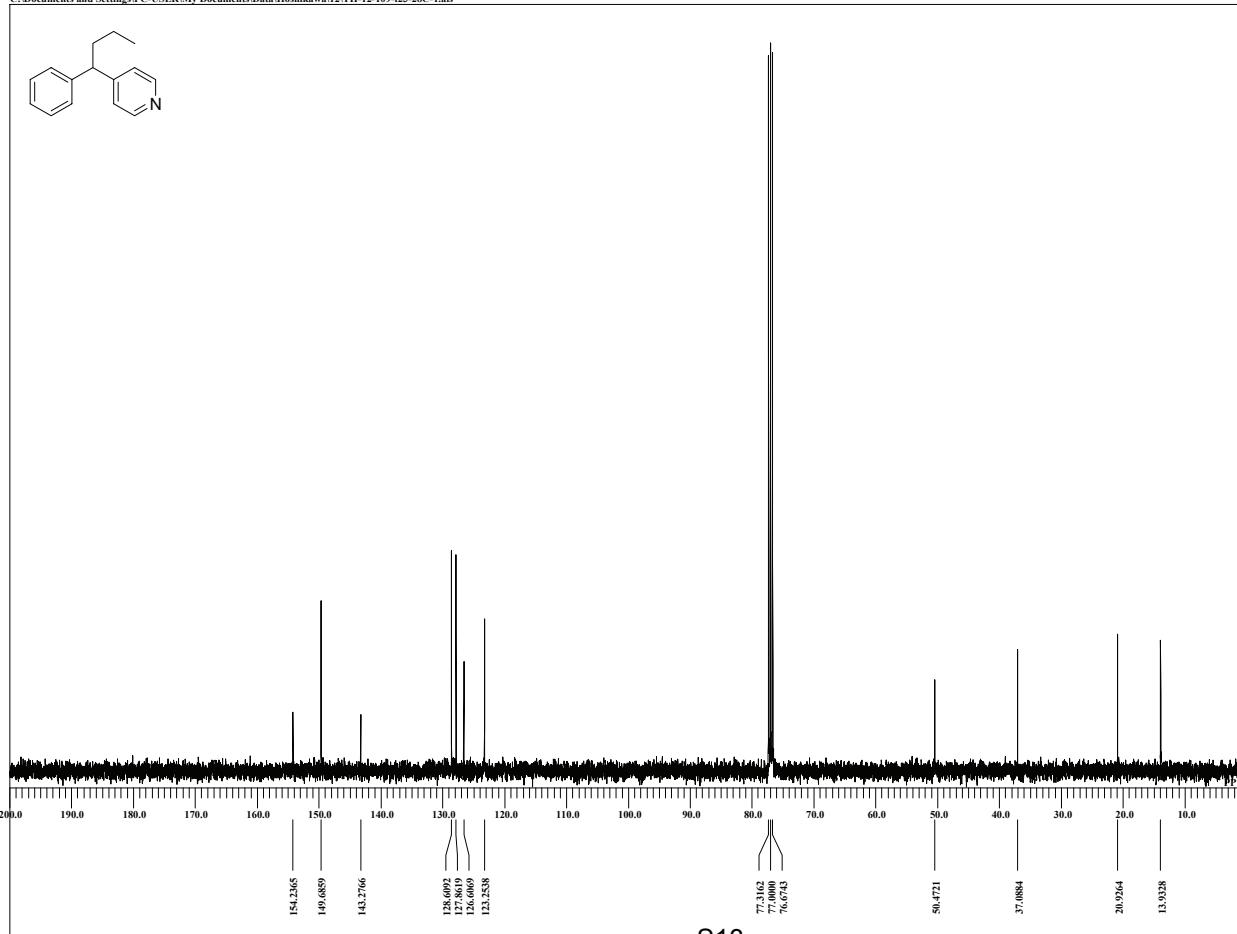
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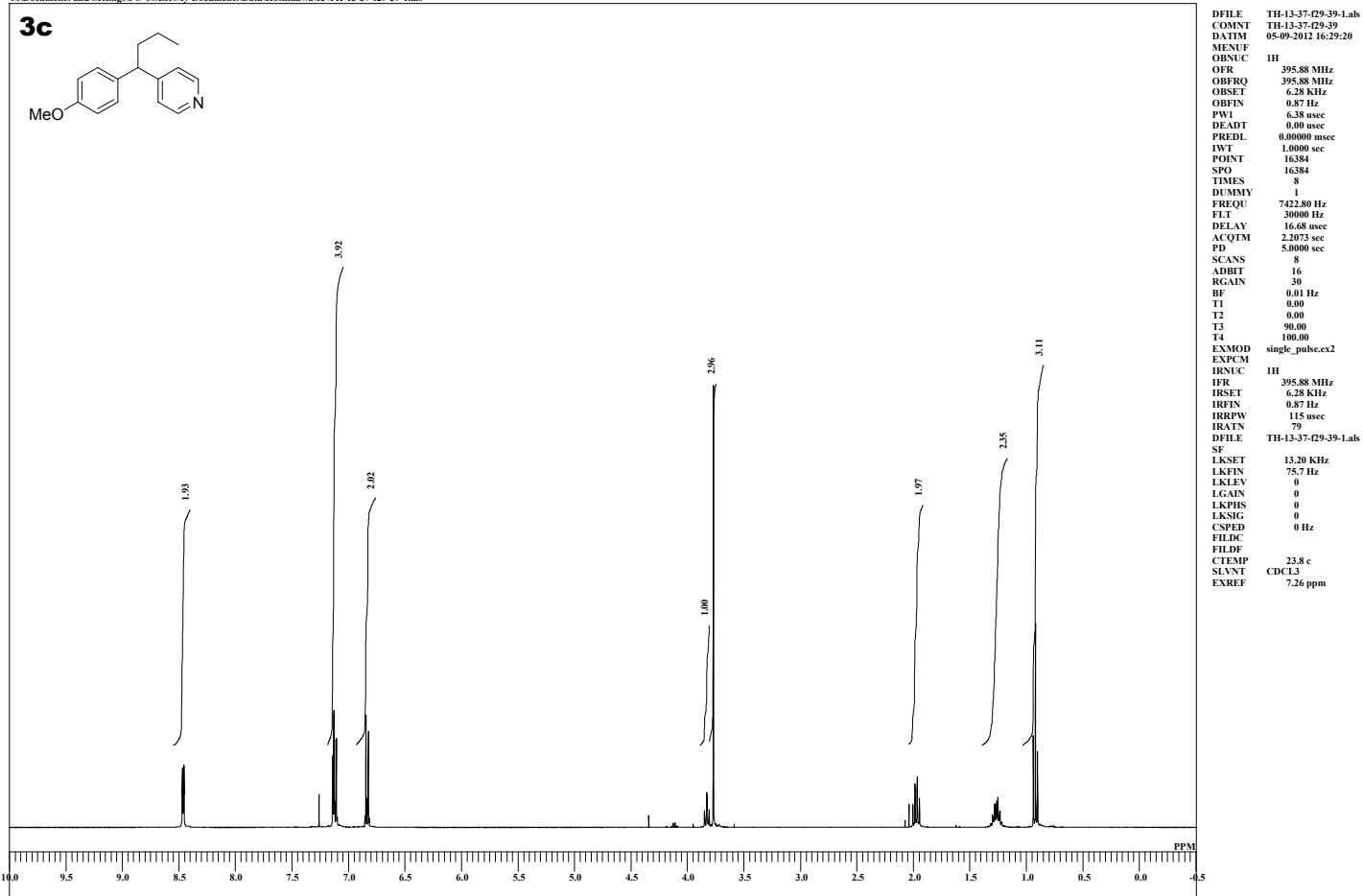
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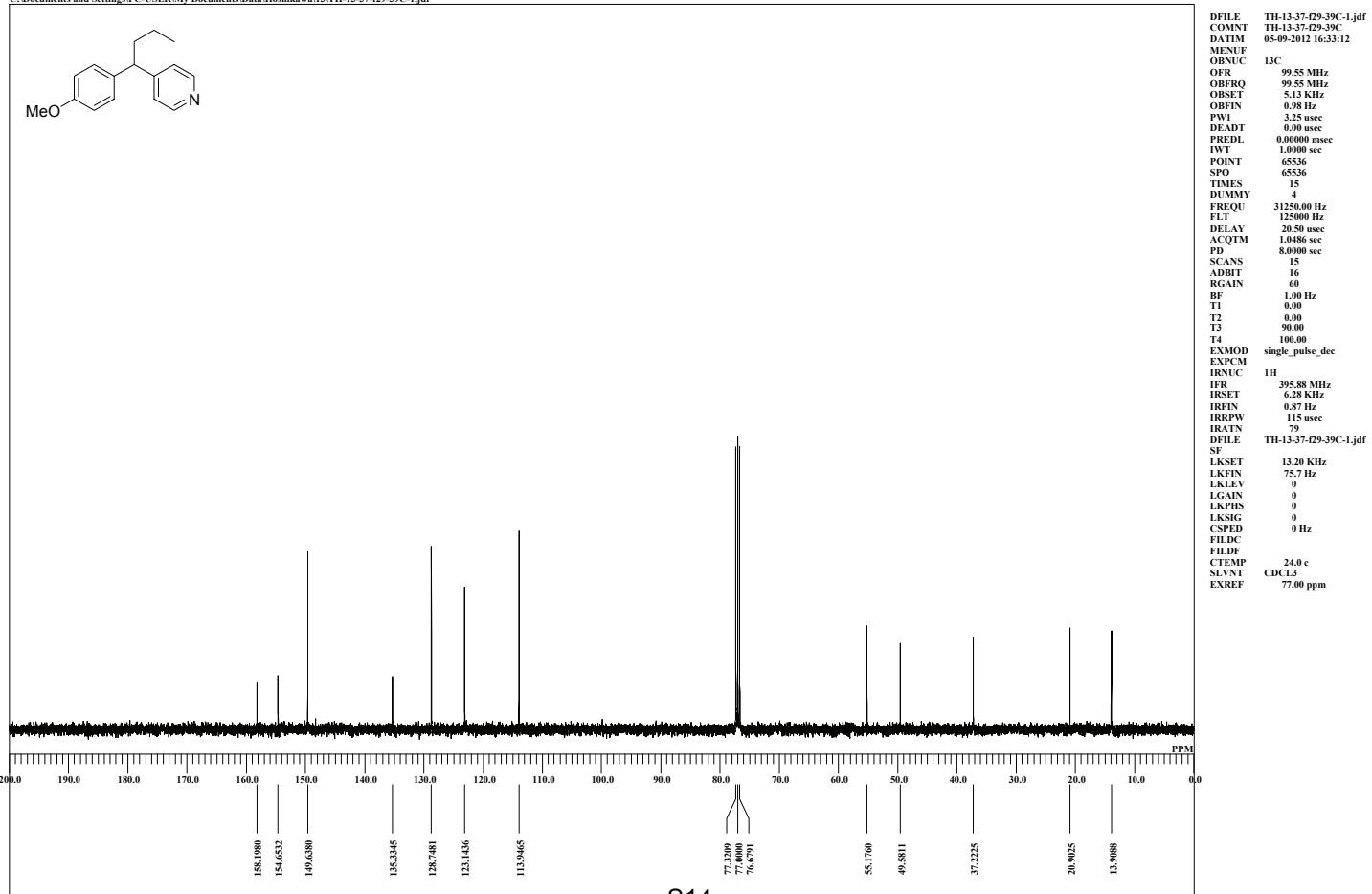
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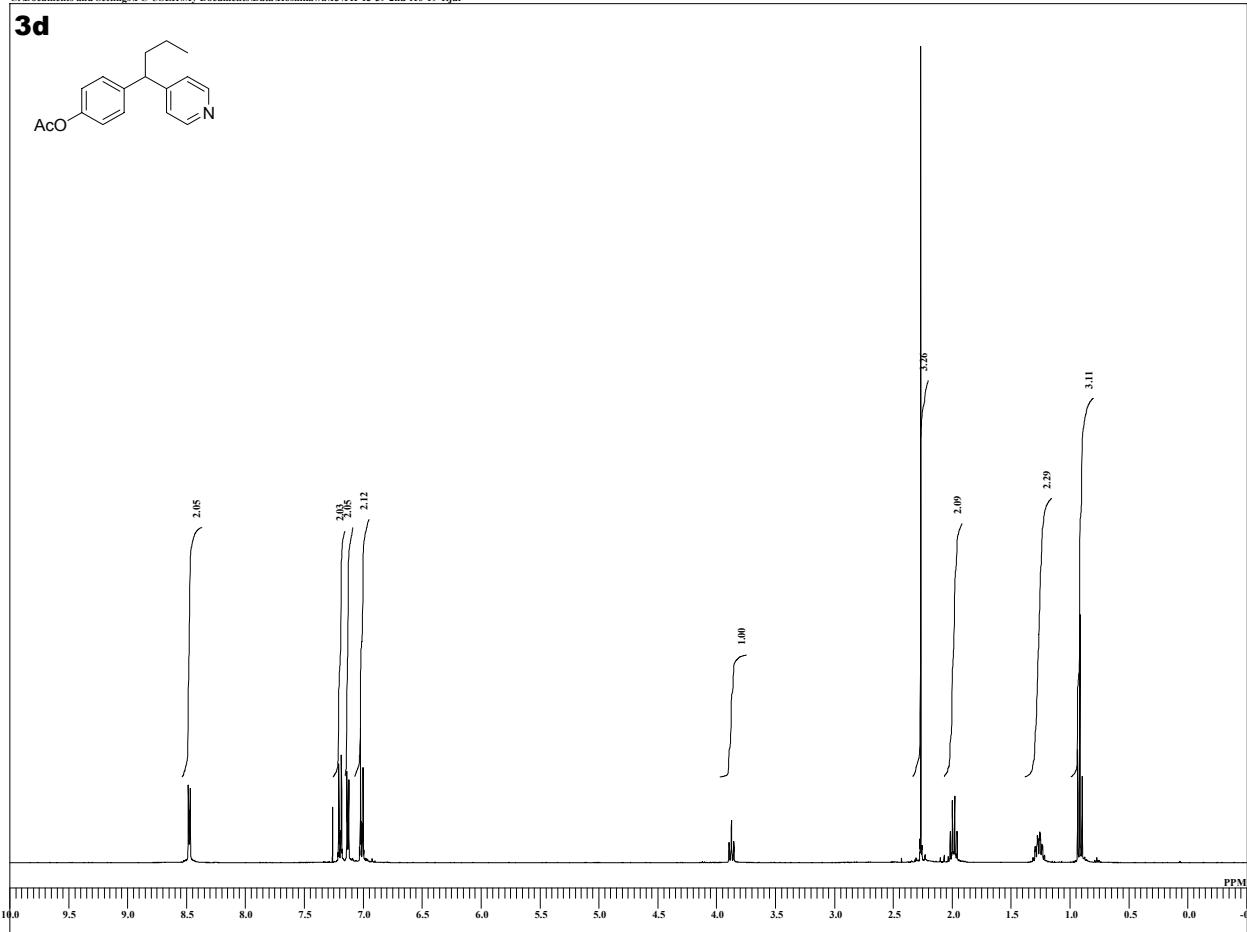
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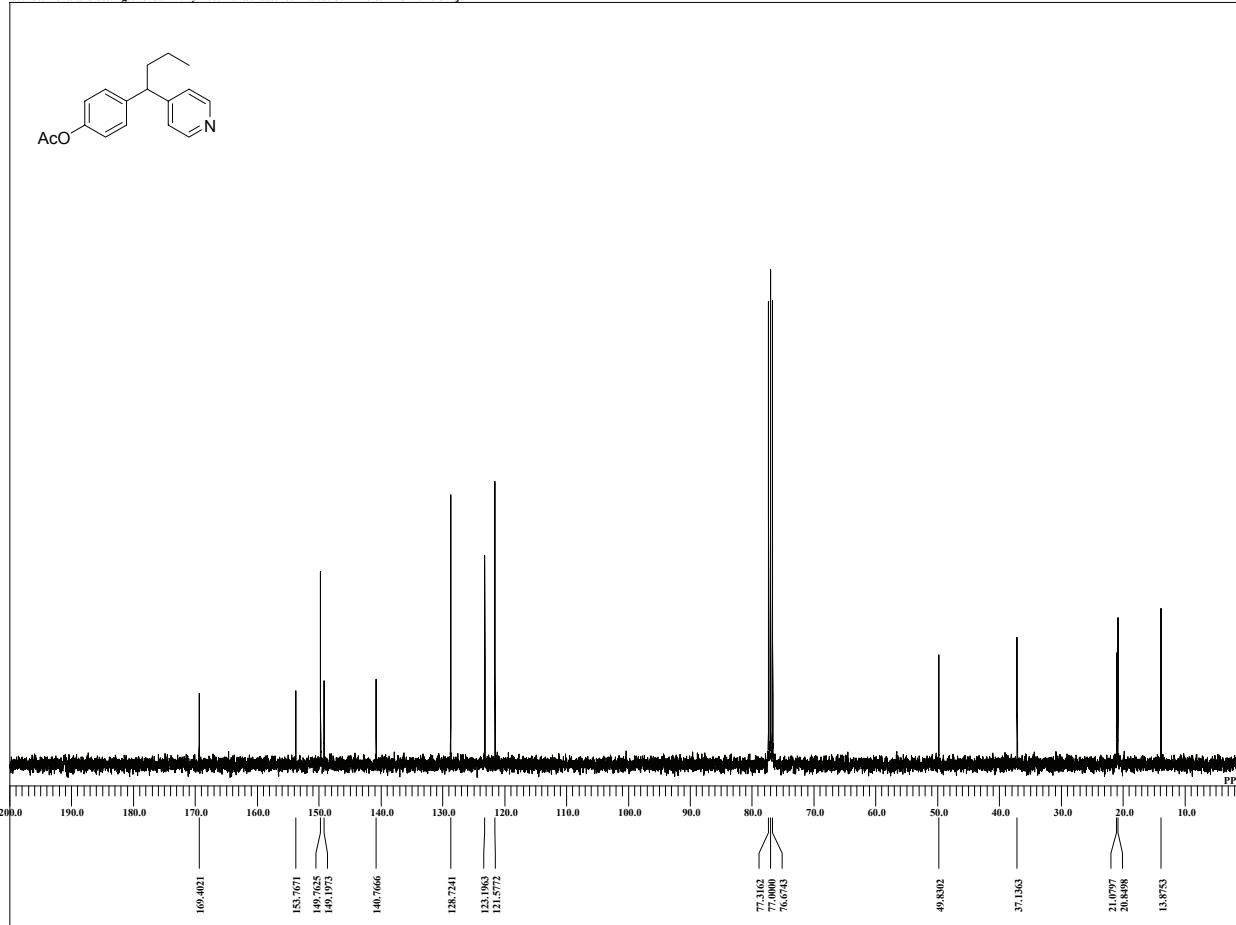
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TH-13-39-2nd-f10-19C

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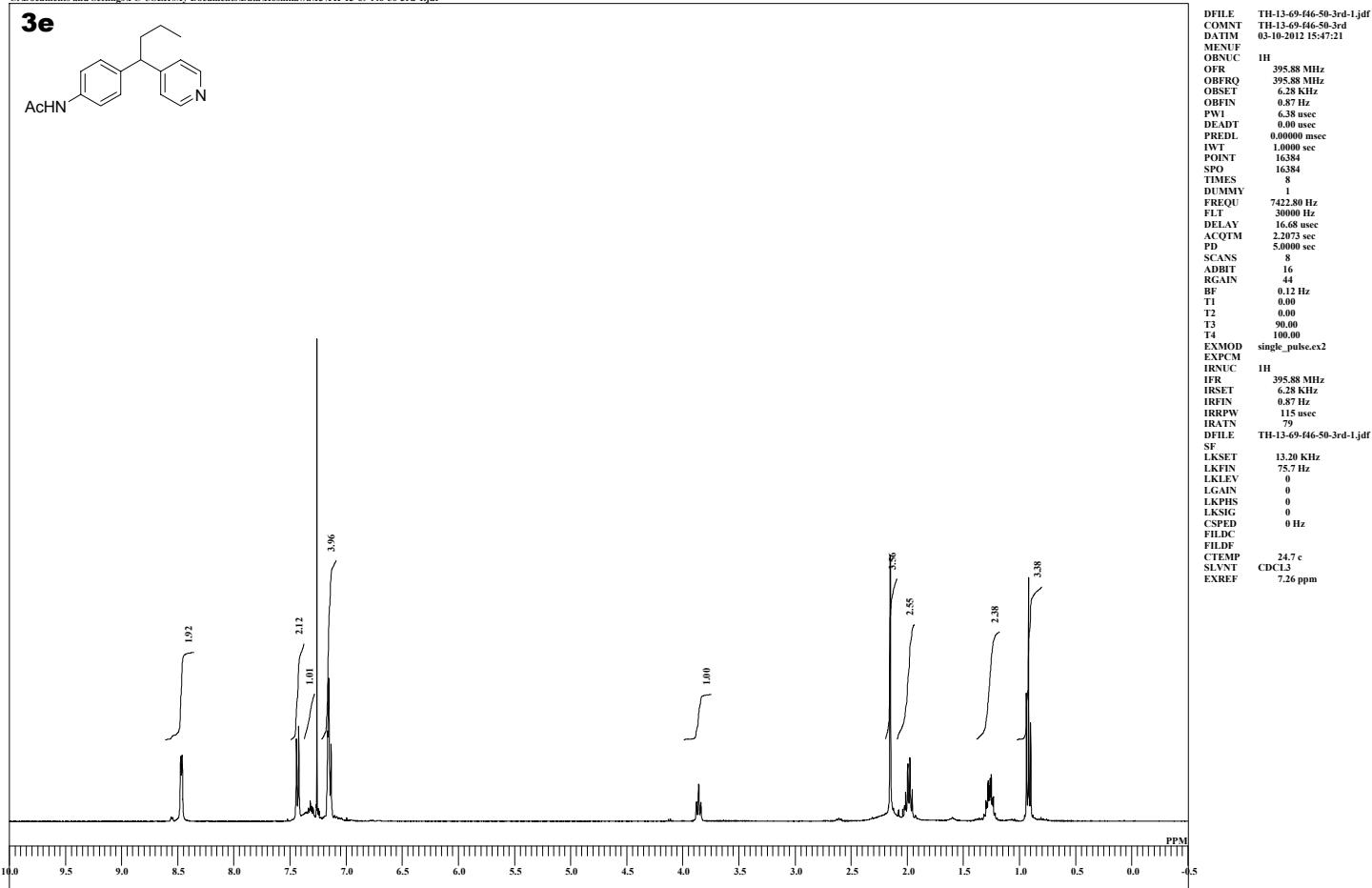
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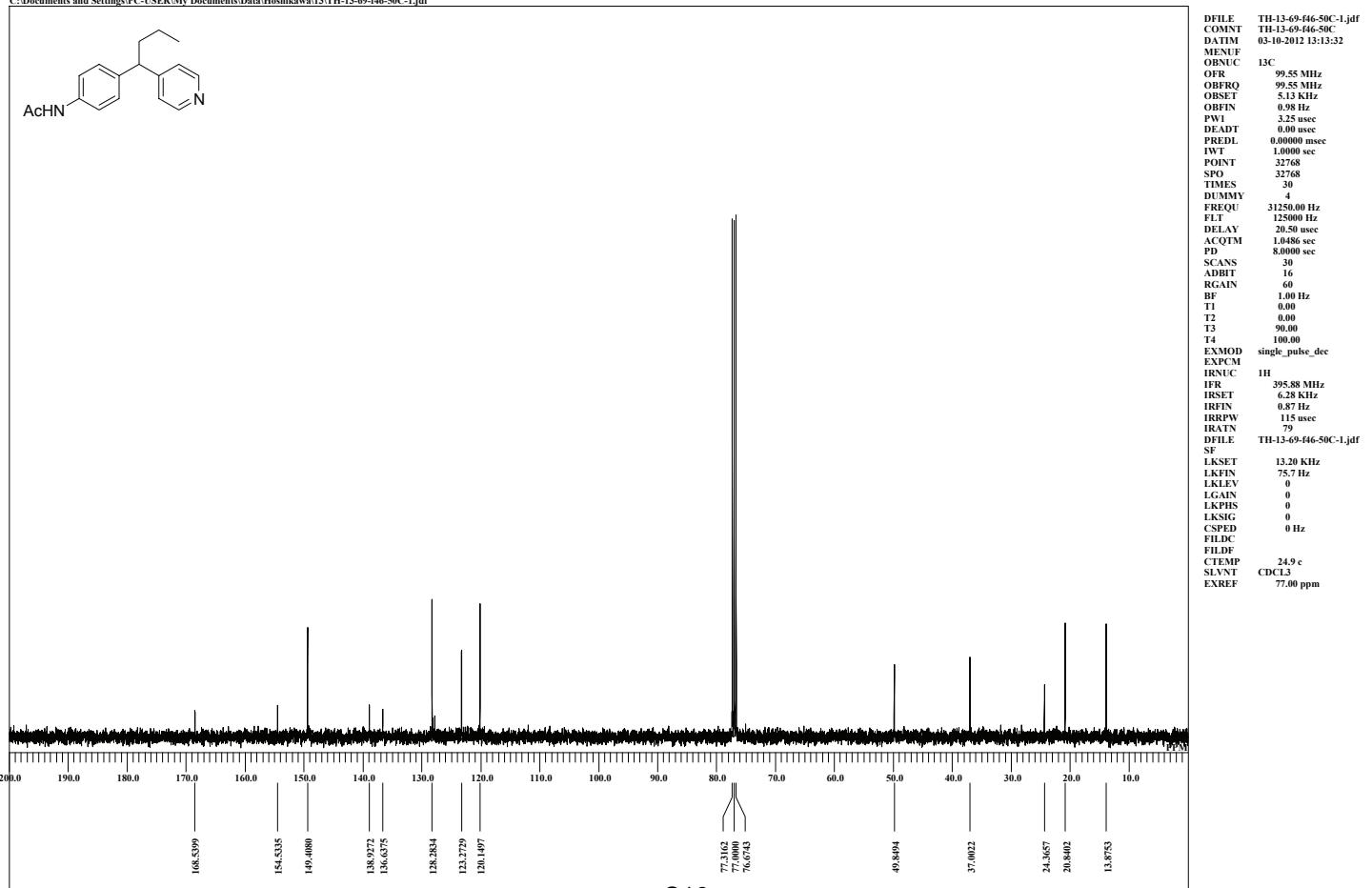
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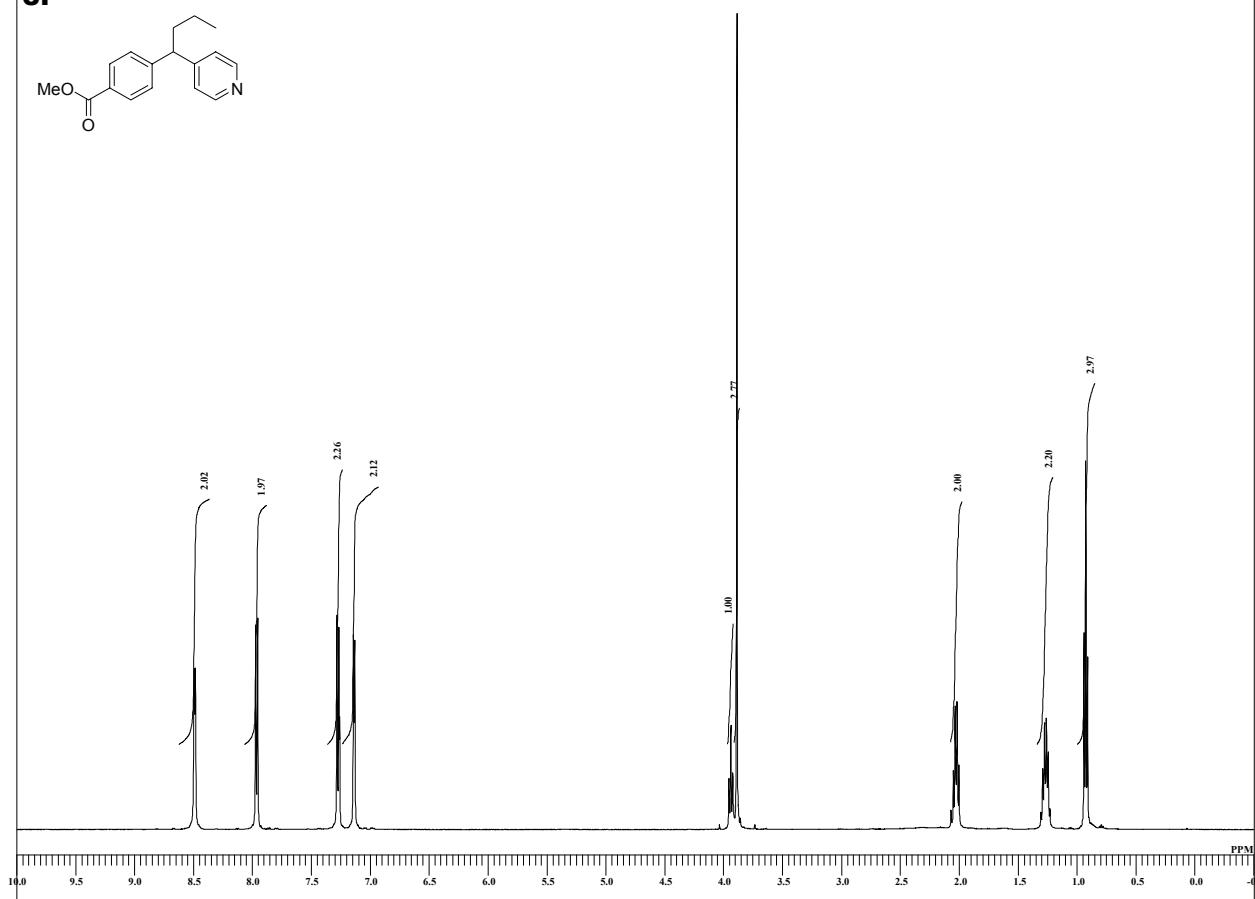
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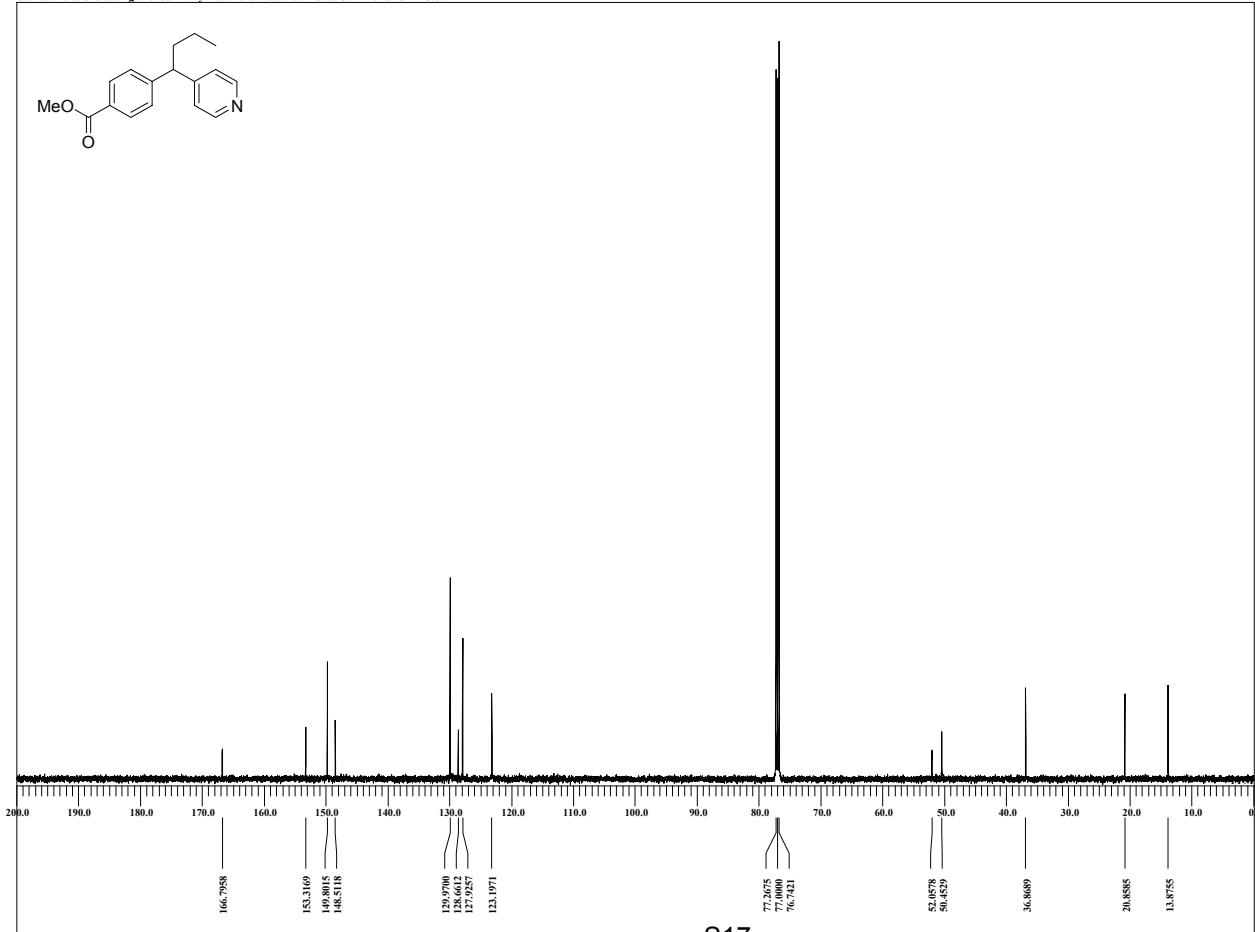
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**3f**



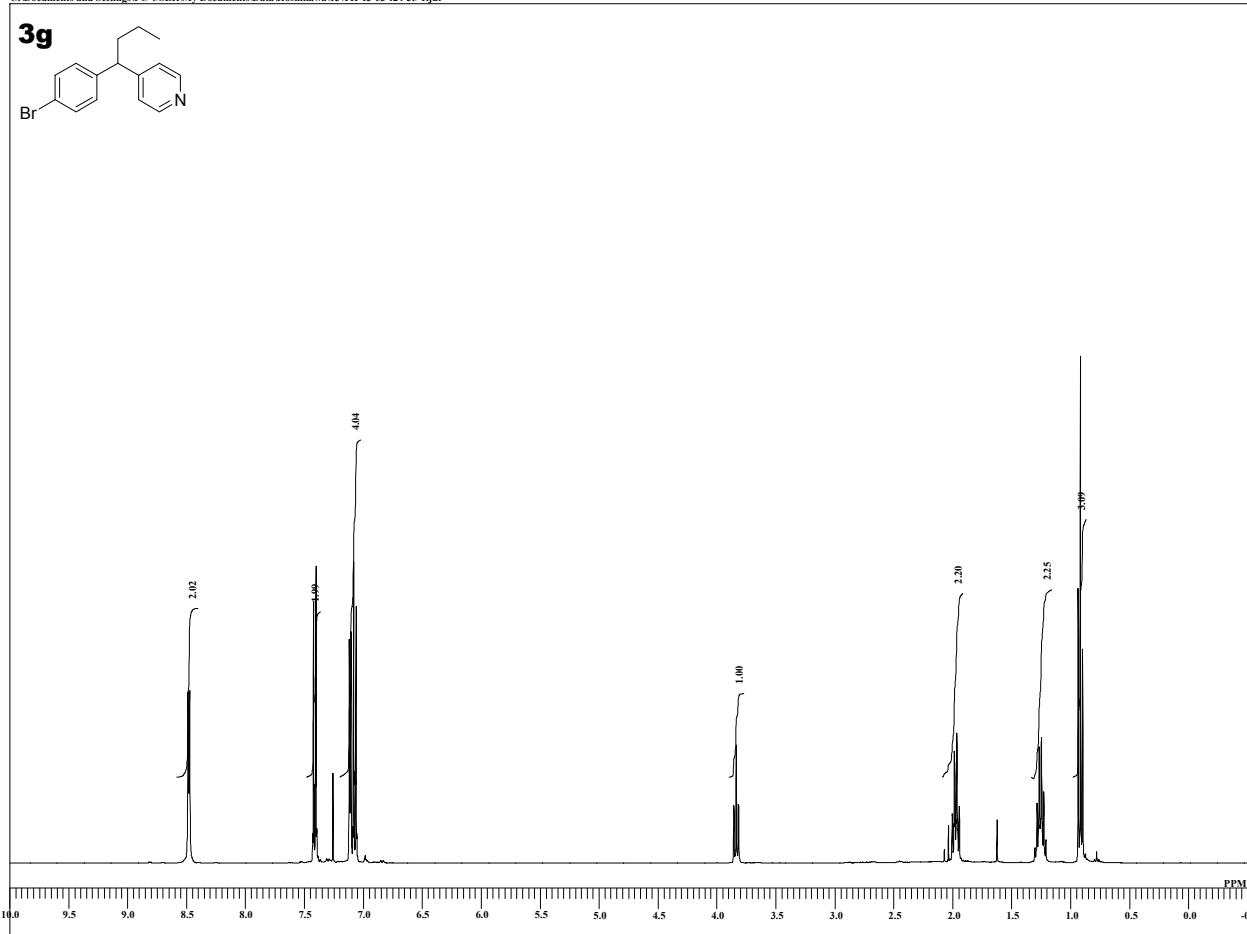
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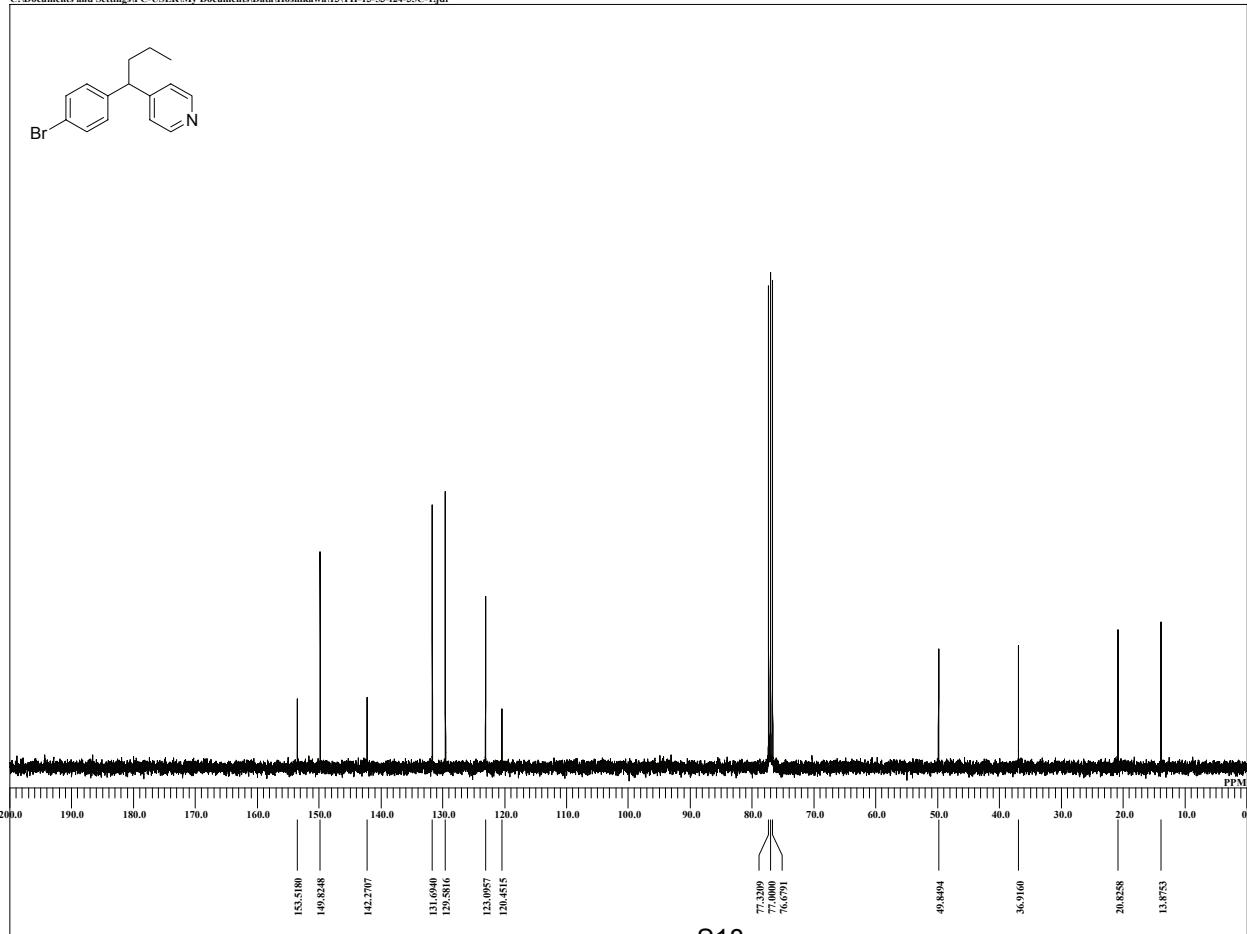
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TH-13-53-f24-35C

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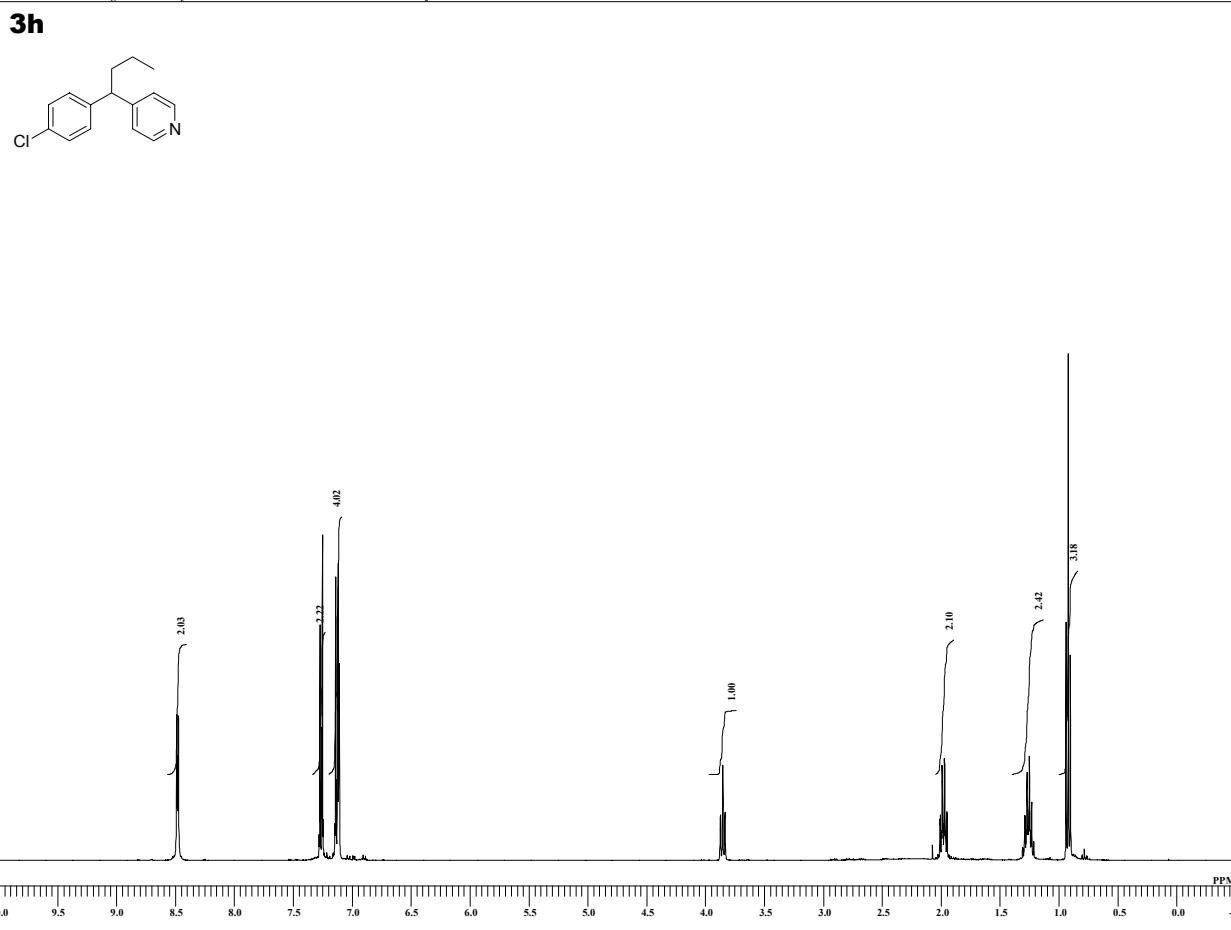
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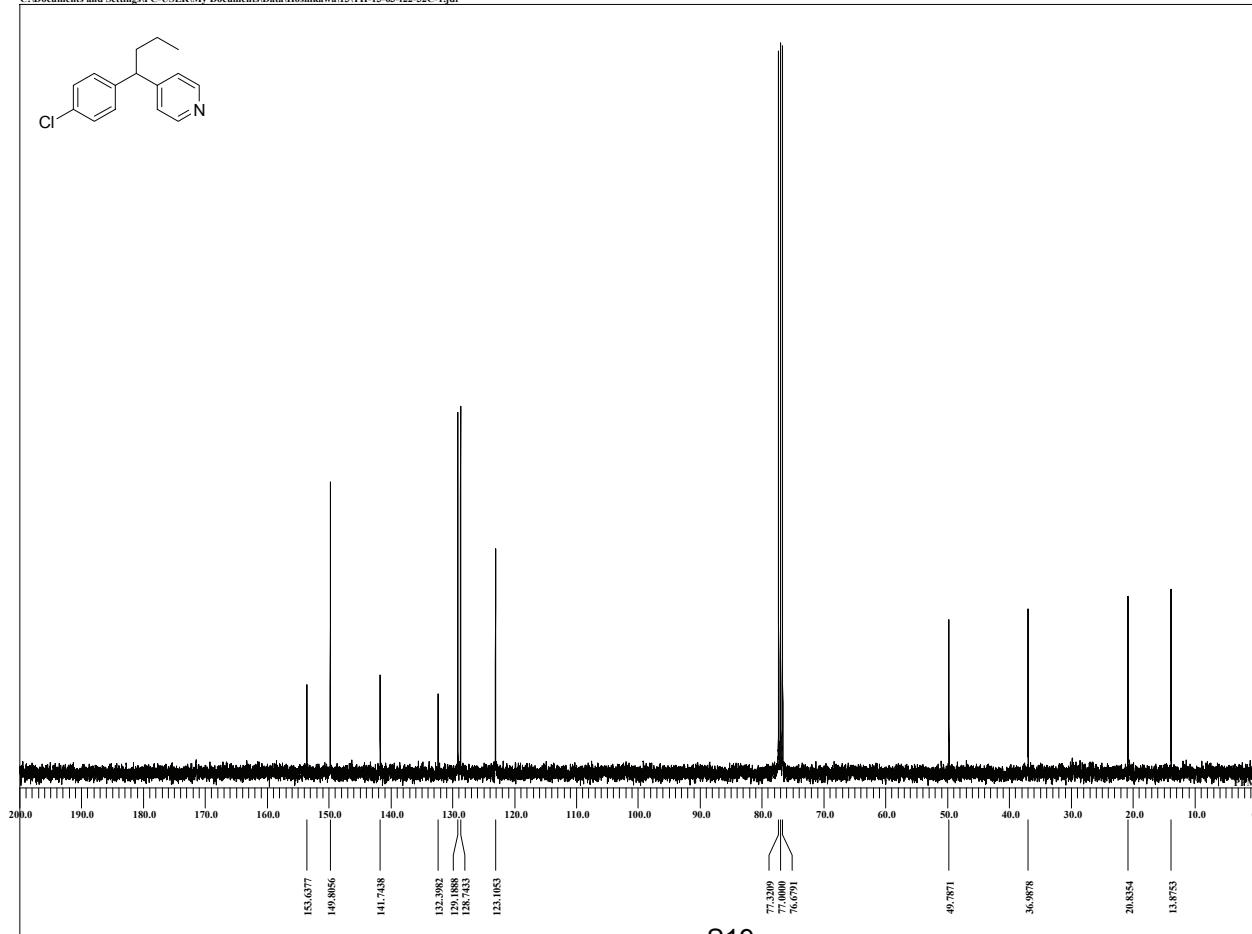
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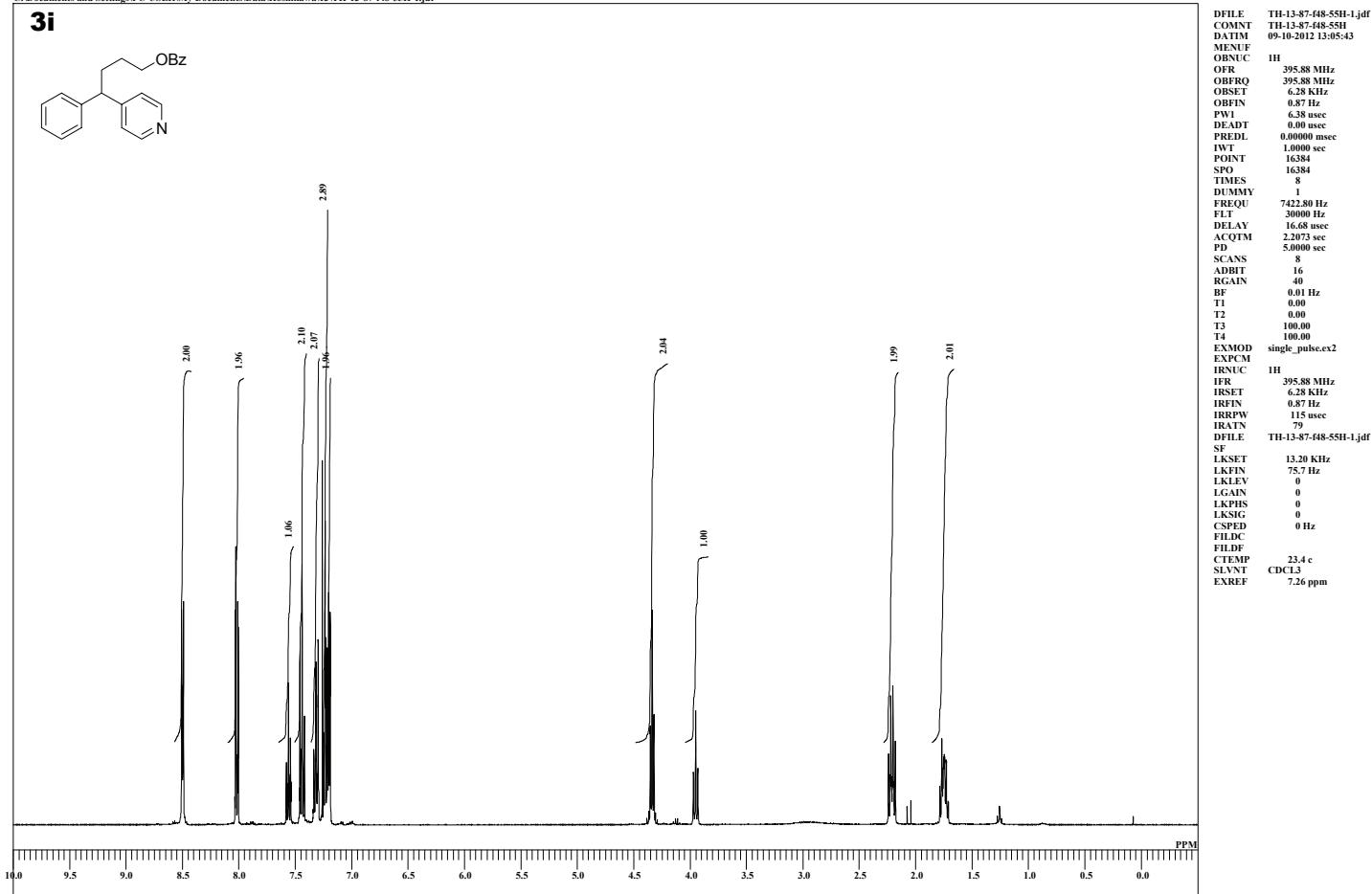
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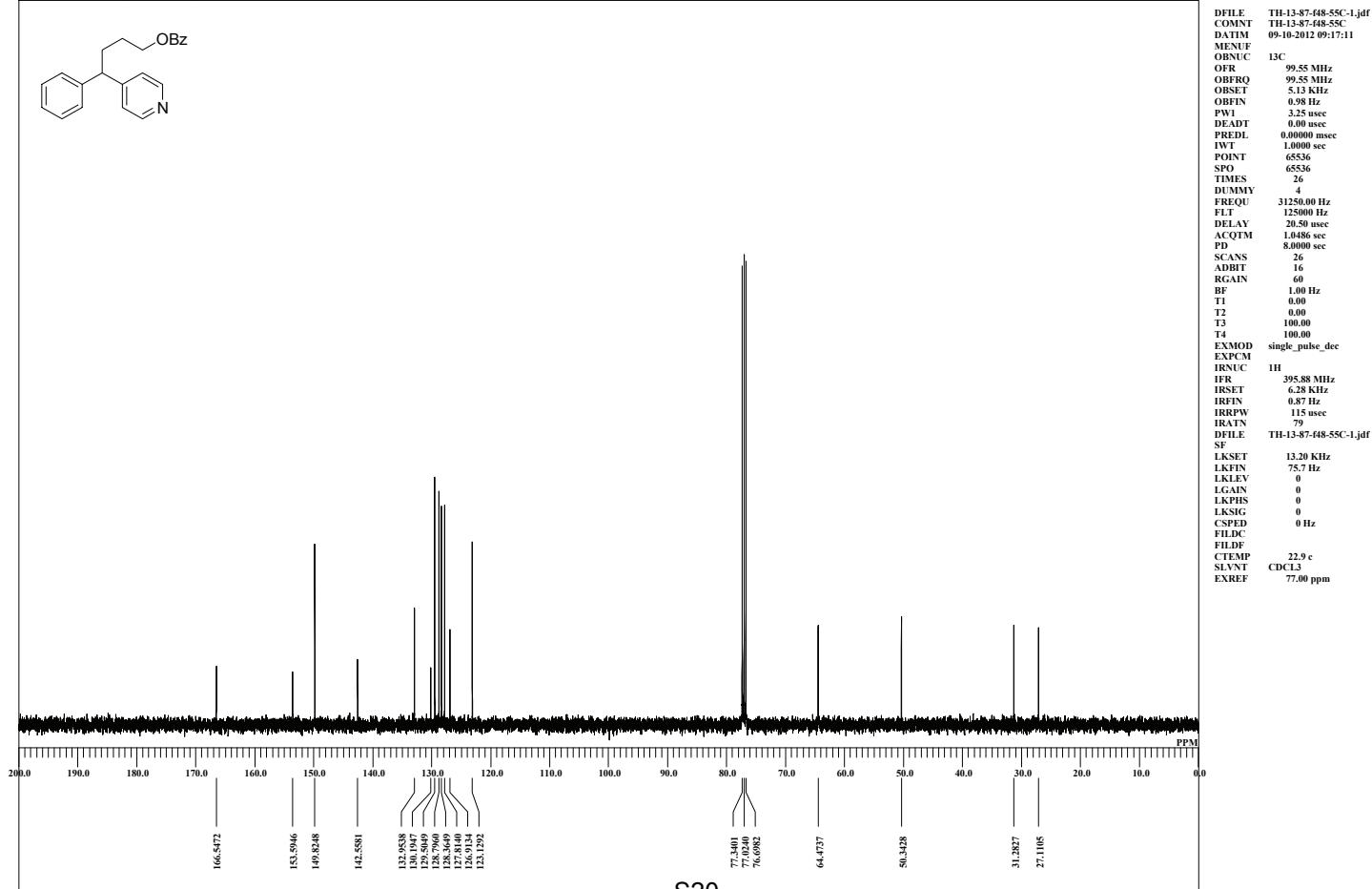
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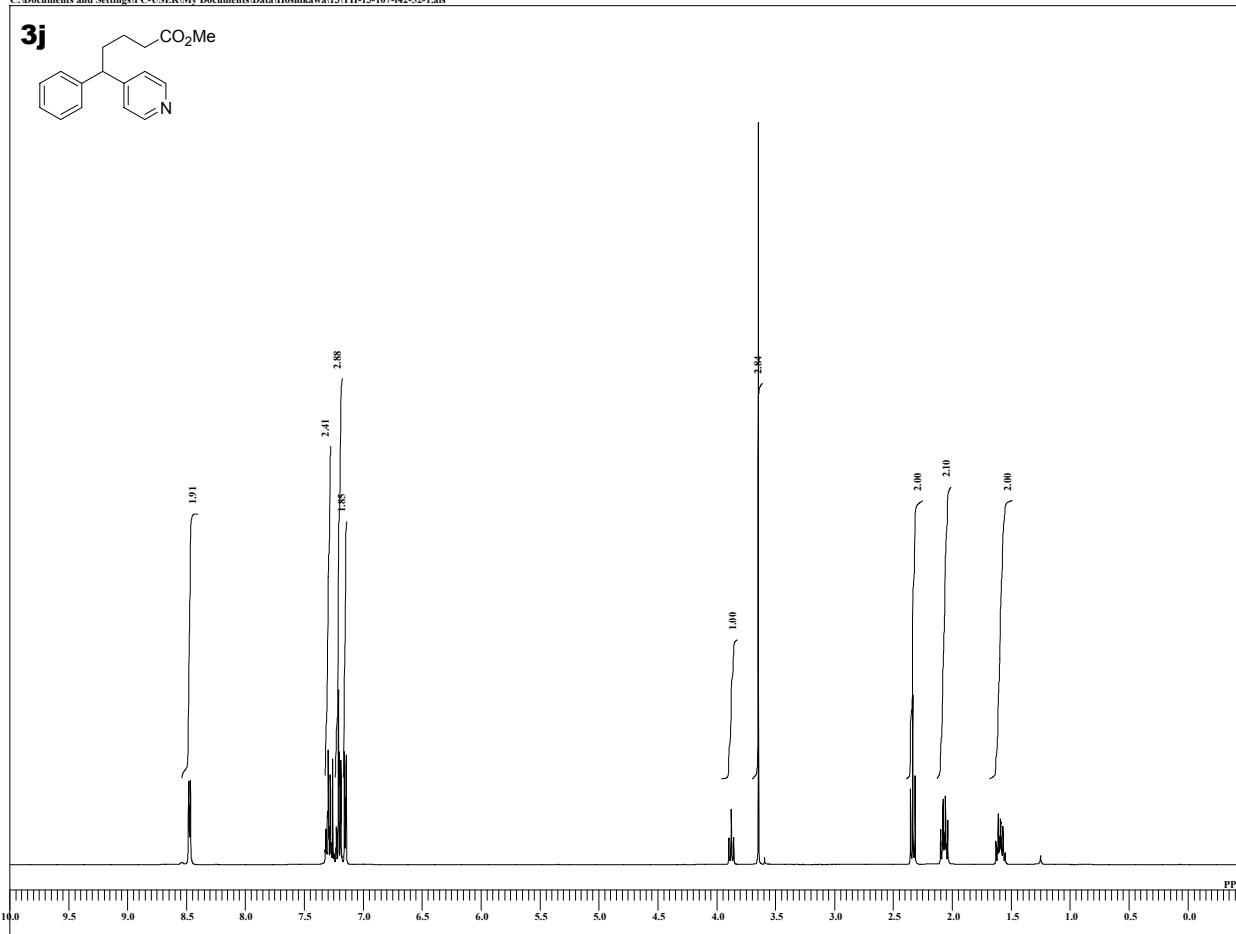
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TH-13-107-f42-52

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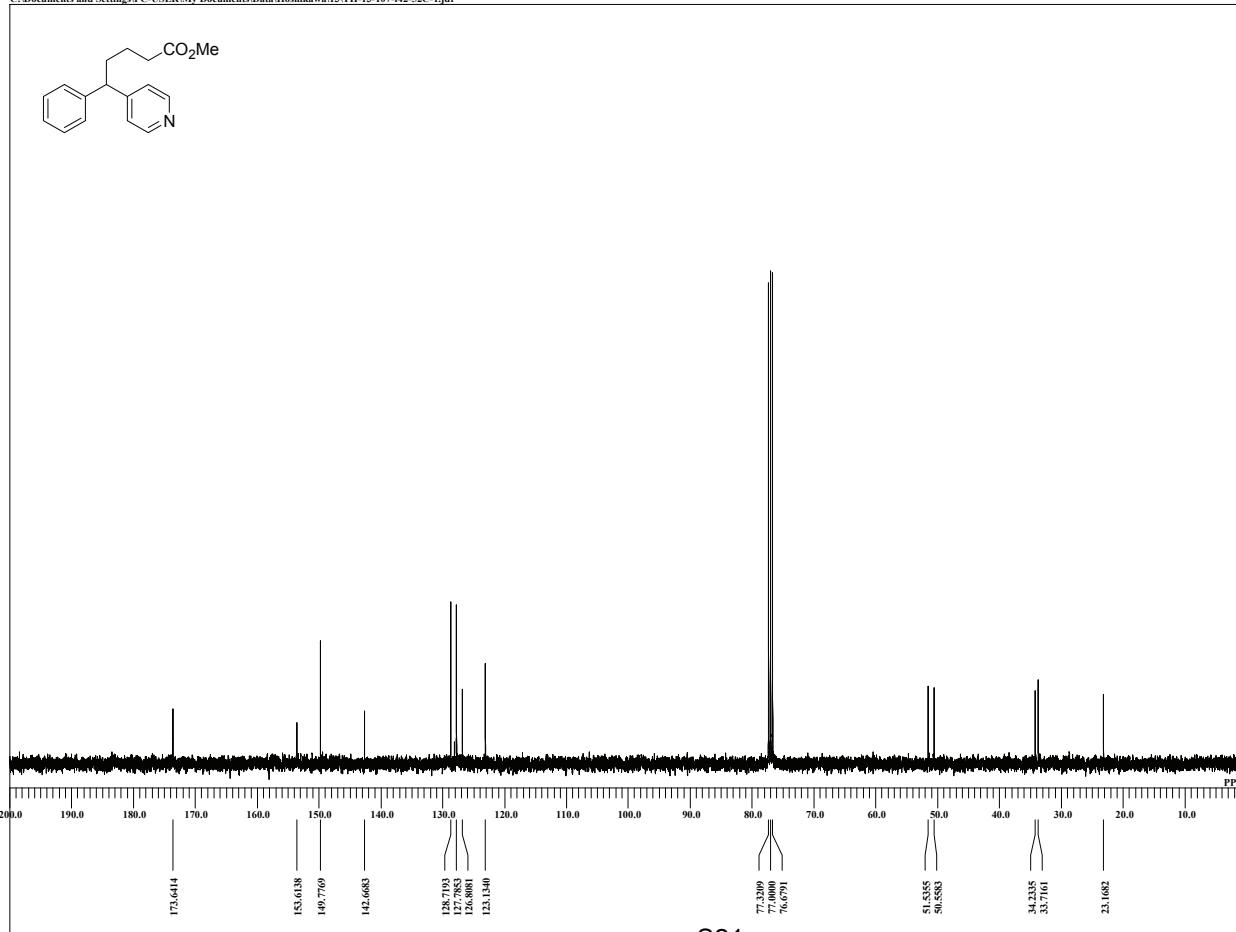
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T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
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TH-13-107-f42-52C

C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\13\TH-13-107-f42-52C-1.jdf



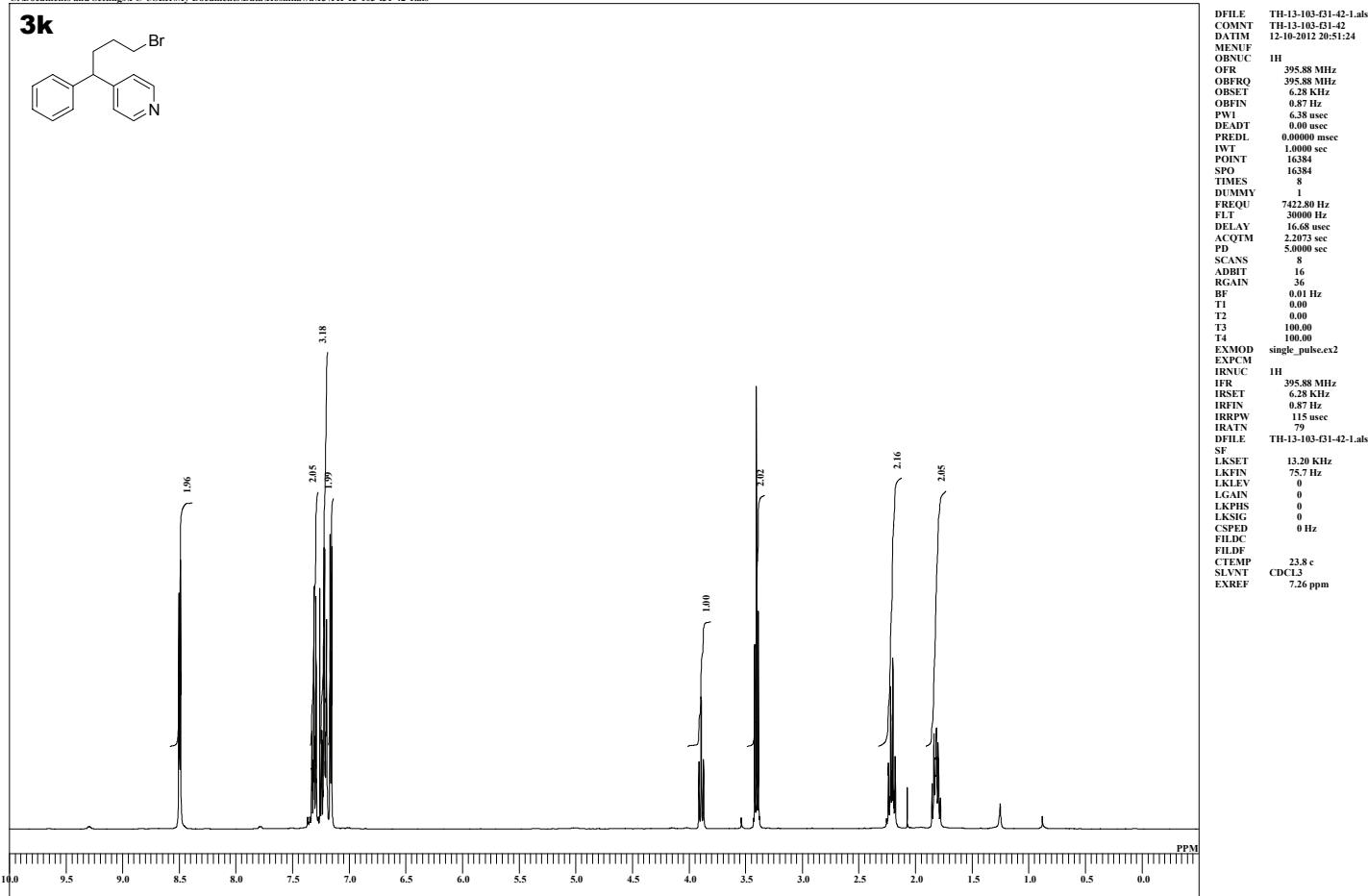
```

DFILE TH-13-107-f42-52C-1.jdf
COMNT TH-13-107-f42-52C
DATIM 16-10-2012 09:43:56
MENUF
OBNUC
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 kHz
OBFIN 0.98 Hz
PWI 3.12 sec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 65536
TIMES 22
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 8.0000 sec
SCANS 22
ADBIT 16
RGAIN 60
BF 1.016 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC IH
IFR 395.88 MHz
IRSET 6.28 kHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE TH-13-107-f42-52C-1.jdf
LKSET 13.20 kHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FLDC
FILDF
CTEMP 23.1 c
SLVNT CDCL3
EXREF 77.00 ppm

```

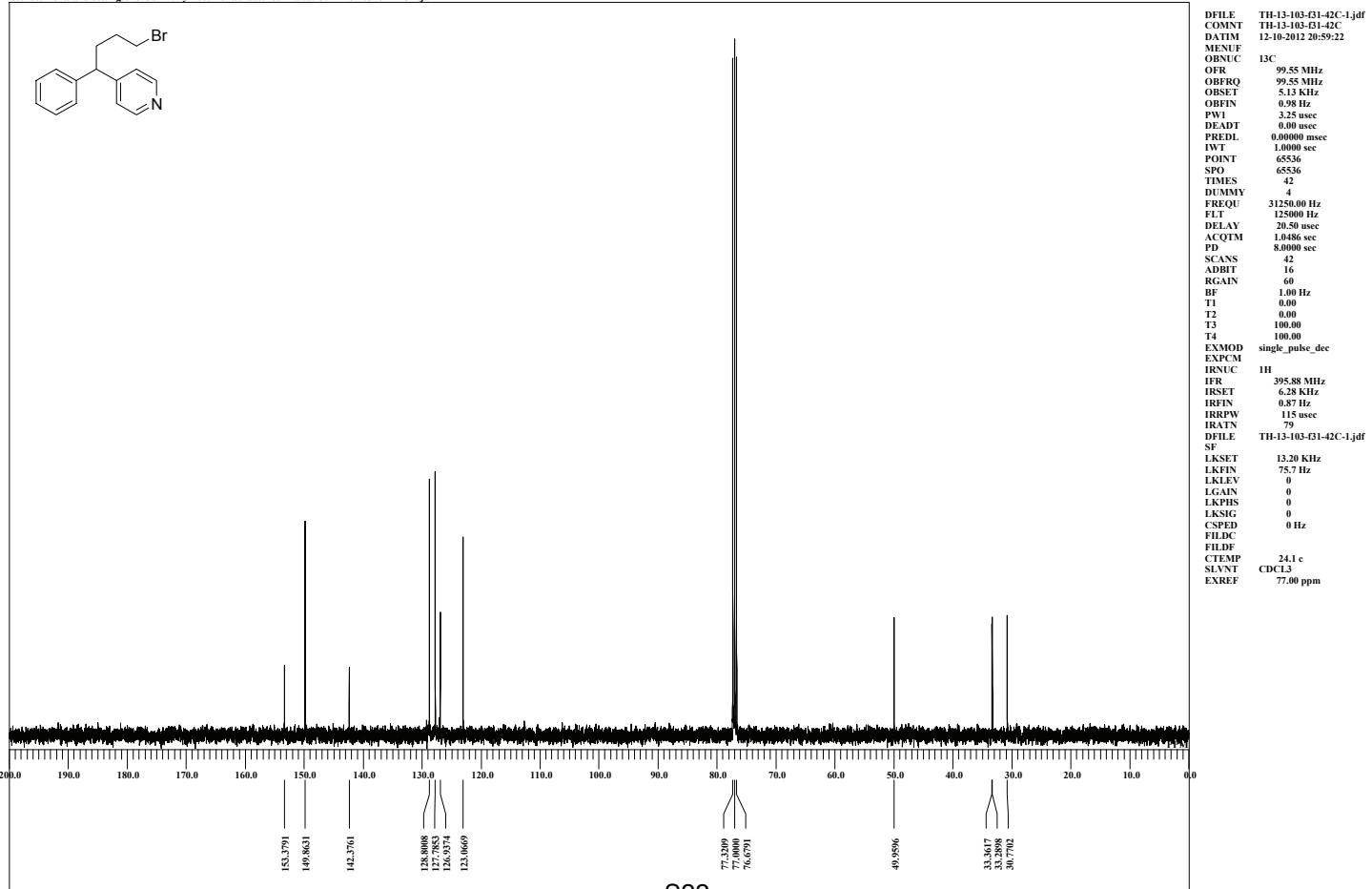
TH-13-103-f31-42

C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\13\TH-13-103-f31-42-1.als



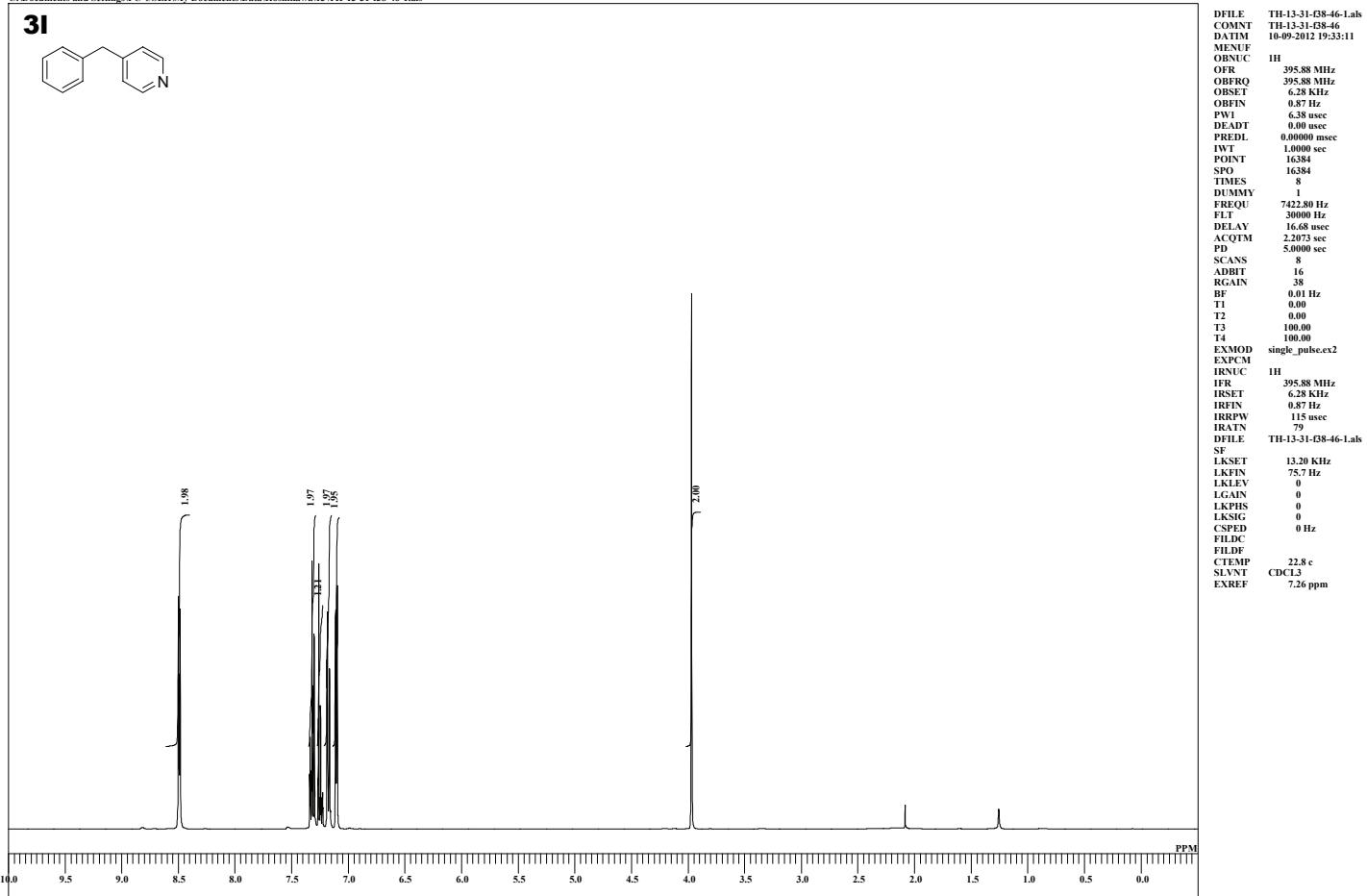
TH-13-103-f31-42C

C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\13\TH-13-103-f31-42C-1.jdf



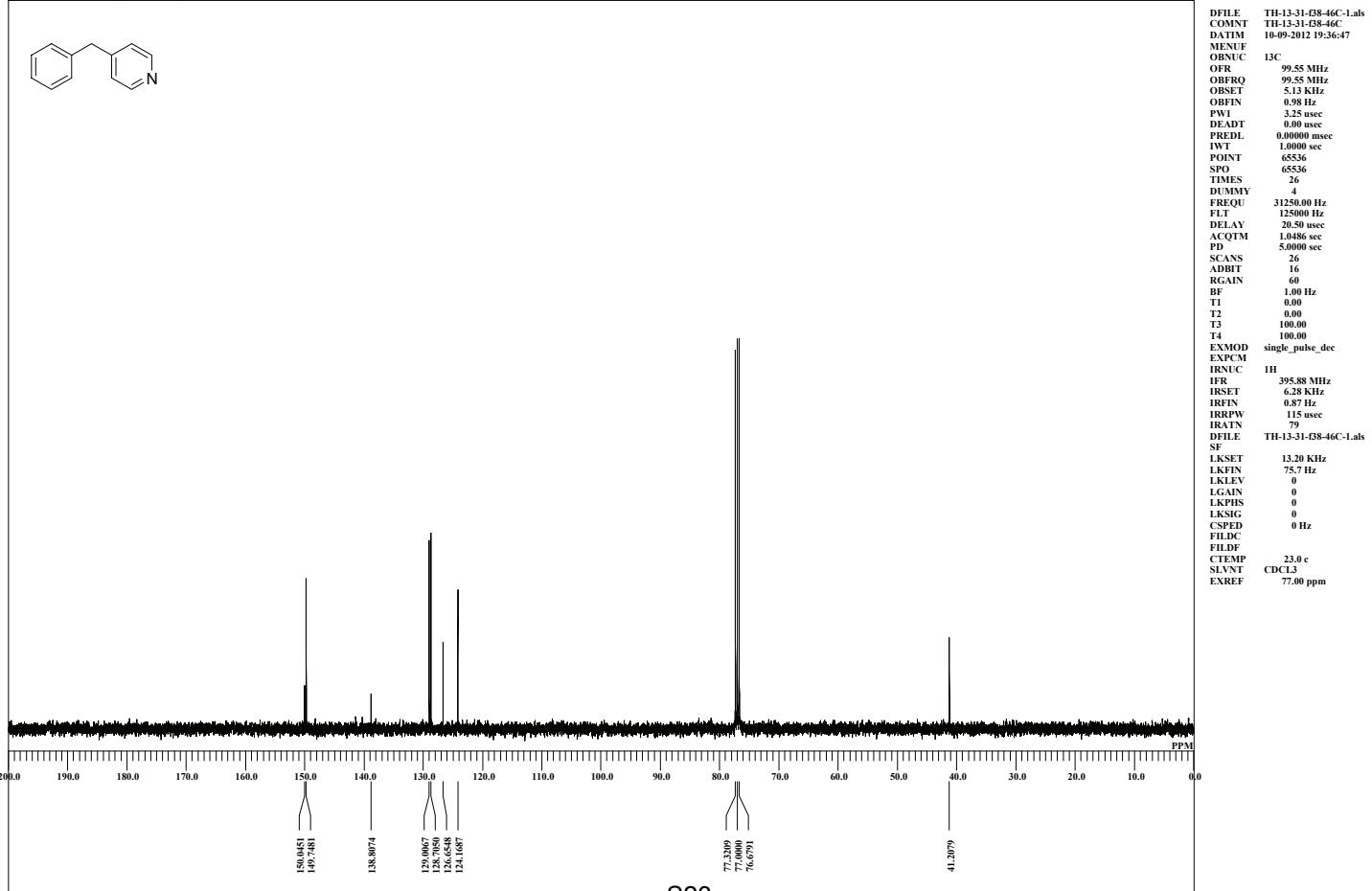
TH-13-31-f38-46

C:\Documents and Settings\PC-USER\My Documents\DATA\Hoshikawa\13\TH-13-31-f38-46-1.als



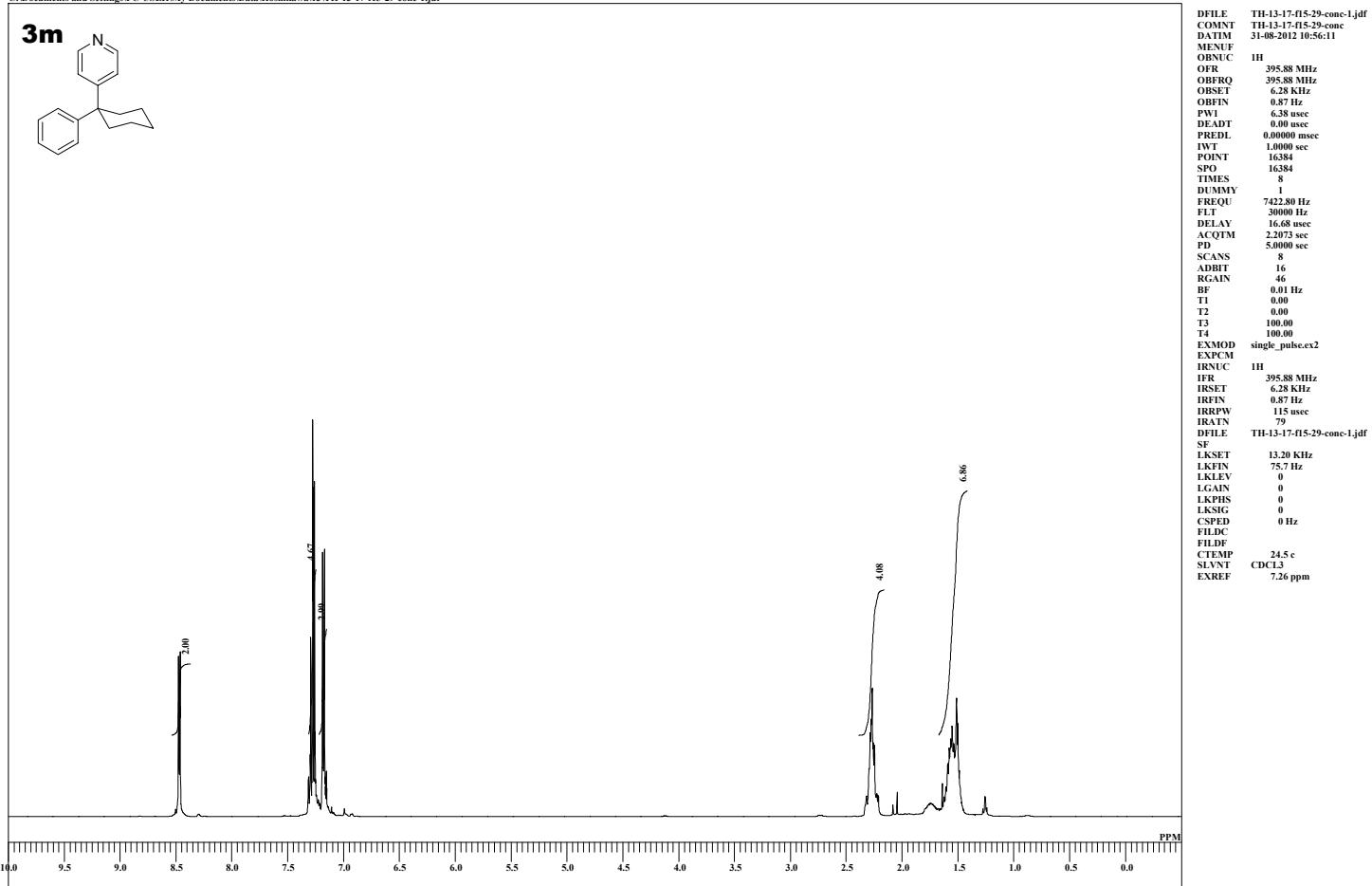
TH-13-31-f38-46C

C:\Documents and Settings\PC-USER\My Documents\DATA\Hoshikawa\13\TH-13-31-f38-46C-1.als



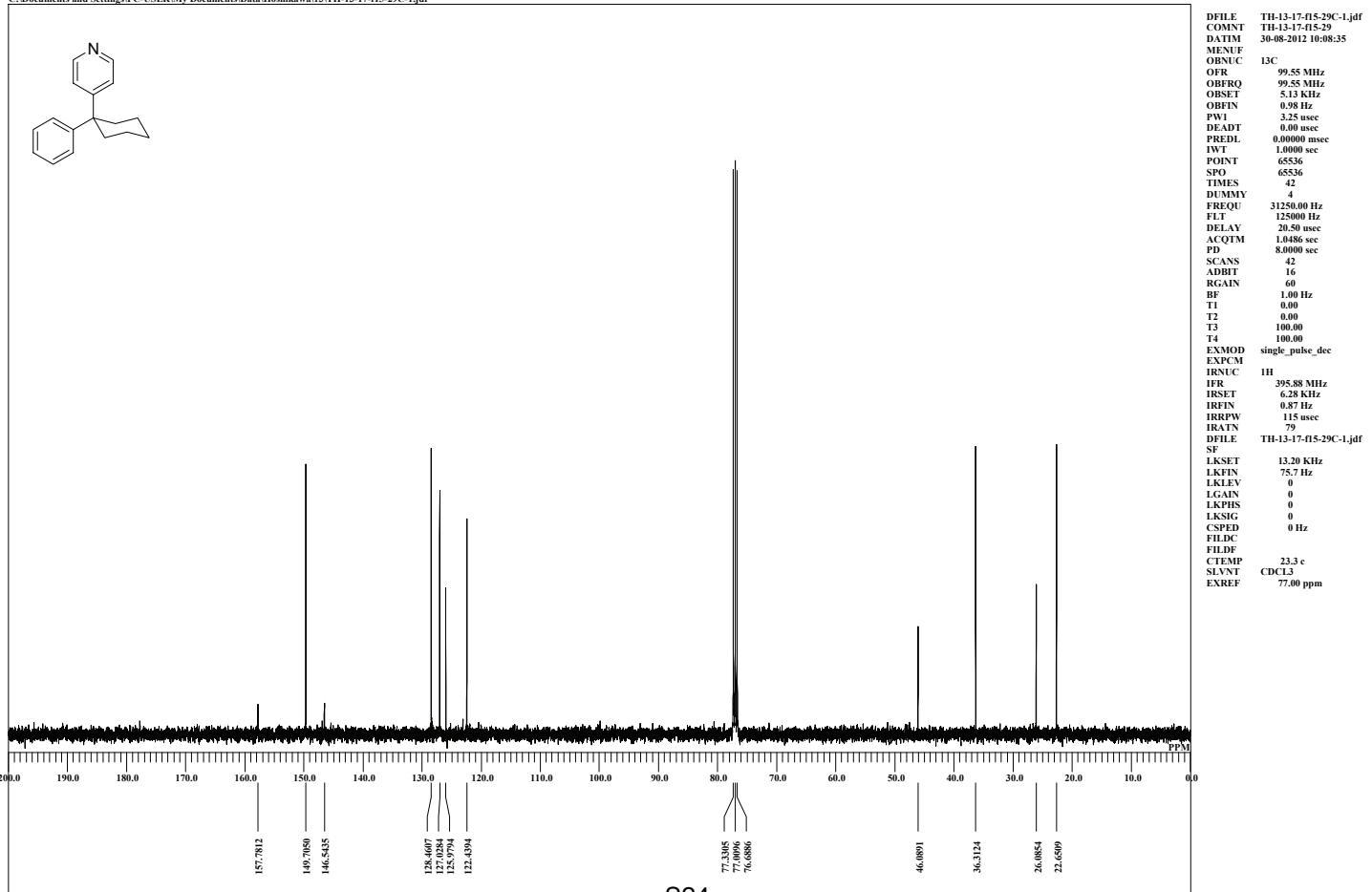
TH-13-17-f15-29-conc

C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\13\TH-13-17-f15-29-conc-1.jdf



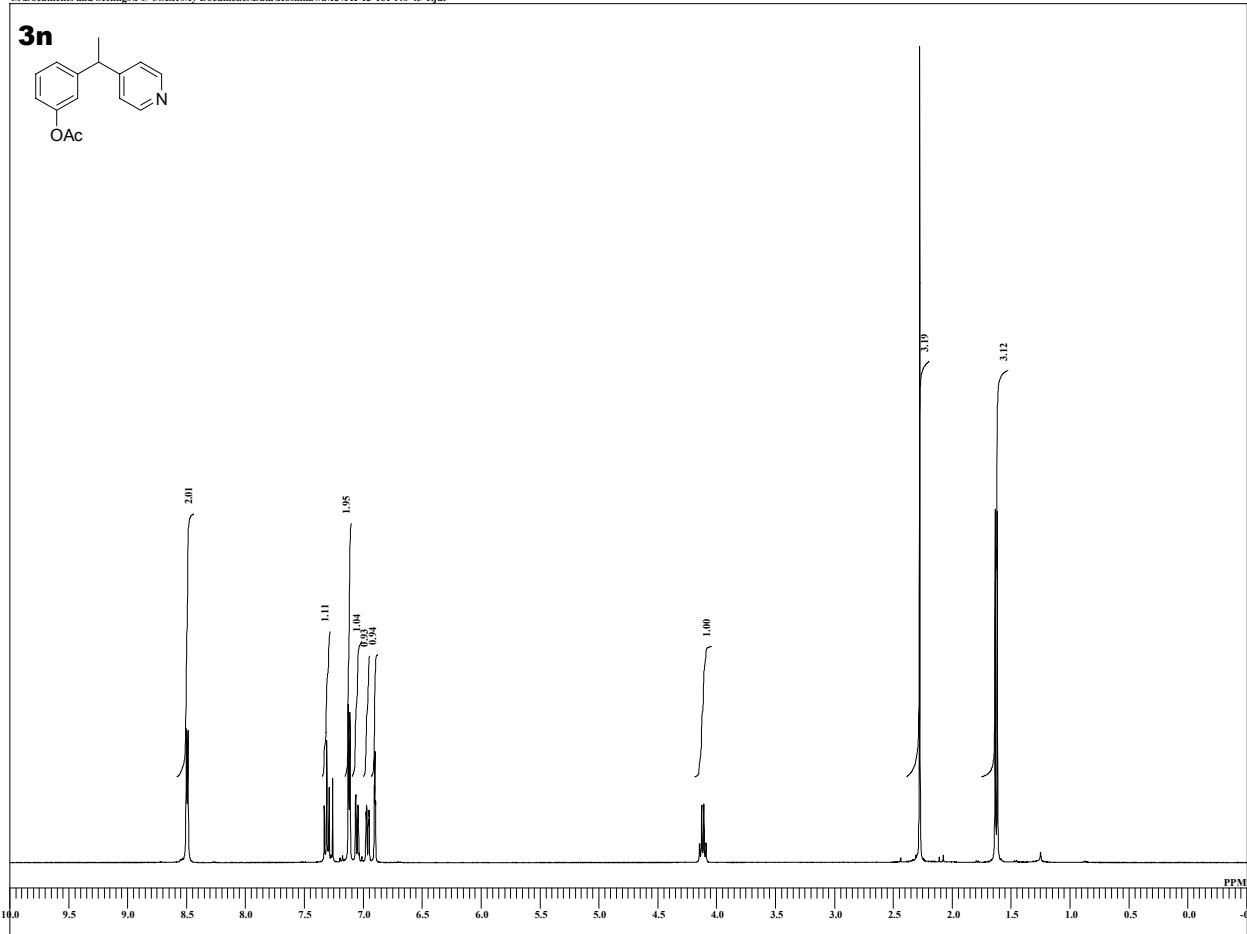
TH-13-17-f15-29

C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\13\TH-13-17-f15-29C-1.jdf



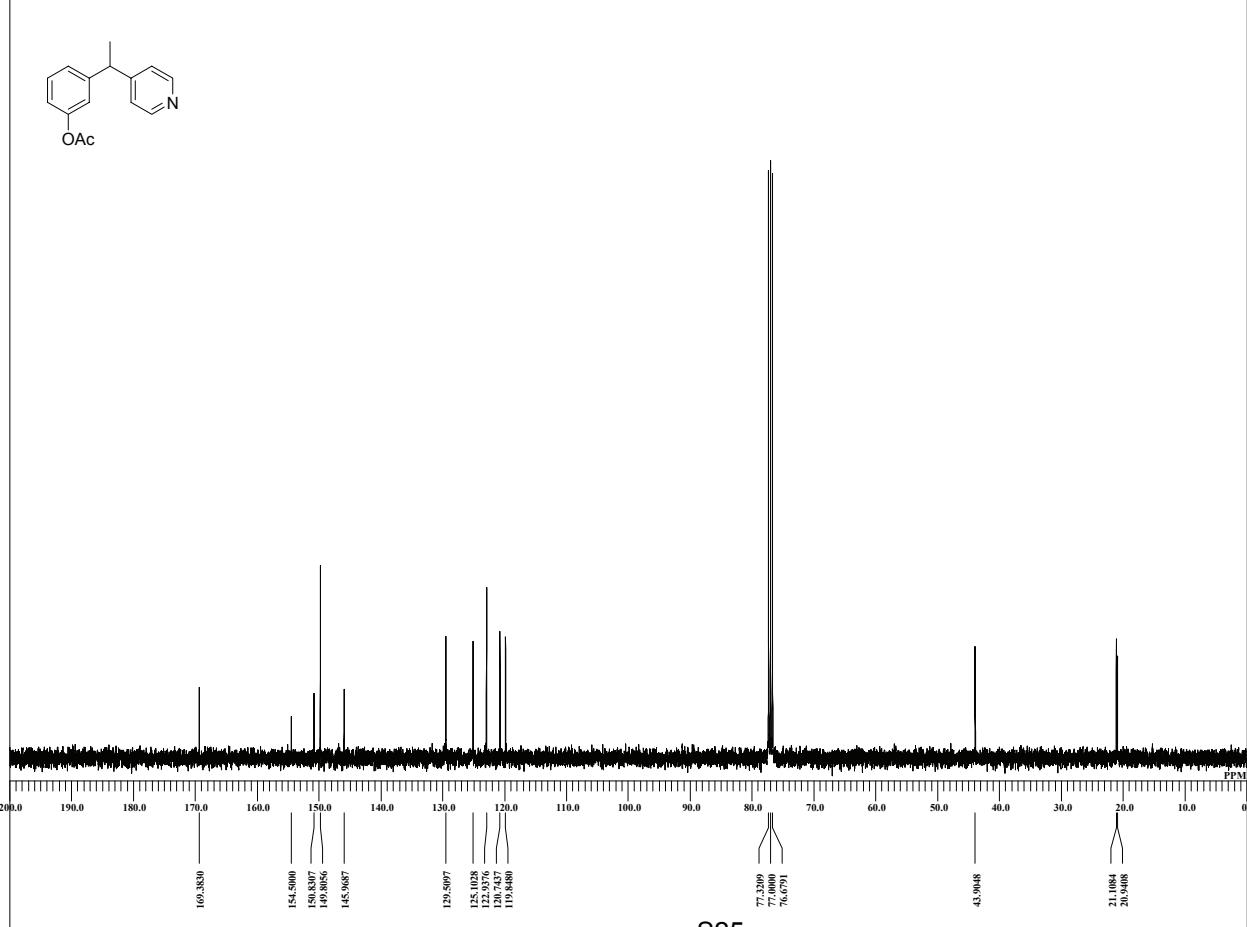
TH-13-161-f40-45

C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\13\TH-13-161-f40-45-1.jdf



TH-13-161-f40-45C

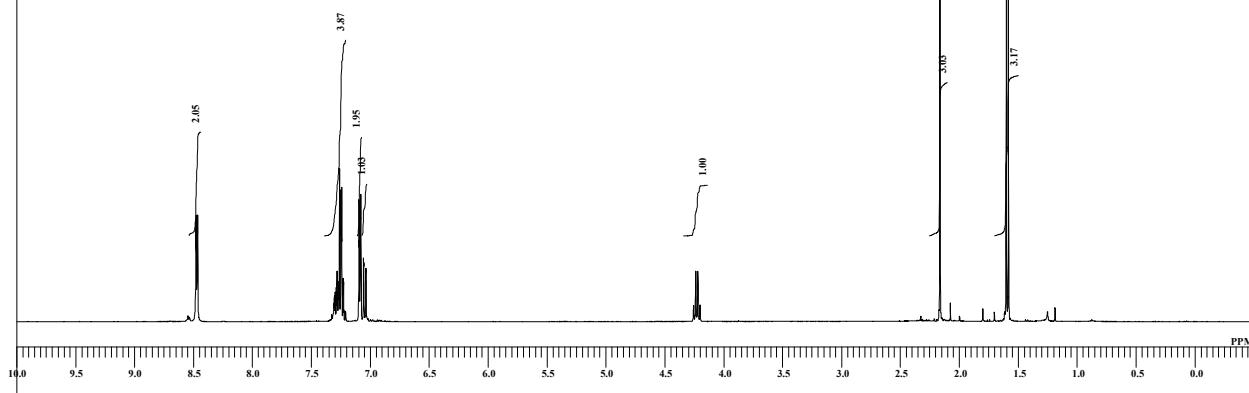
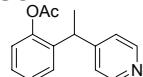
C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\13\TH-13-161-f40-45C-1.jdf



TH-13-159-f52-65

C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\13\TH-13-159-f52-65-1.jdf

**30**



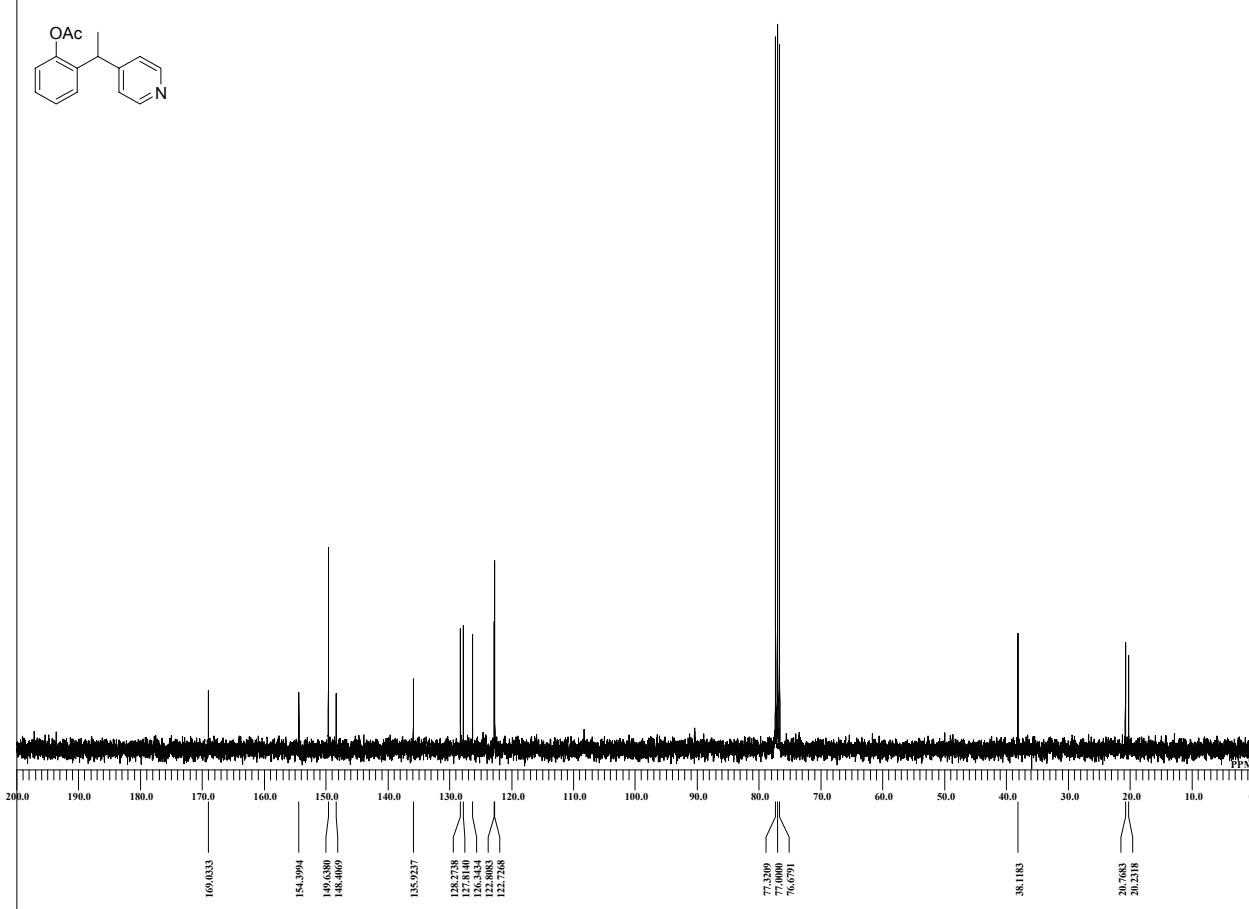
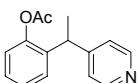
```

DFILE TH-13-159-f52-65-1.jdf
COMNT TH-13-159-f52-65
DATIM 13-11-2012 10:46:37
MENUF
OBNUC
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 kHz
OBFIN 0.87 Hz
PWI 6.38 sec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2072 sec
PD 5.0000 sec
SCANS 8
ADBIT 16
RGAIN 36
BF 0.01 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC IH
IRSET 395.88 MHz
IRFIN 6.28 kHz
IRPW 0.87 Hz
IRATN 79
DFILE TH-13-159-f52-65-1.jdf
LKSET 13.20 kHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPBS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 22.5 c
SLVNT CDCL3
EXREF 7.26 ppm

```

TH-13-159-f52-65C

C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\13\TH-13-159-f52-65C-1.jdf



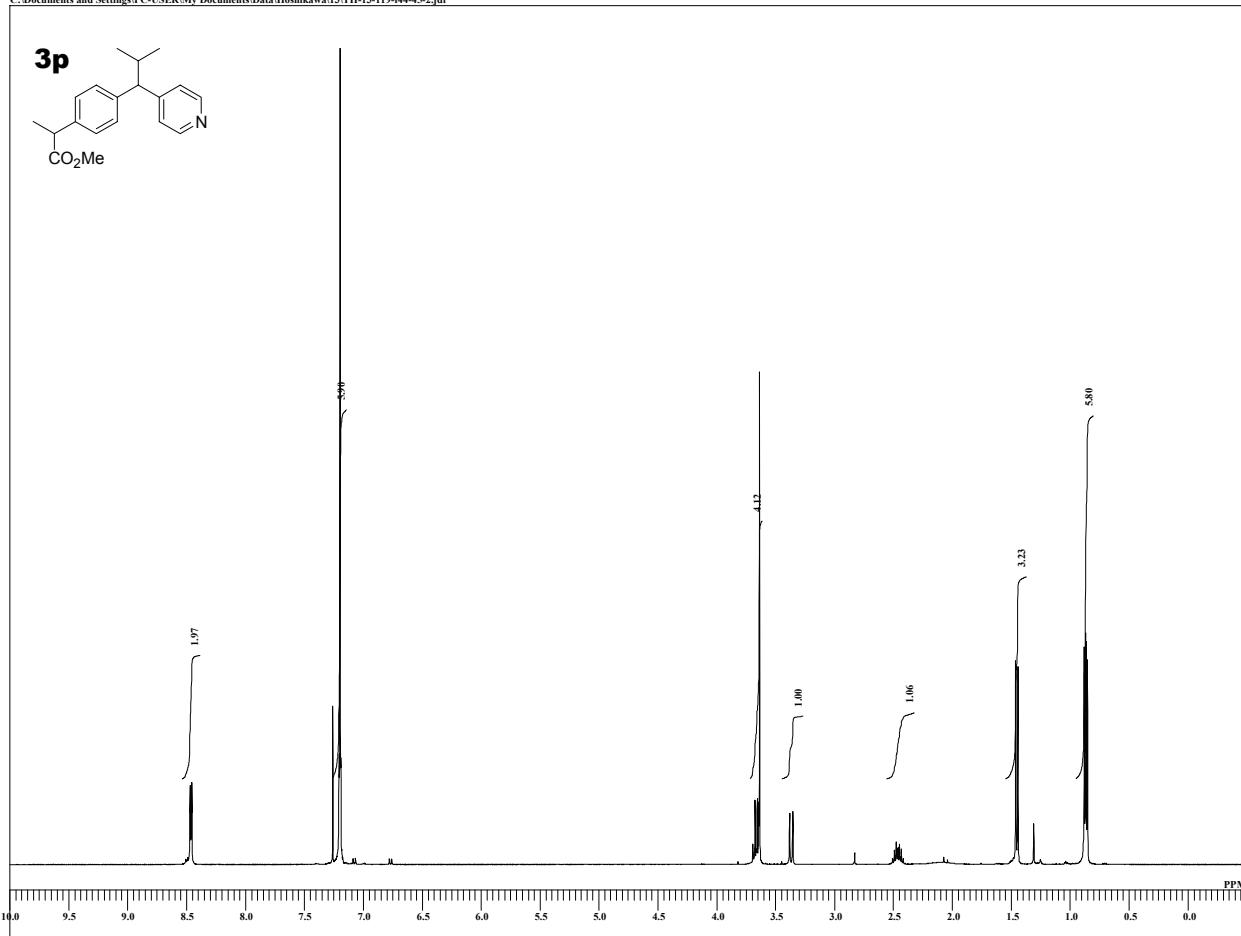
```

DFILE TH-13-159-f52-65C-1.jdf
COMNT TH-13-159-f52-65C
DATIM 13-11-2012 10:51:59
MENUF
OBNUC
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 kHz
OBFIN 0.98 Hz
PWI 3.24 sec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 65536
DO 65536
TIMES 26
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 8.0000 sec
SCANS 26
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC IH
IRSET 395.88 MHz
IRFIN 6.28 kHz
IRPW 0.87 Hz
IRATN 79
DFILE TH-13-159-f52-65C-1.jdf
LKSET 13.20 kHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPBS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 22.8 c
SLVNT CDCL3
EXREF 77.00 ppm

```

TH-13-119-f44-45

C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\13\TH-13-119-f44-45-2.jdf



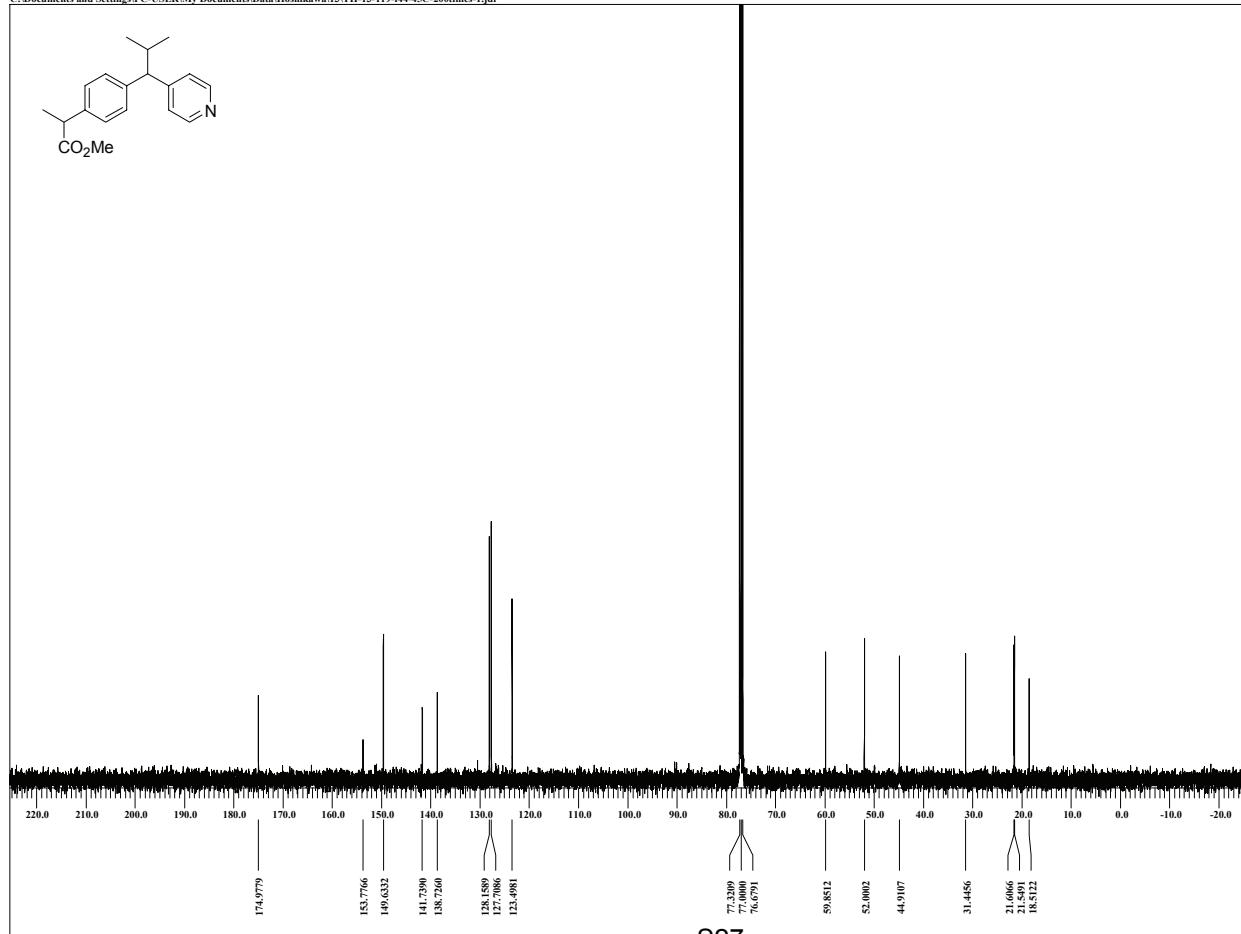
```

DFILE TH-13-119-f44-45-2.jdf
COMNT TH-13-119-f44-45
DATIM 20-10-2012 13:53:09
MENUF
OBNUC
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 kHz
OBFIN 0.87 Hz
PWI 6.38 sec
PWTADT 0.0000 sec
PREDL 0.0000 msec
IWT 1.0000 sec
POINT 20480
SPO 20480
TIMES 8
DUMMY 1
FREQU 9278.64 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2072 sec
PD 5.0000 sec
SCANS 8
ADBIT 16
RGAIN 40
BF 0.01 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC IH
IRSET 395.88 MHz
IRFIN 6.28 kHz
IRPW 0.87 Hz
IRATN 79
DFILE TH-13-119-f44-45-2.jdf
LKSET 13.20 kHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 23.4 c
SLVNT CDCL3
EXREF 7.26 ppm

```

TH-13-119-f44-45C-200times

C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\13\TH-13-119-f44-45C-200times-1.jdf



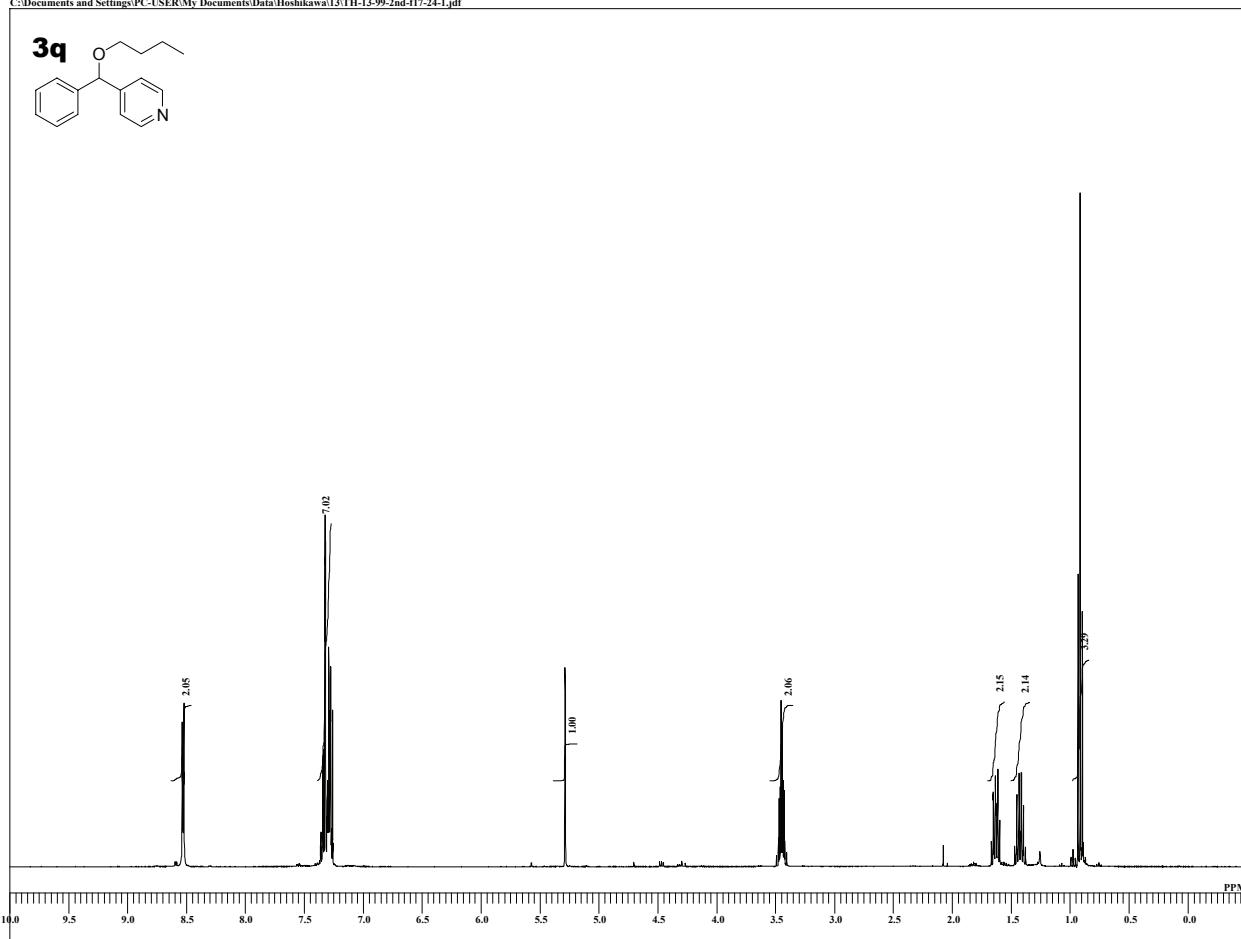
```

DFILE TH-13-119-f44-45C-200times-1.jdf
COMNT TH-13-119-f44-45C-200times
DATIM 22-10-2012 09:47:31
MENUF
OBNUC
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 kHz
OBFIN 0.98 Hz
PWI 3.14 sec
PWTADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 65536
T1 200
TIMES 200
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 0.0480 sec
ACQTM 8.0000 sec
PD 8.0000 sec
SCANS 200
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC IH
IRSET 395.88 MHz
IRFIN 6.28 kHz
IRPW 0.87 Hz
IRATN 79
DFILE TH-13-119-f44-45C-200times-1.jdf
LKSET 13.20 kHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 23.0 c
SLVNT CDCL3
EXREF 77.00 ppm

```

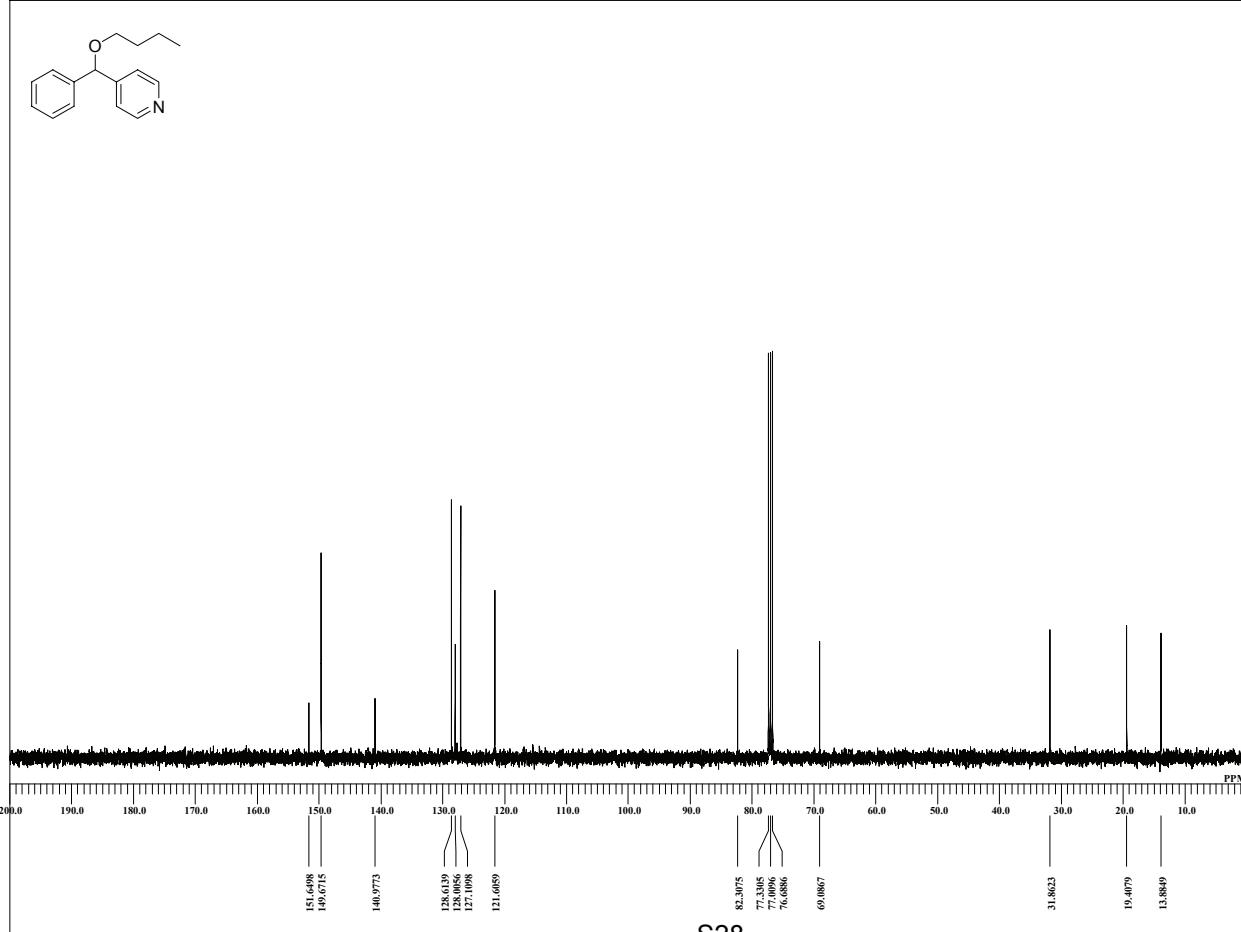
TH-13-99-2nd-f17-24

C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\13\TH-13-99-2nd-f17-24-1.jdf



TH-13-99-2nd-f17-24C

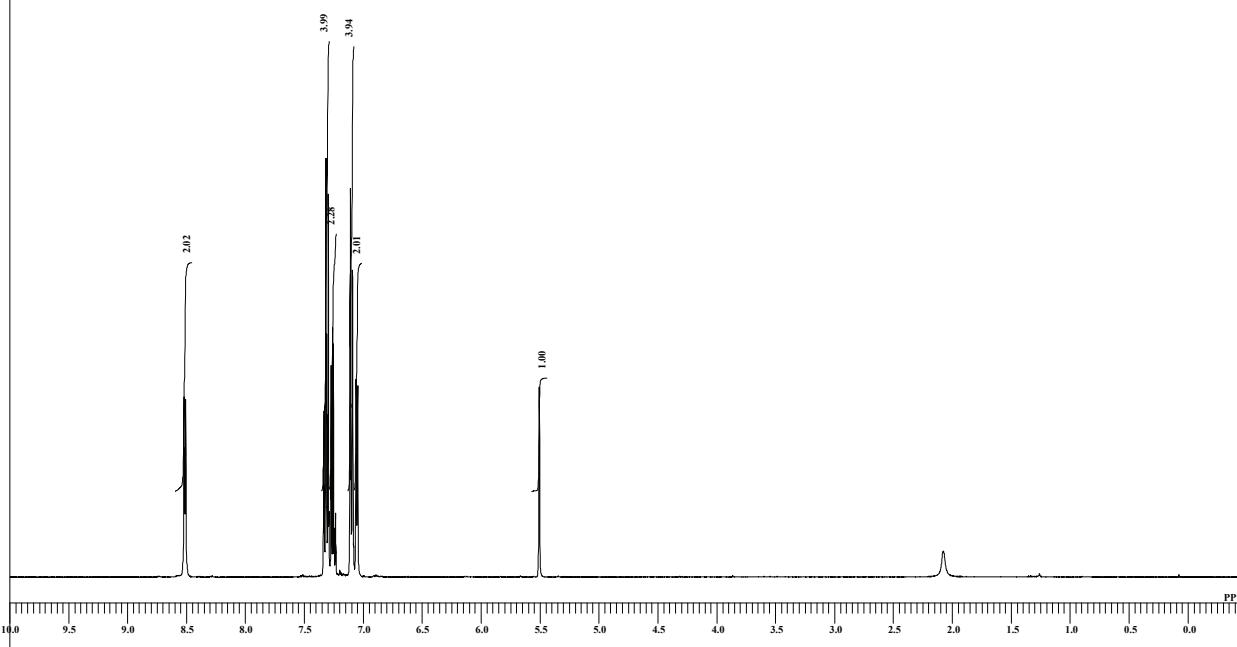
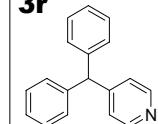
C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\13\TH-13-99-2nd-f17-24C-1.jdf



TH-13-57-f24-37-crystal

C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\13\TH-13-57-f24-37-crystal-H.jdf

**3r**



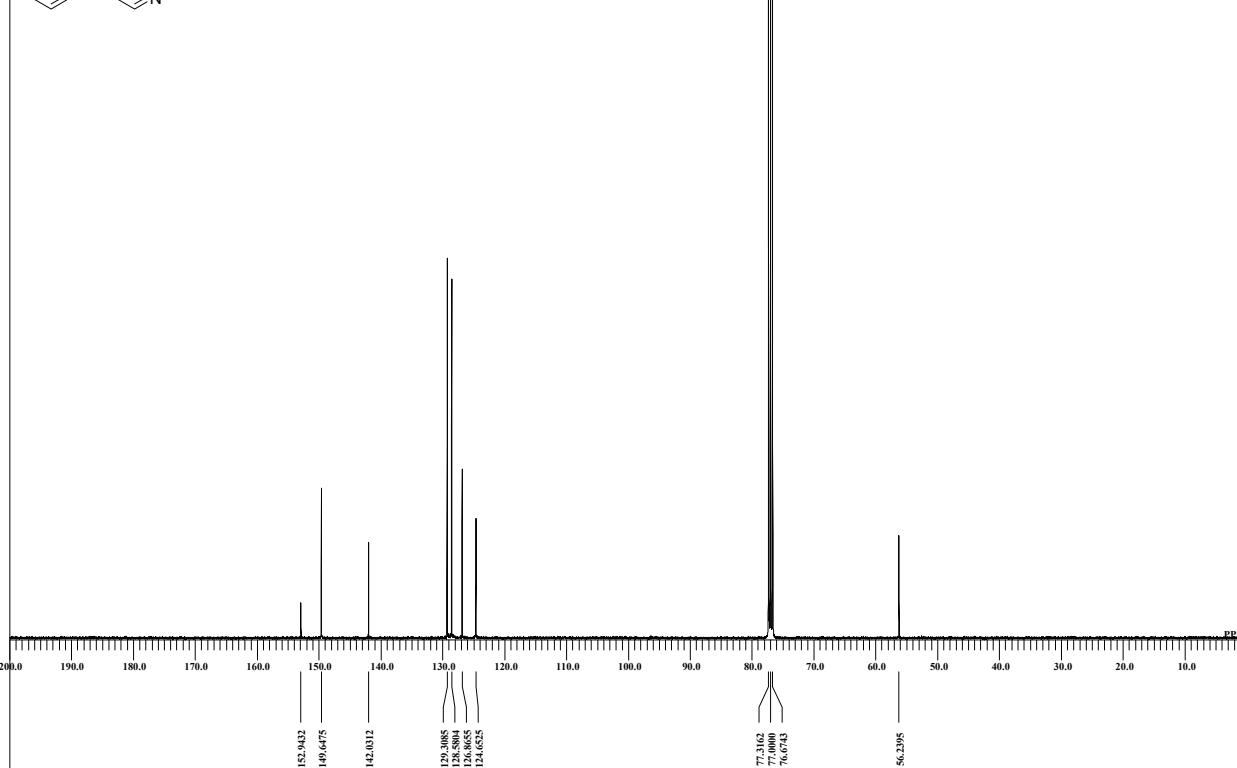
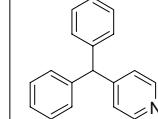
```

DFILE TH-13-57-f24-37-crystal-H.jdf
COMNT TH-13-57-f24-37-crystal
DATUM 21-09-2012 21:30:44
MENUF
OBNUC
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 kHz
OBFIN 0.87 Hz
PWI 6.38 sec
PREAD 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2072 sec
PD 5.0000 sec
SCANS 8
ADBIT 16
RGAIN 40
BF 0.01 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC IH
IRSET 395.88 MHz
IRFIN 6.28 kHz
IRRIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE TH-13-57-f24-37-crystal-H.jdf
LKSET 13.20 kHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPBS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDG
CTEMP 25.5 c
SLVNT CDCL3
EXREF 7.26 ppm

```

TH-13-57-f24-37-crystal

C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\13\TH-13-57-f24-37-crystal-C.jdf



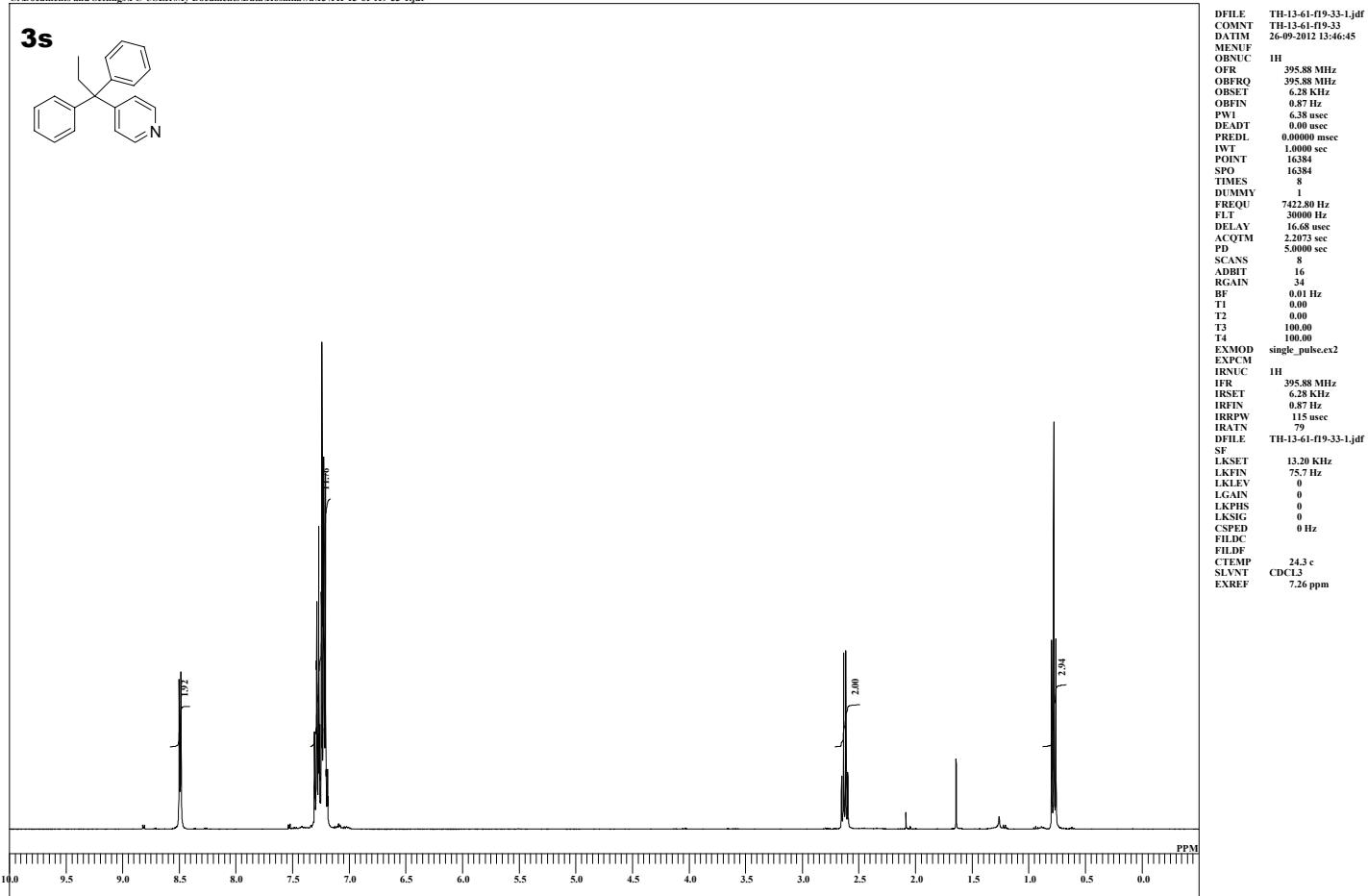
```

DFILE TH-13-57-f24-37-crystal-C.jdf
COMNT TH-13-57-f24-37-crystal
DATUM 22-09-2012 05:35:59
MENUF
OBNUC
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 kHz
OBFIN 0.98 Hz
PWI 3.14 sec
PREAD 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
T2 32768
TIMS 3200
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 8.0000 sec
SCANS 3200
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC IH
IFR 395.88 MHz
IRSET 6.28 kHz
IRFIN 0.87 Hz
IRRIN 115 usec
IRATN 79
DFILE TH-13-57-f24-37-crystal-C.jdf
LKSET 13.20 kHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPBS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDG
CTEMP 25.2 c
SLVNT CDCL3
EXREF 77.00 ppm

```

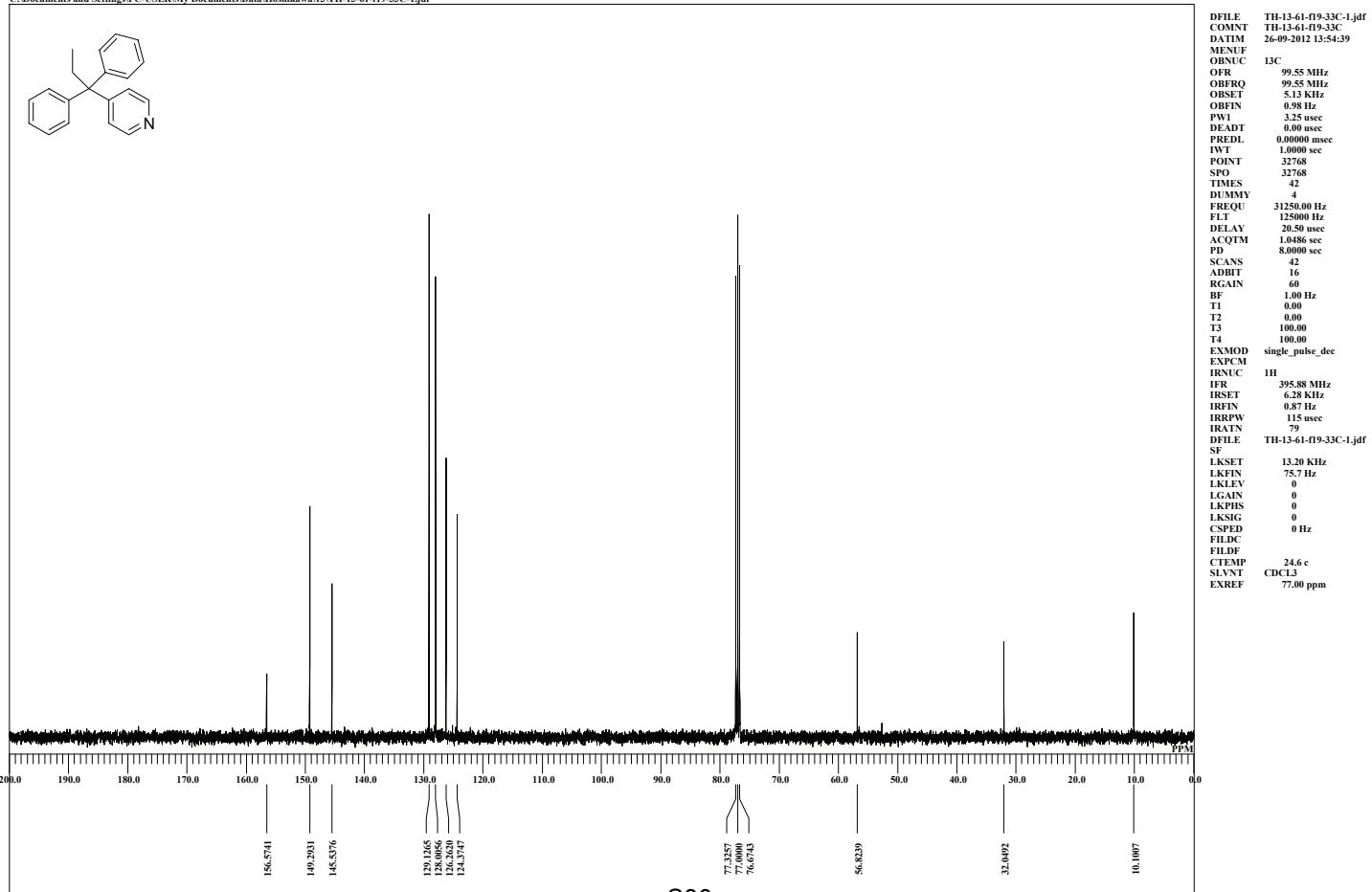
TH-13-61-f19-33

C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\13\TH-13-61-f19-33-1.jdf



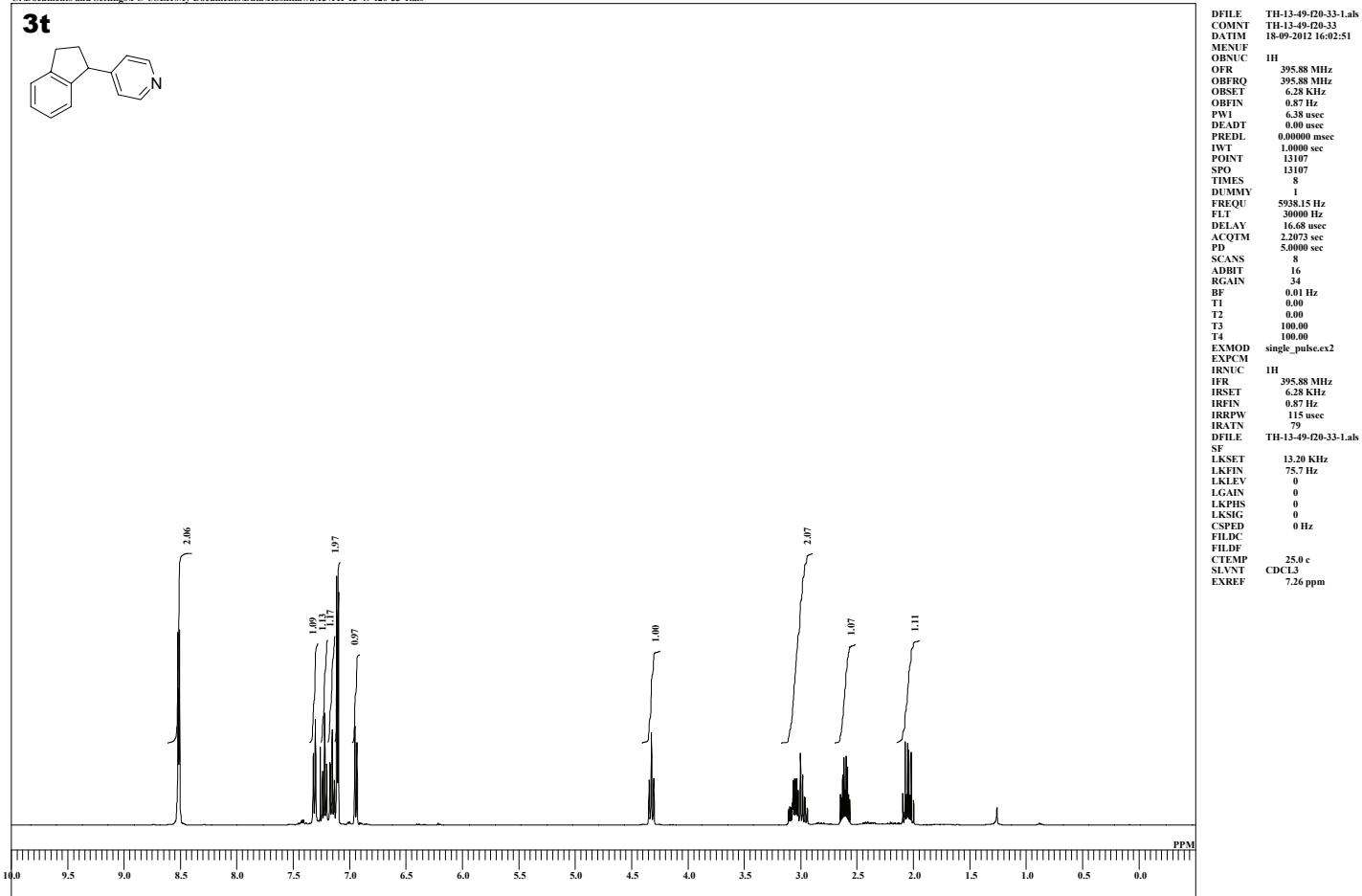
TH-13-61-f19-33C

C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\13\TH-13-61-f19-33C-1.jdf



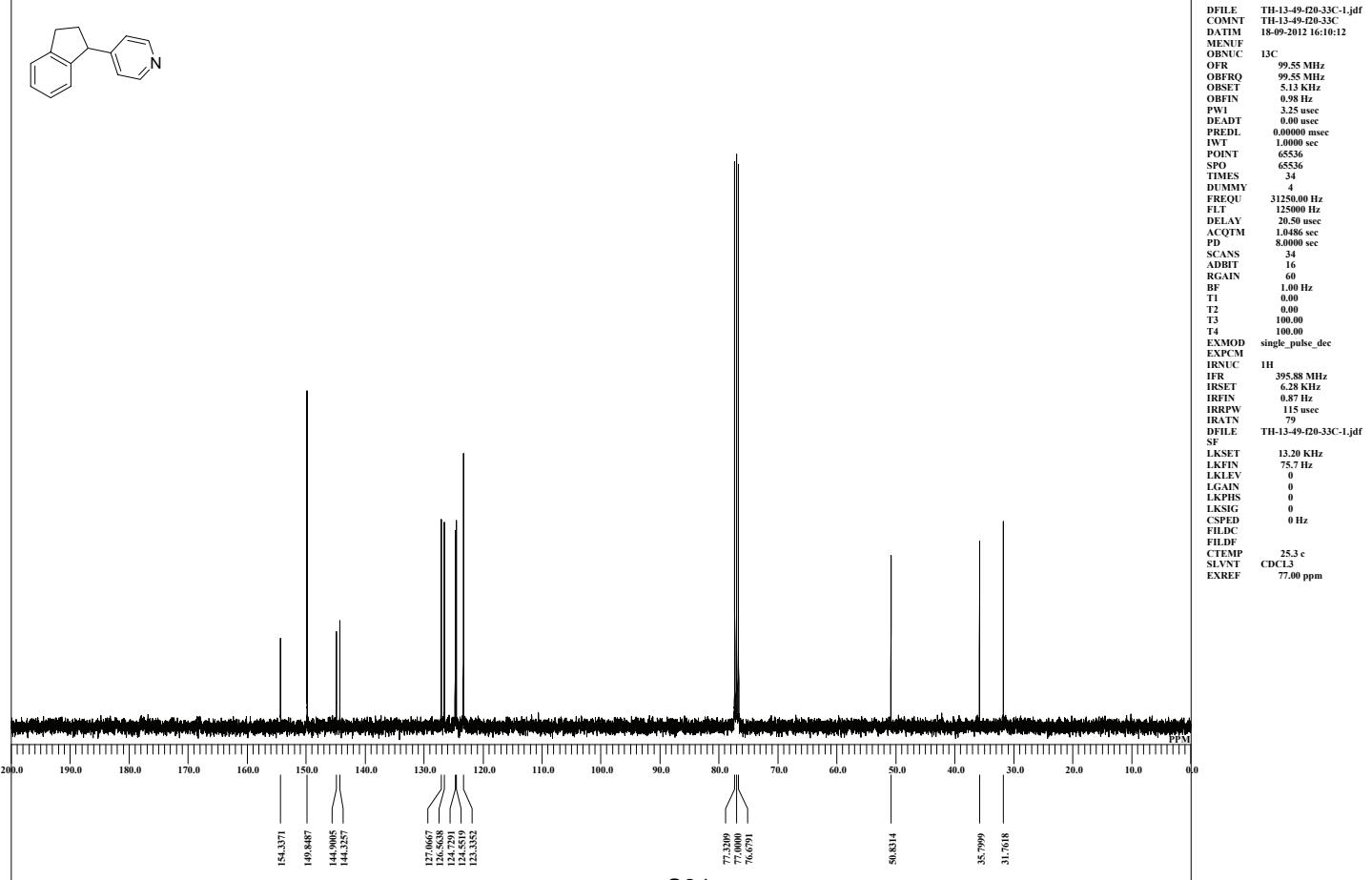
TH-13-49-f20-33

C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\13\TH-13-49-f20-33-1.xls



TH-13-49-f20-33C

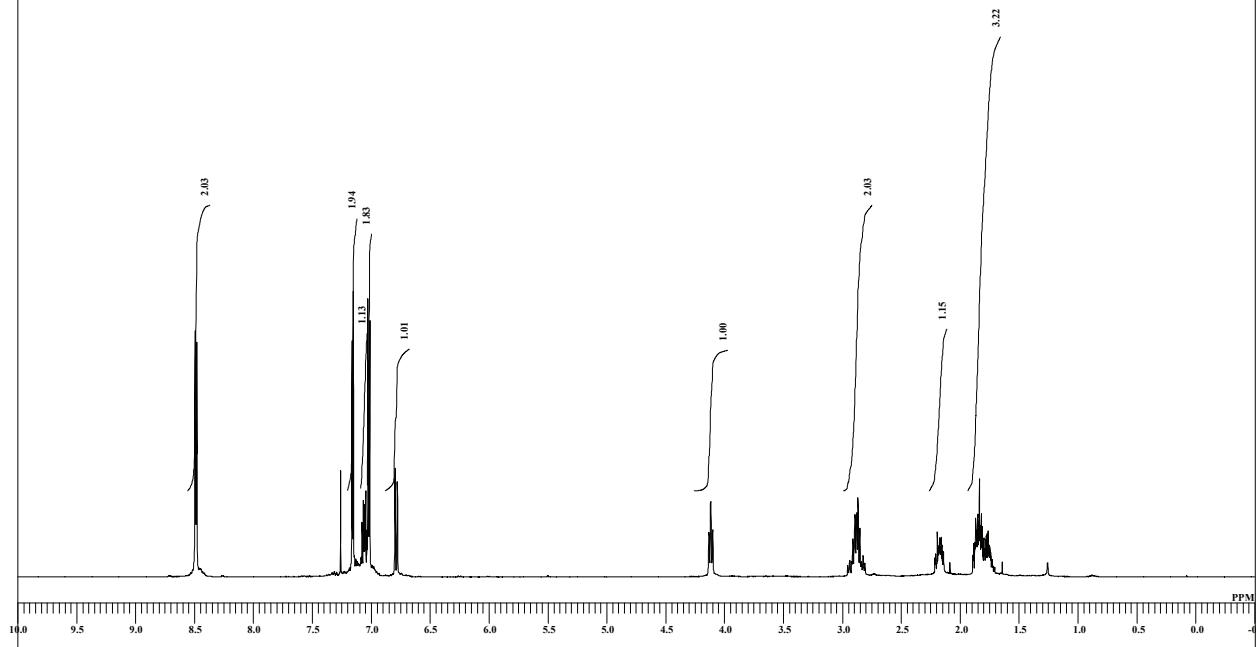
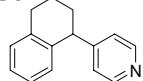
C:\Documents and Settings\PC-USER\My Documents\Data\Hoshikawa\13\TH-13-49-f20-33C-1.jdf



TH-13-45-f20-33

C:\Documents and Settings\PC-USER\My Documents\DATA\Hoshikawa\13\TH-13-45-f20-33-1.jdf

**3u**



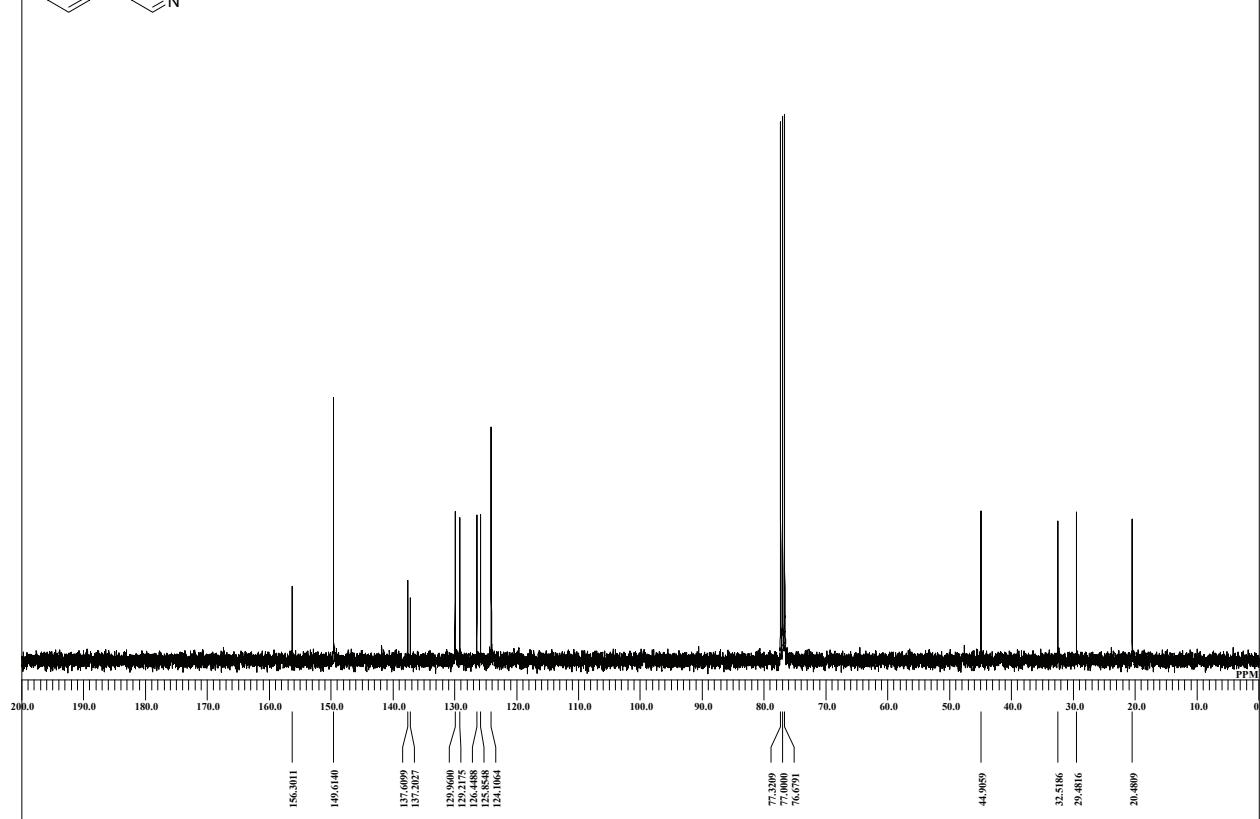
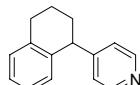
```

DFILE TH-13-45-f20-33-1.jdf
COMNT TH-13-45-f20-33
DATUM 07-09-2012 13:07:32
MENUF
MENUC
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 kHz
OBFIN 0.87 Hz
PWI 6.38 sec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2072 sec
PD 5.0000 sec
SCANS 8
ADBIT 16
RGAIN 34
BF 0.01 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC IH
IRSET 395.88 MHz
IRFIN 6.28 kHz
IRPW 0.87 Hz
IRATN 79
DFILE TH-13-45-f20-33-1.jdf
LKSET 13.20 kHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 24.4 c
SLVNT CDCL3
EXREF 7.26 ppm

```

TH-13-45-f20-33C

C:\Documents and Settings\PC-USER\My Documents\DATA\Hoshikawa\13\TH-13-45-f20-33C-1.jdf



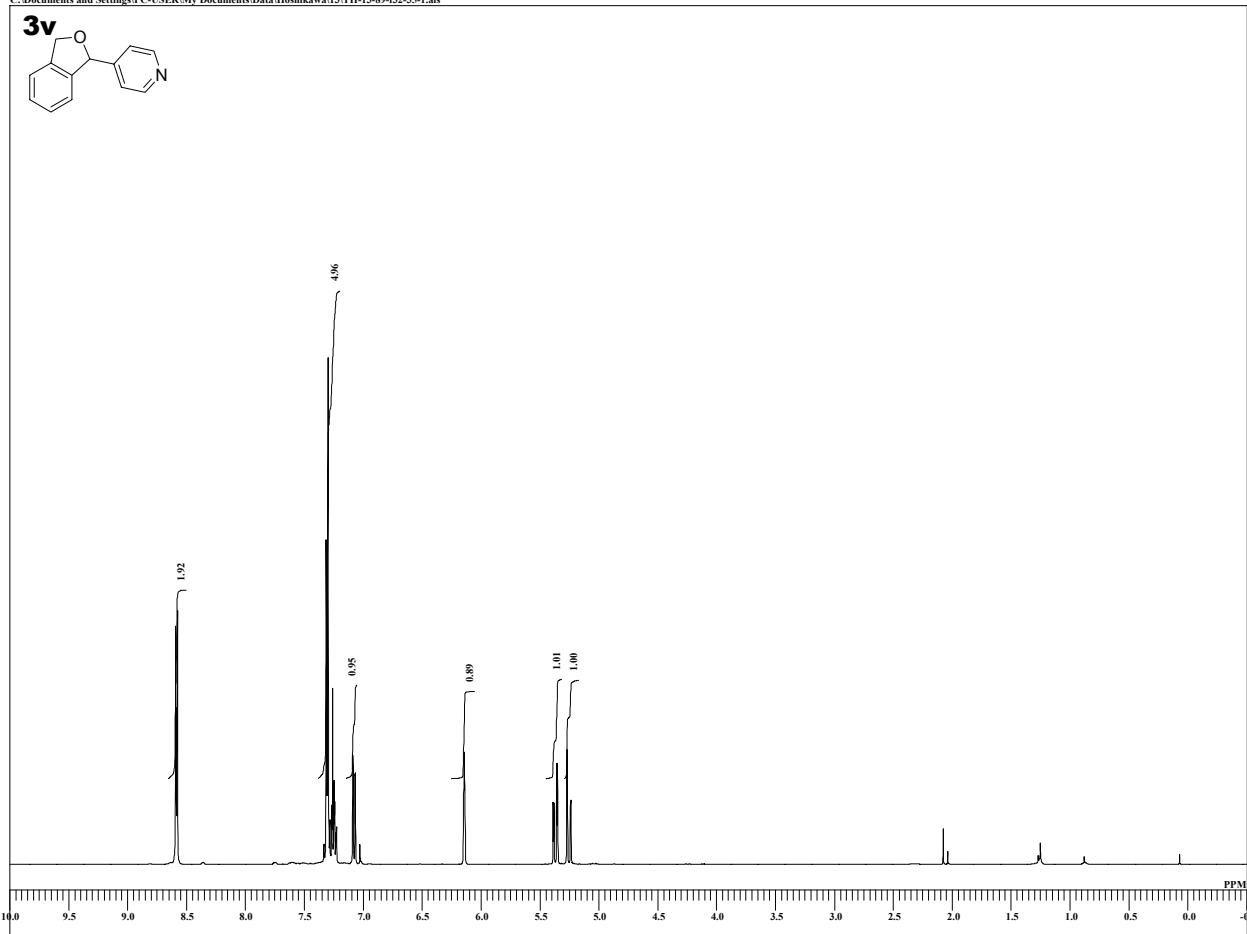
```

DFILE TH-13-45-f20-33C-1.jdf
COMNT TH-13-45-f20-33C
DATUM 07-09-2012 13:14:25
MENUF
MENUC
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 kHz
OBFIN 0.98 Hz
PWI 3.24 sec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 65536
GSO36
TIMES 34
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0480 sec
PD 8.0000 sec
SCANS 34
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC IH
IRSET 395.88 MHz
IRFIN 6.28 kHz
IRPW 0.87 Hz
IRATN 79
DFILE TH-13-45-f20-33C-1.jdf
SF 13.20 kHz
LKSET 75.7 Hz
LKFIN 0
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 24.6 c
SLVNT CDCL3
EXREF 77.00 ppm

```

TH-13-89-f32-35

C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\13\TH-13-89-f32-35-1.als



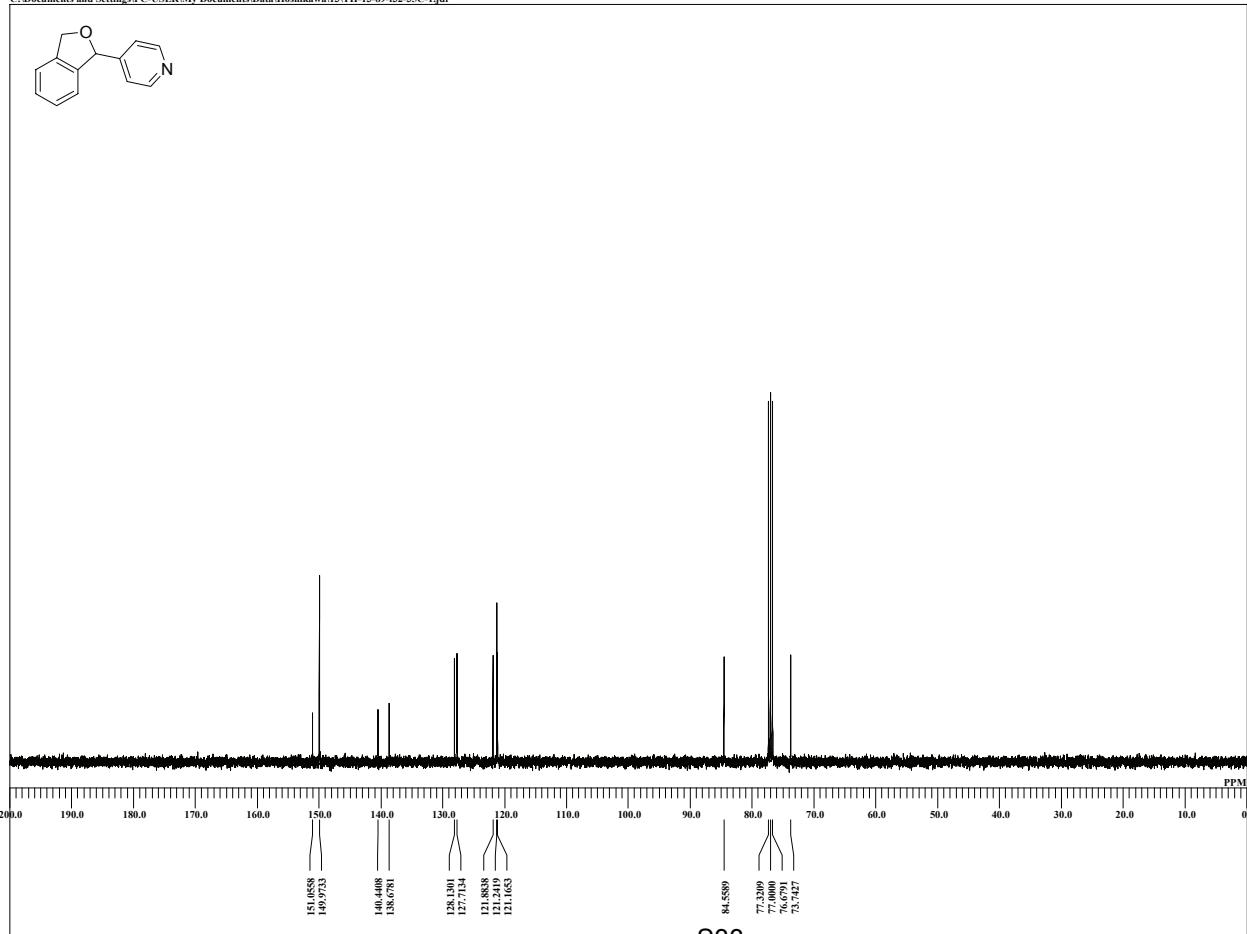
```

DFILE TH-13-89-f32-35-1.als
COMNT TH-13-89-f32-35
DATIM 05-10-2012 16:25:28
MENUF
MENUC
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 kHz
OBFIN 0.87 Hz
PWI 6.38 sec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2072 sec
PD 5.0000 sec
SCANS 8
ADBIT 16
RGAIN 38
BF 0.01 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC IH
IRSET 395.88 MHz
IRFIN 6.28 kHz
IRFIN 0.87 Hz
IRRPW 147 usec
IRATN 79
DFILE TH-13-89-f32-35-1.als
LKSET 13.20 kHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FLDC
FILDF
CTEMP 24.3 c
SLVNT CDCL3
EXREF 7.26 ppm

```

TH-13-89-f32-35C

C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\13\TH-13-89-f32-35C-1.jdf



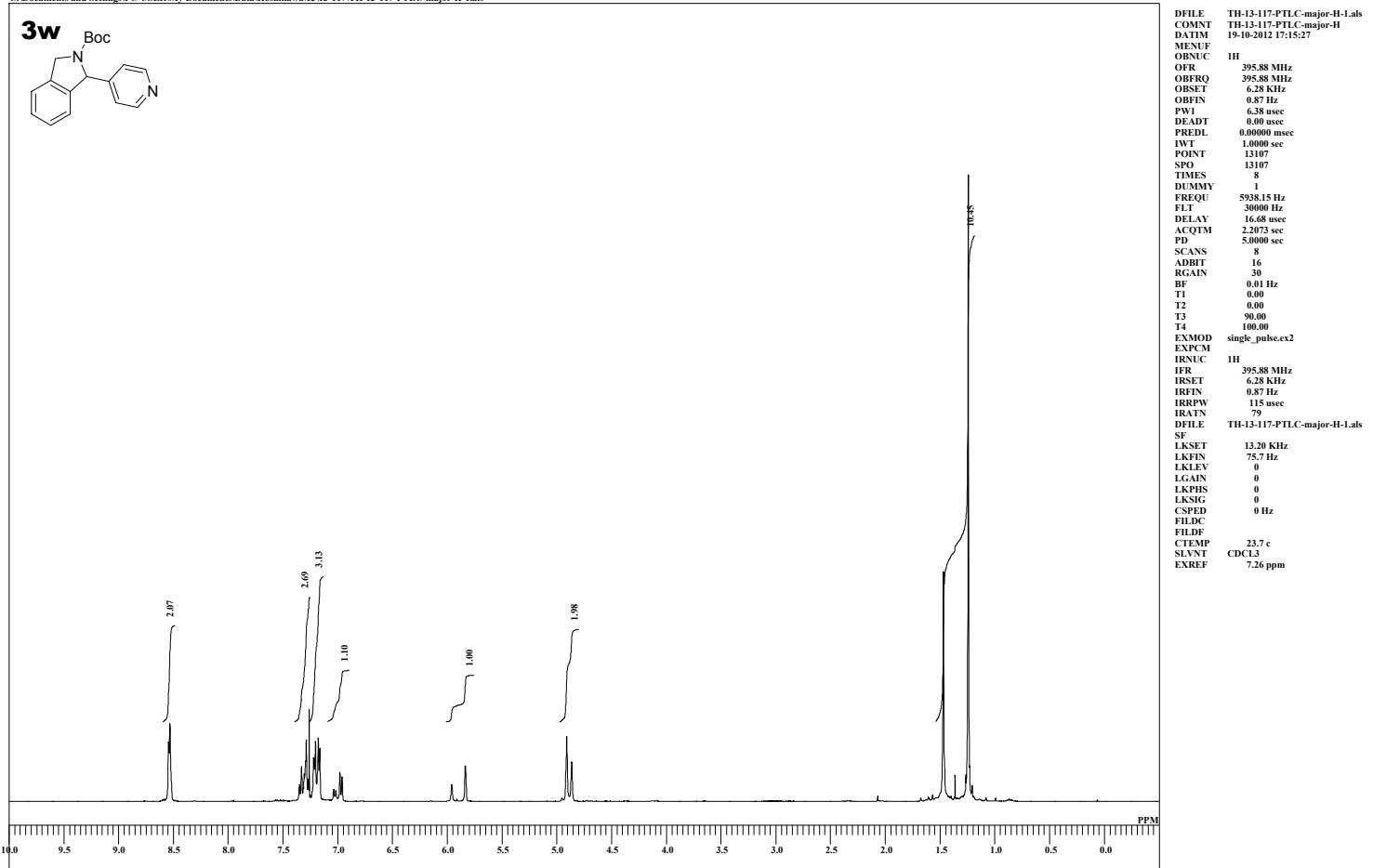
```

DFILE TH-13-89-f32-35C-1.jdf
COMNT TH-13-89-f32-35C
DATIM 05-10-2012 16:32:11
MENUF
MENUC
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 kHz
OBFIN 0.98 Hz
PWI 3.24 sec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 65536
GSO36
TIMES 34
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 8.0000 sec
SCANS 34
ADBIT 16
RGAIN 60
BF 1.016 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC IH
IRSET 395.88 MHz
IRFIN 6.28 kHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE TH-13-89-f32-35C-1.jdf
SF
LKSET 13.20 kHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FLDC
FILDF
CTEMP 24.5 c
SLVNT CDCL3
EXREF 77.00 ppm

```

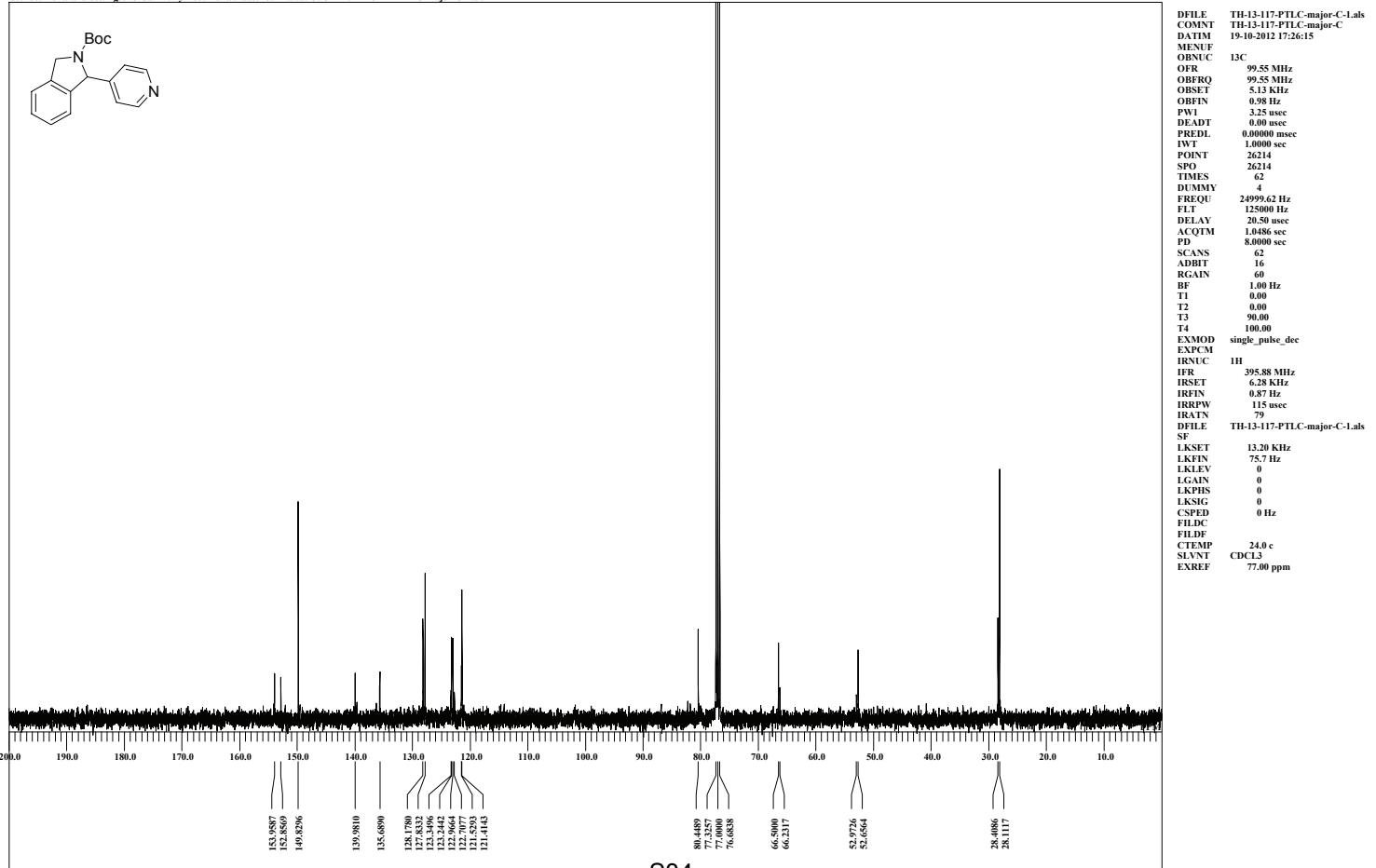
TH-13-117-PTLC-major-H

C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\13\13-117\TH-13-117-PTLC-major-H.lals



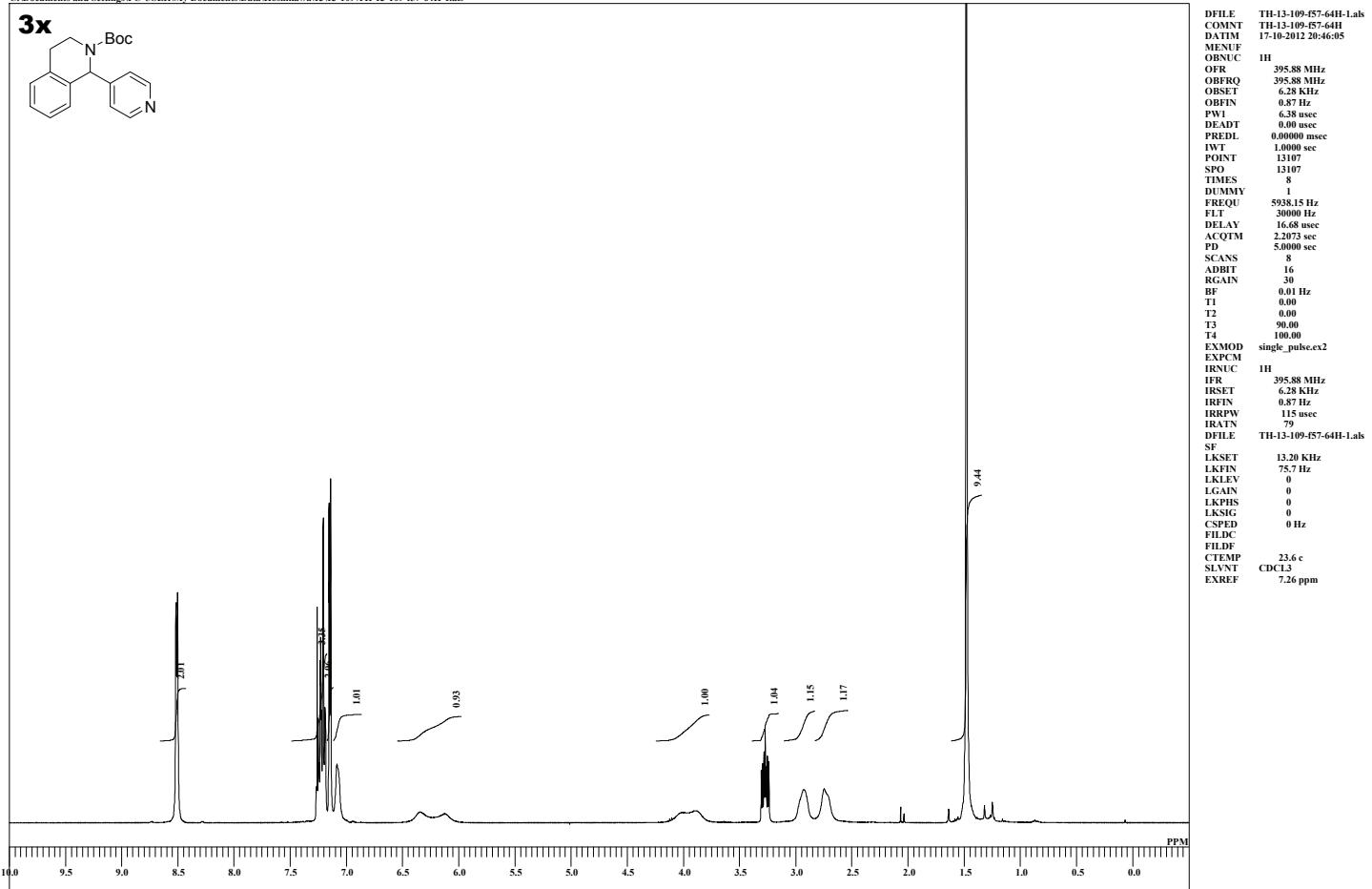
TH-13-117-PTLC-major-C

C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\13\13-117\TH-13-117-PTLC-major-C.lals



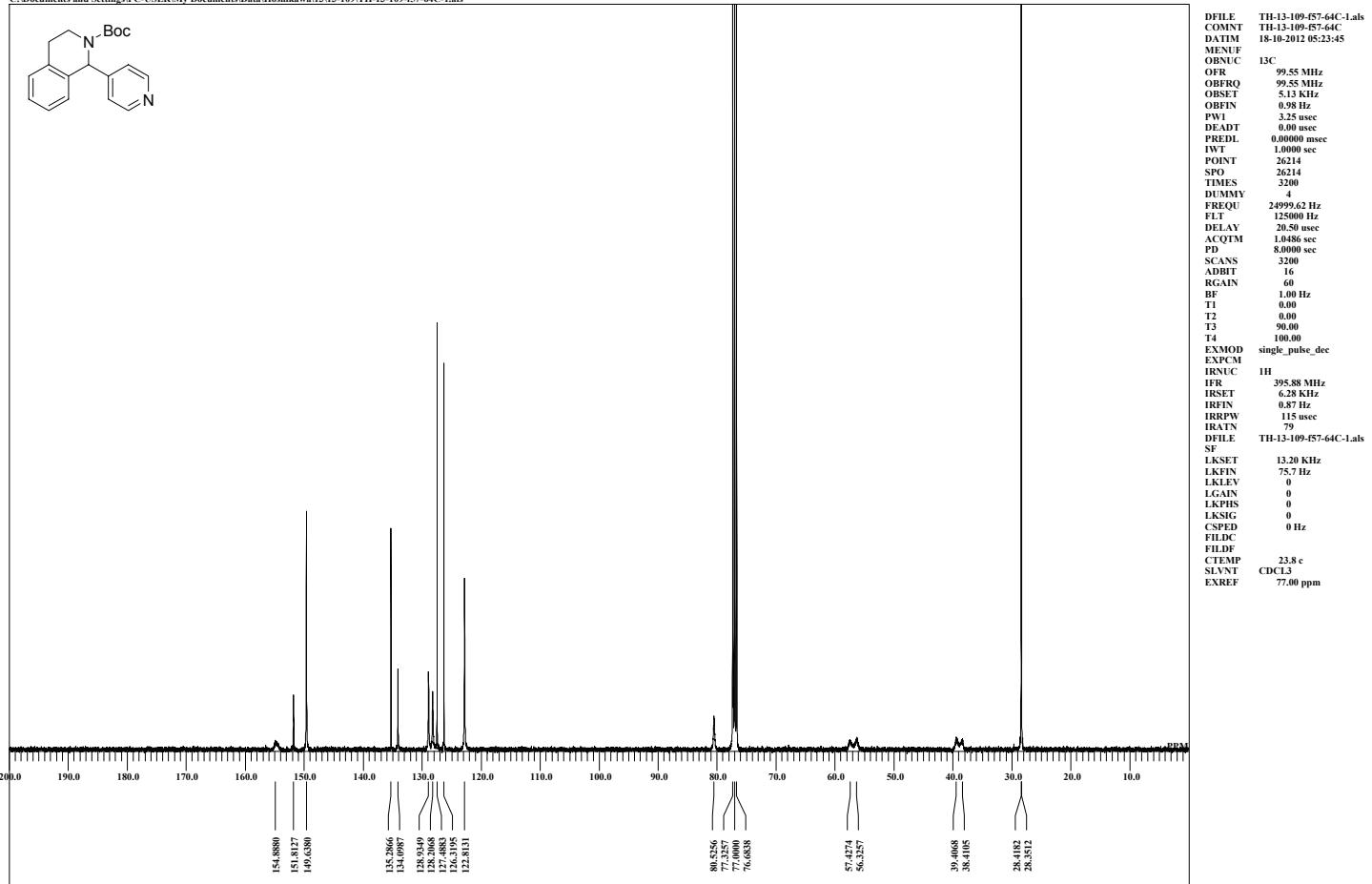
TH-13-109-f57-64H

C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\13\13-109\TH-13-109-f57-64H-labs



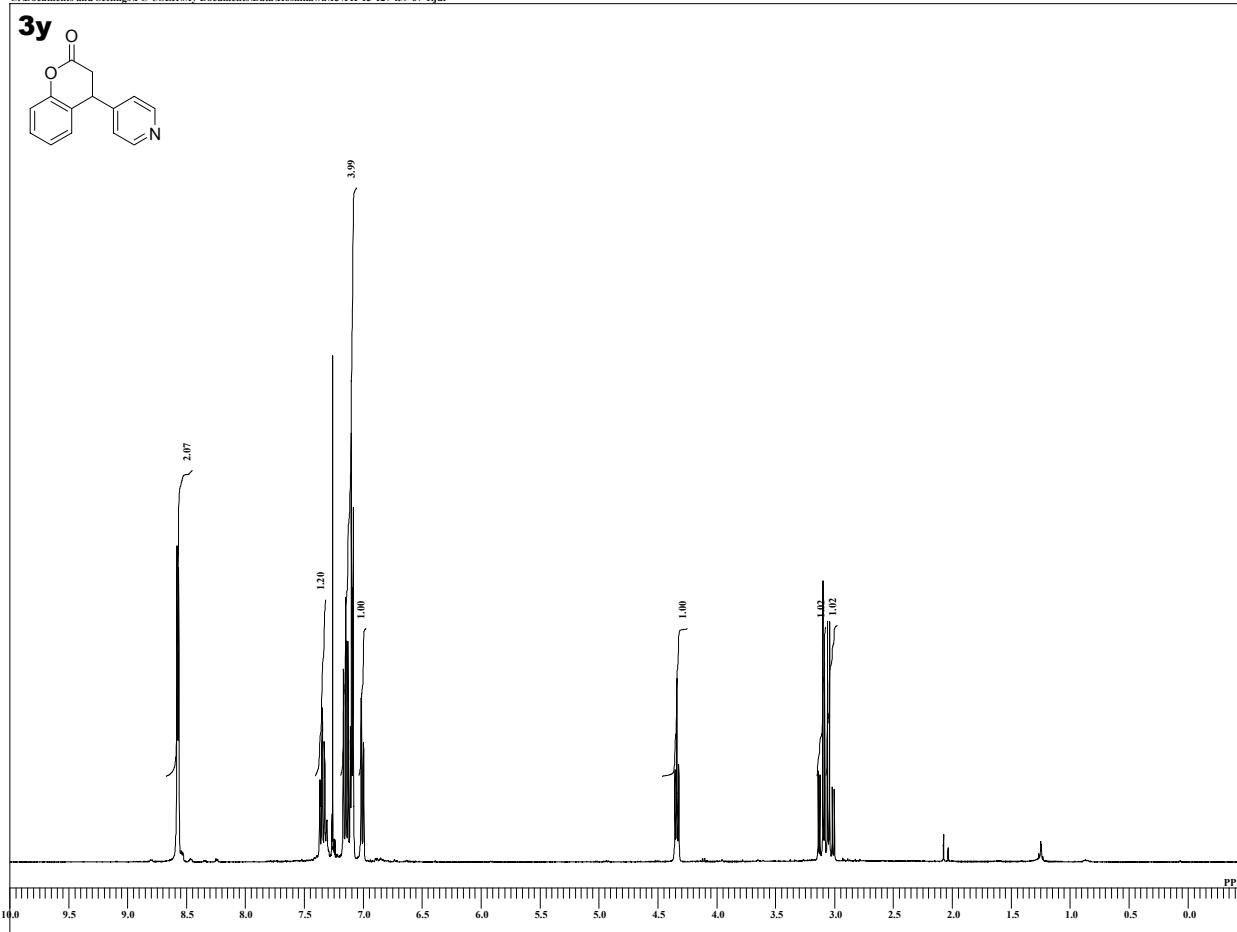
TH-13-109-f57-64C

C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\13\13-109\TH-13-109-f57-64C-labs



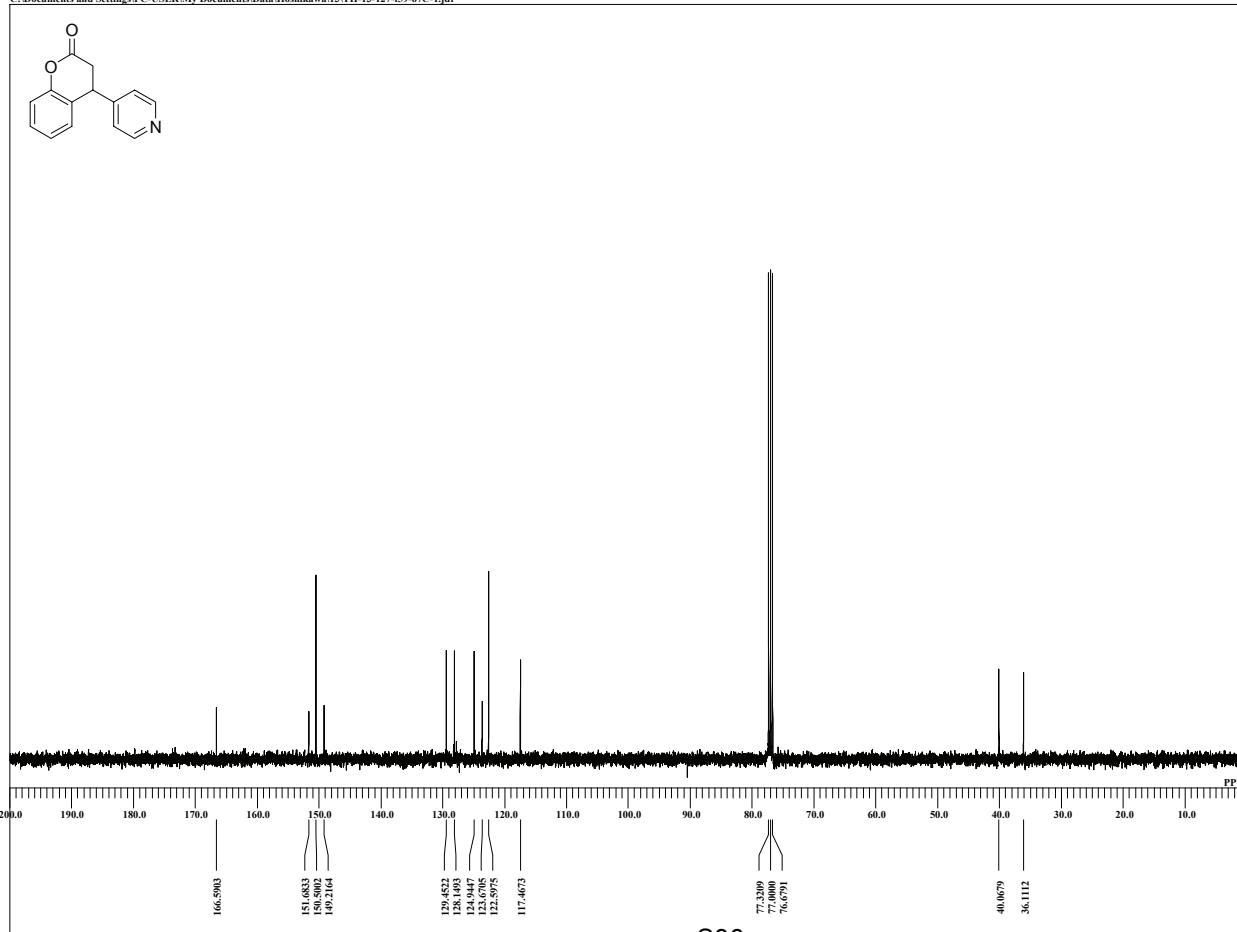
TH-13-127-f59-67

C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\13\TH-13-127-f59-67-1.jdf



TH-13-127-f59-67C

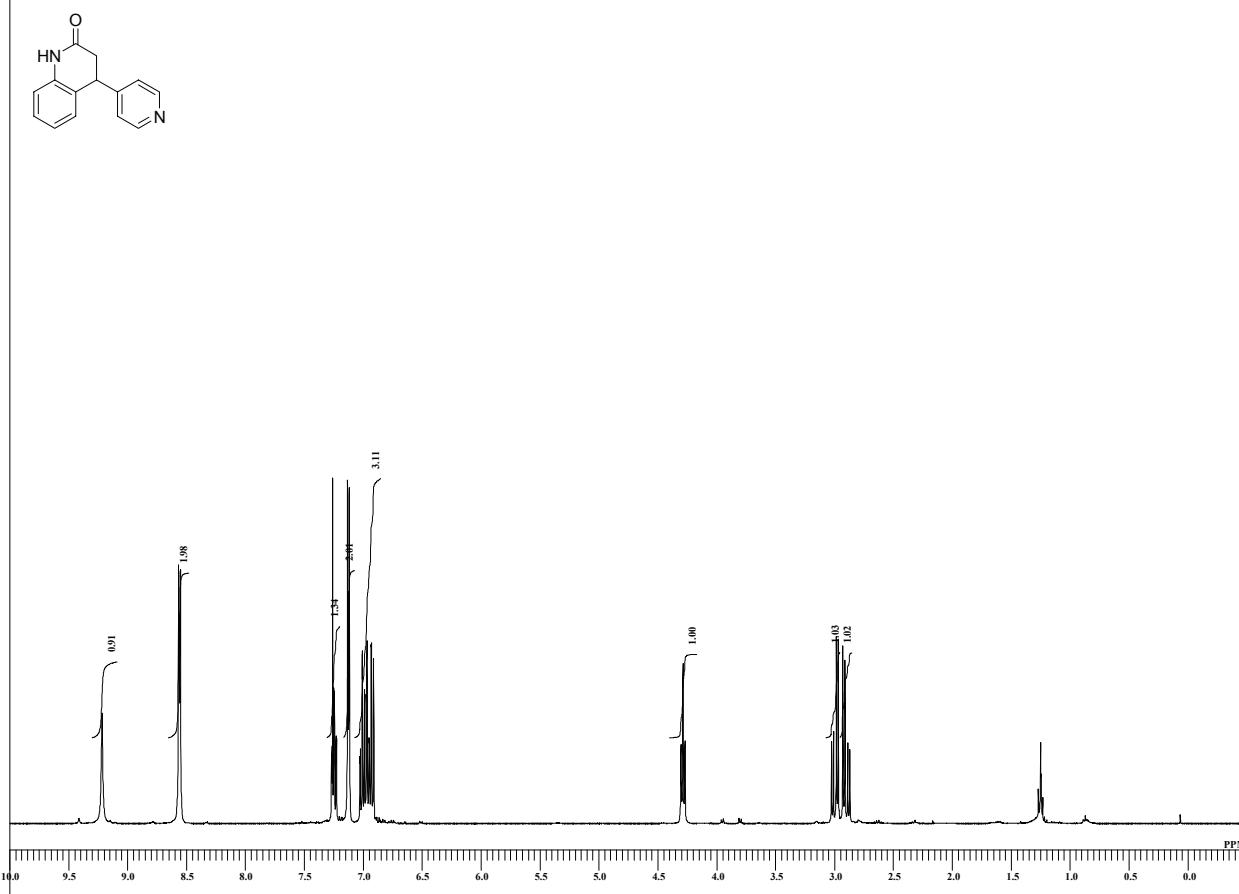
C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\13\TH-13-127-f59-67C-1.jdf



TH-13-167-f44-54-2nd

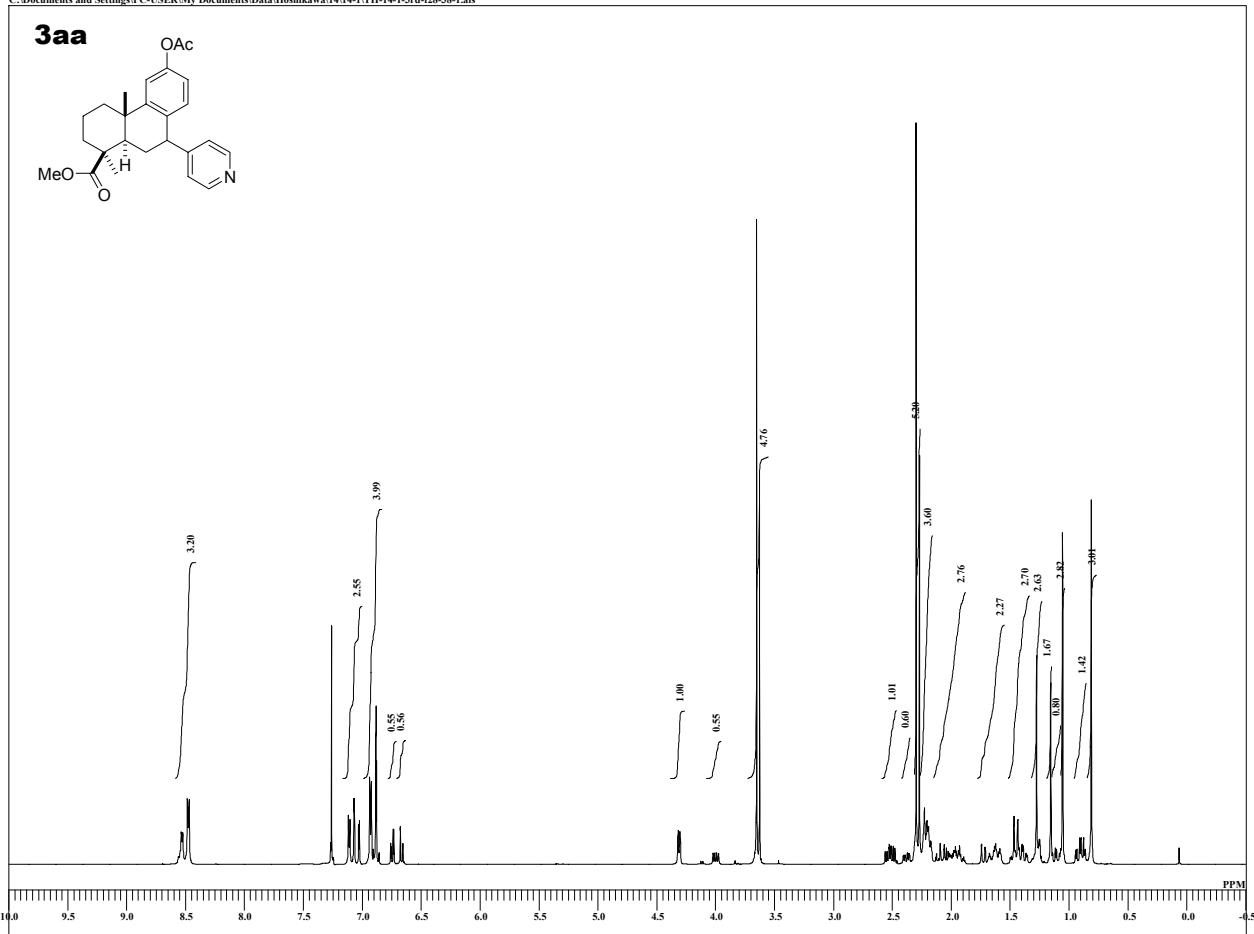
C:\Documents and Settings\PC-USER\My Documents\DATA\Hoshikawa\13\13-167\TH-13-167-f44-54-2nd-lals

**3z**



TH-14-1-3rd-f28-38

C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\14\14-1\TH-14-1-3rd-f28-38-1.als



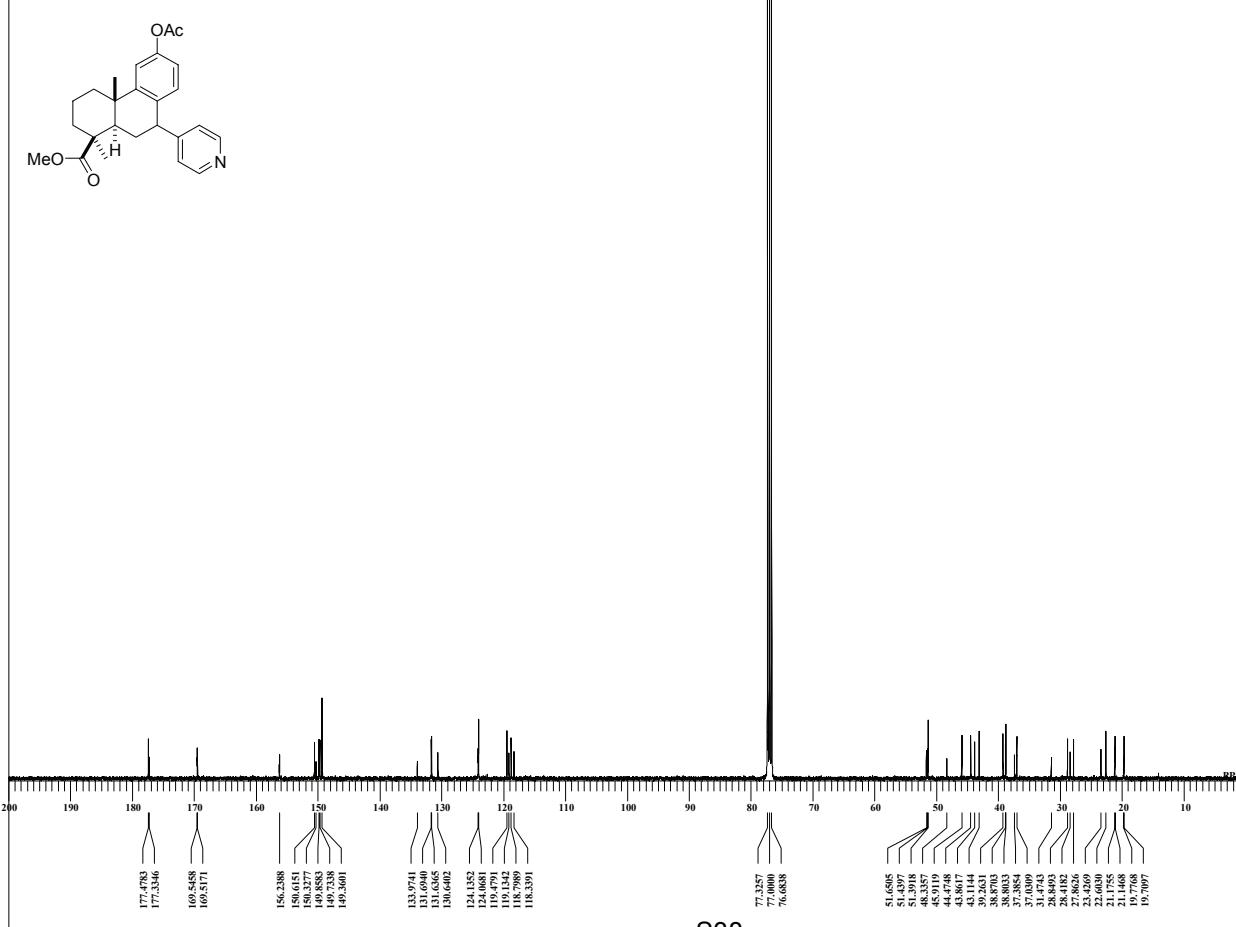
```

DFILE TH-14-1-3rd-f28-38-1.als
COMNT TH-14-1-3rd-f28-38
DATIM 06-03-2013 21:21:37
MENUF
OBNUC
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 kHz
OBFIN 0.87 Hz
PWI 6.38 sec
PREADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 13107
SPO 13107
TIMES 8
FREQU 5938.15 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2072 sec
PD 5.0000 sec
SCANS 8
ADBIT 16
RGAIN 38
BF 0.01 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC IH
IRSET 395.88 MHz
IRFIN 6.28 kHz
IRFW 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE TH-14-1-3rd-f28-38-1.als
LKSET 13.20 kHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 20.4 c
SLVNT CDCL3
EXREF 7.26 ppm

```

TH-41-1-3rd-f28-38C

C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\14\14-1\TH-41-1-3rd-f28-38C-1.als



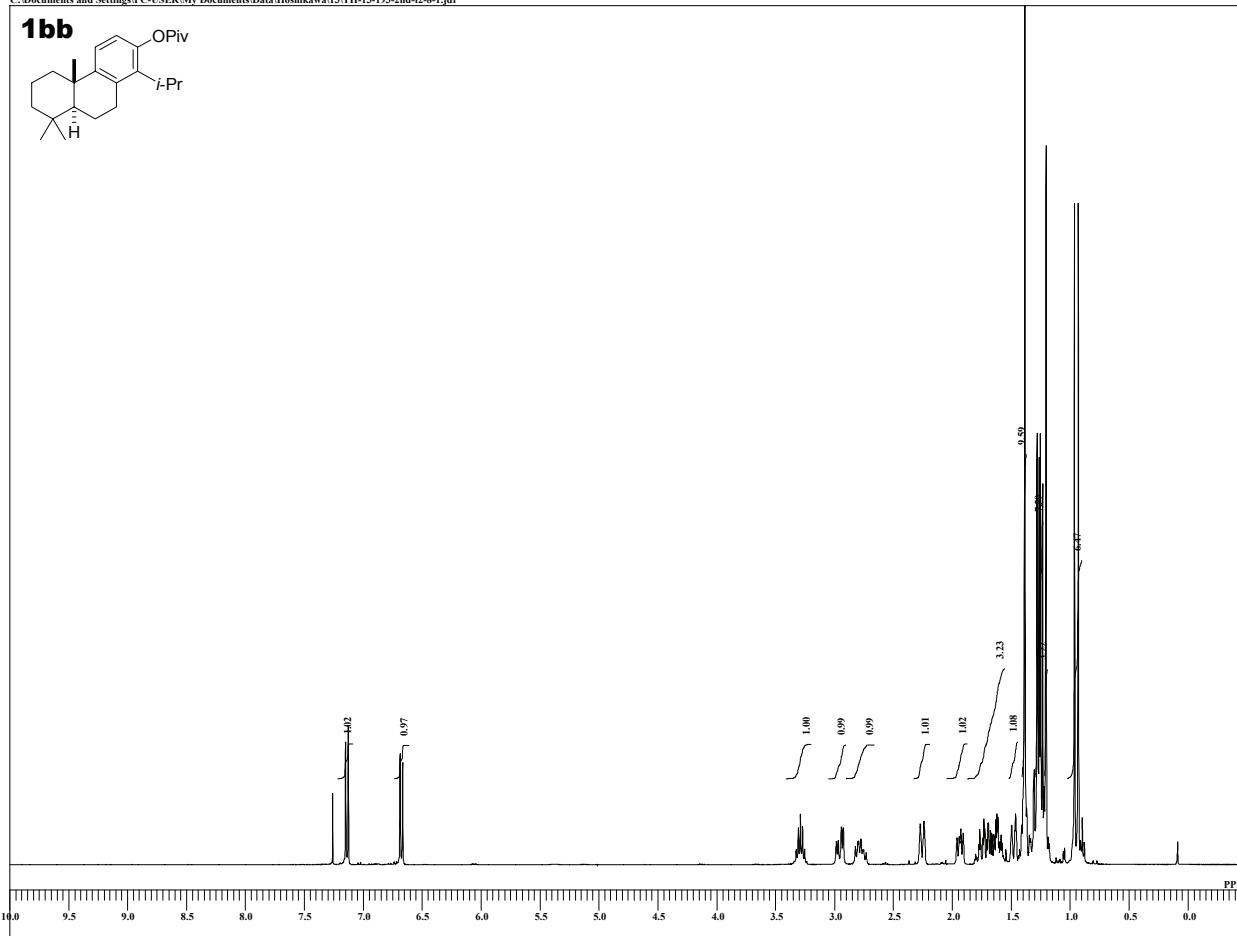
```

DFILE TH-41-1-3rd-f28-38C-1.als
COMNT TH-41-1-3rd-f28-38C
DATIM 07-03-2013 06:26:25
MENUF
OBNUC
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 kHz
OBFIN 0.98 Hz
PWI 3.14 sec
PREADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 32768
IRATN 32768
TIMES 3400
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
ACQTM 1.0480 sec
PD 8.0000 sec
SCANS 3600
ADBIT 16
RGAIN 60
BF 1.016 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC IH
IFR 395.88 MHz
IRSET 6.28 kHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE TH-41-1-3rd-f28-38C-1.als
LKSET 13.20 kHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 20.4 c
SLVNT CDCL3
EXREF 77.00 ppm

```

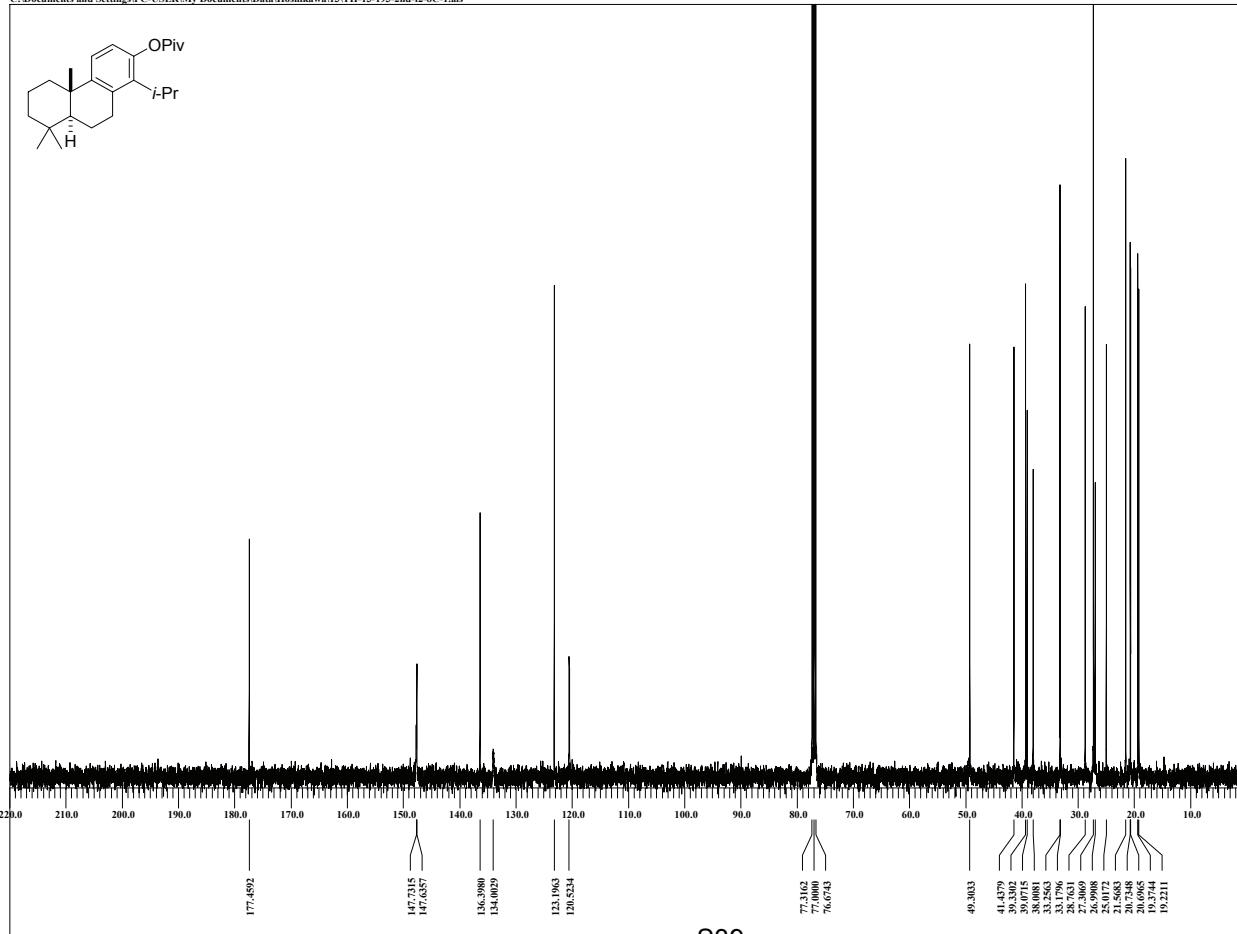
TH-13-193-2nd-f2-8

C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\13\TH-13-193-2nd-f2-8-1.jdf



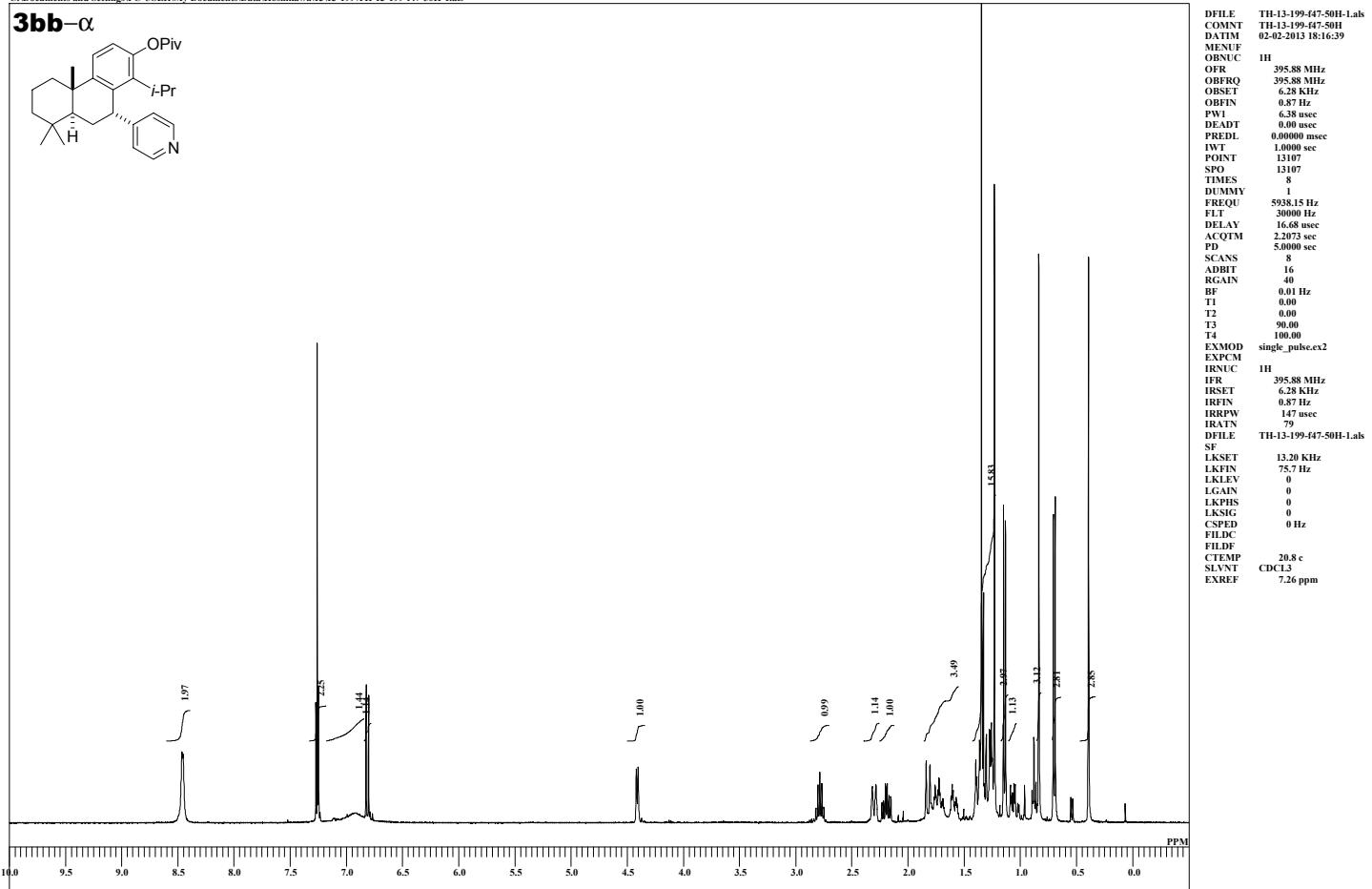
TH-13-193-2nd-f2-8C

C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\13\TH-13-193-2nd-f2-8C-1.als



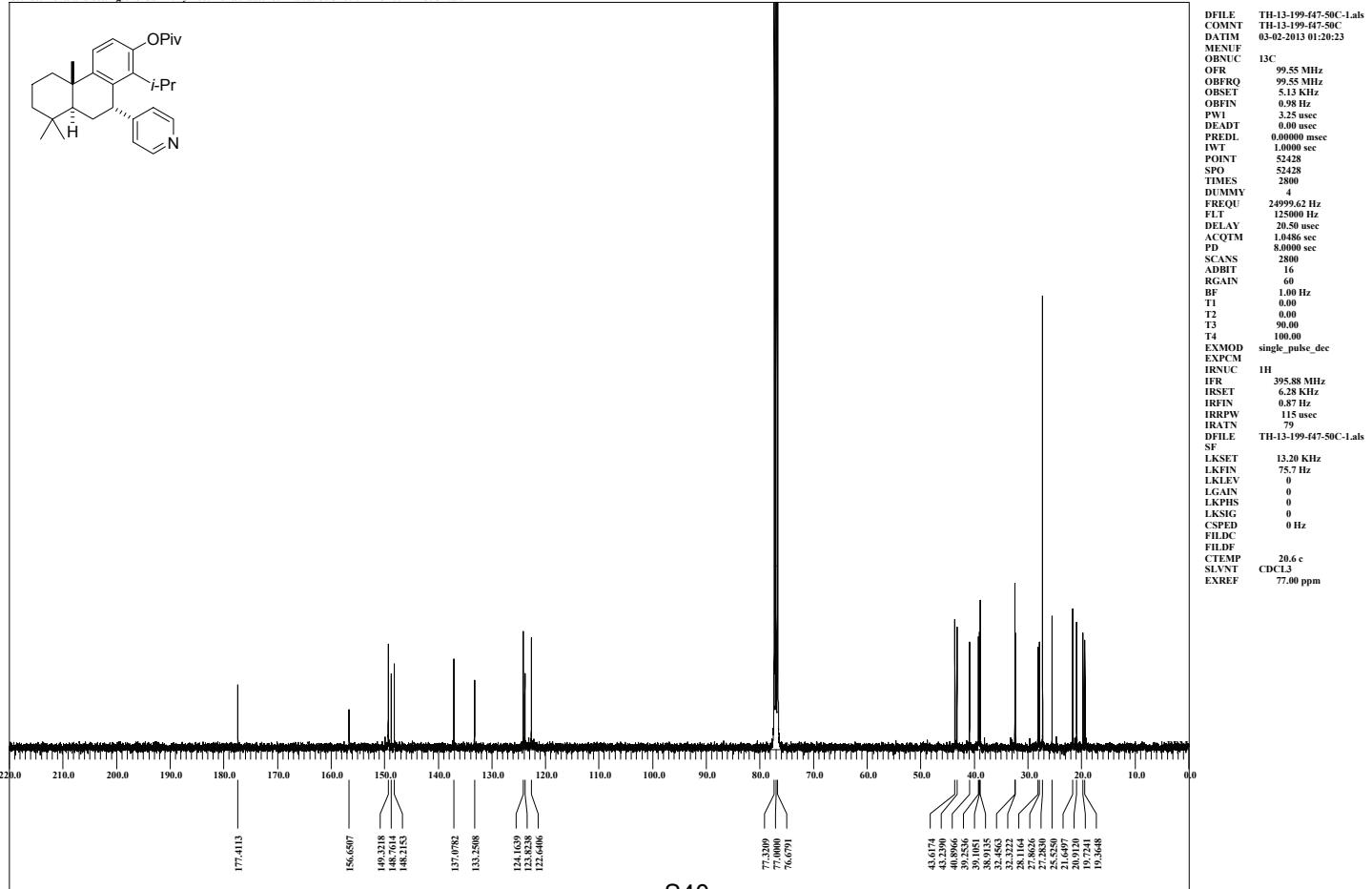
TH-13-199-f47-50H

C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\13\13-199\TH-13-199-f47-50H-labs



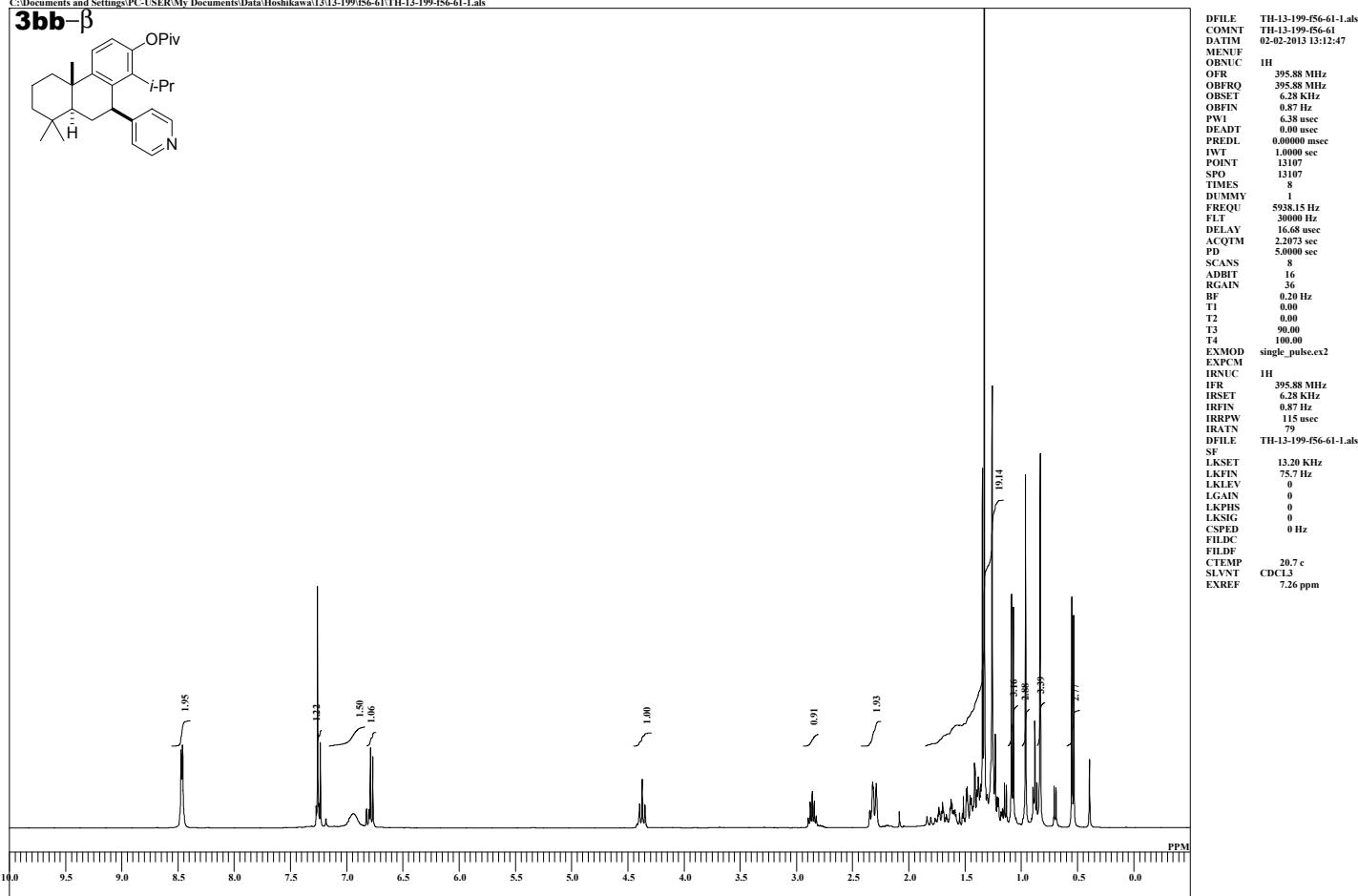
TH-13-199-f47-50C

C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\13\13-199\TH-13-199-f47-50C-labs



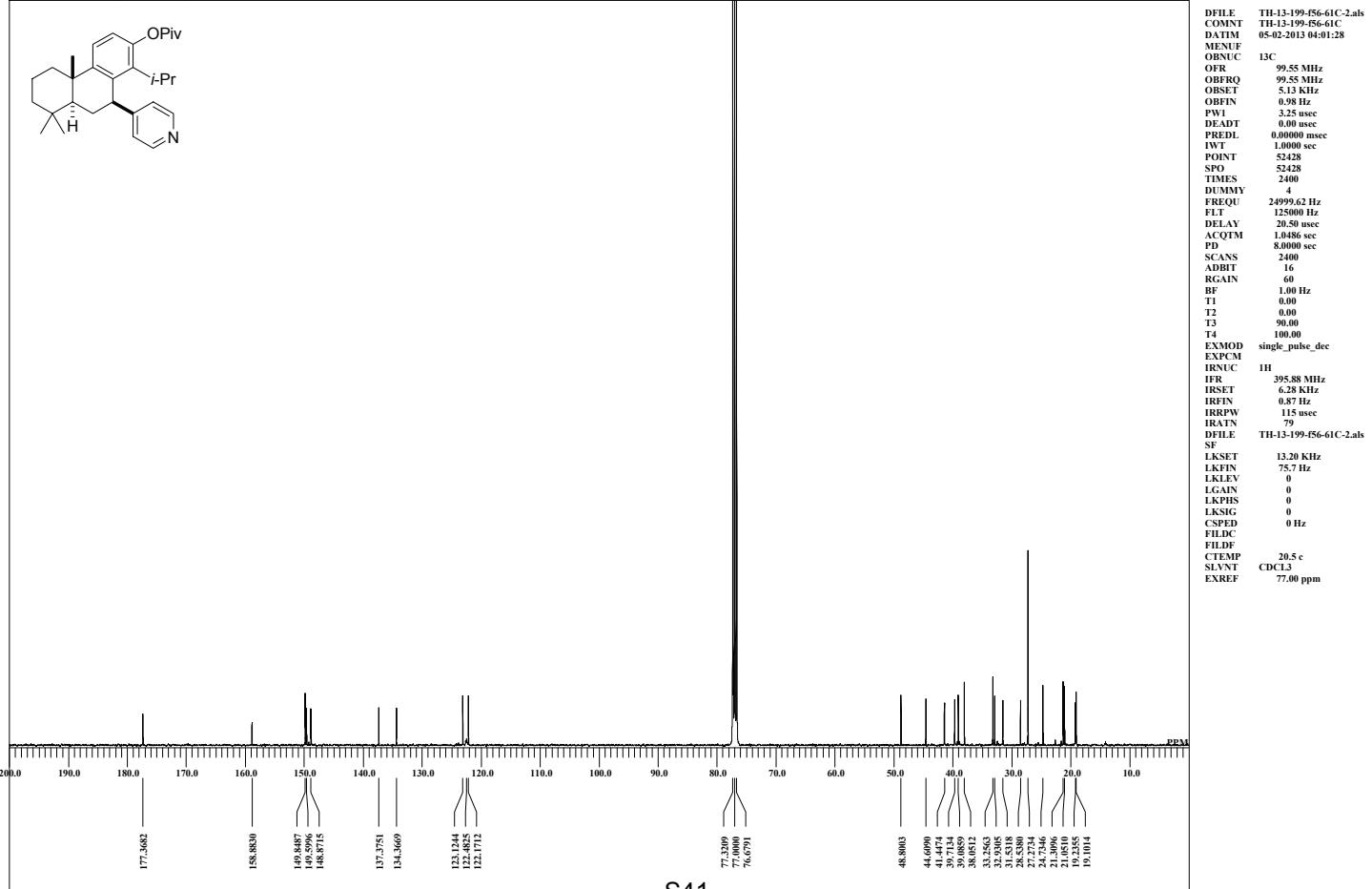
TH-13-199-f56-61

C:\Documents and Settings\PC-USER\My Documents\DATA\Hoshikawa\13\13-199\f56-61\TH-13-199-f56-61-1.als



TH-13-199-f56-61C

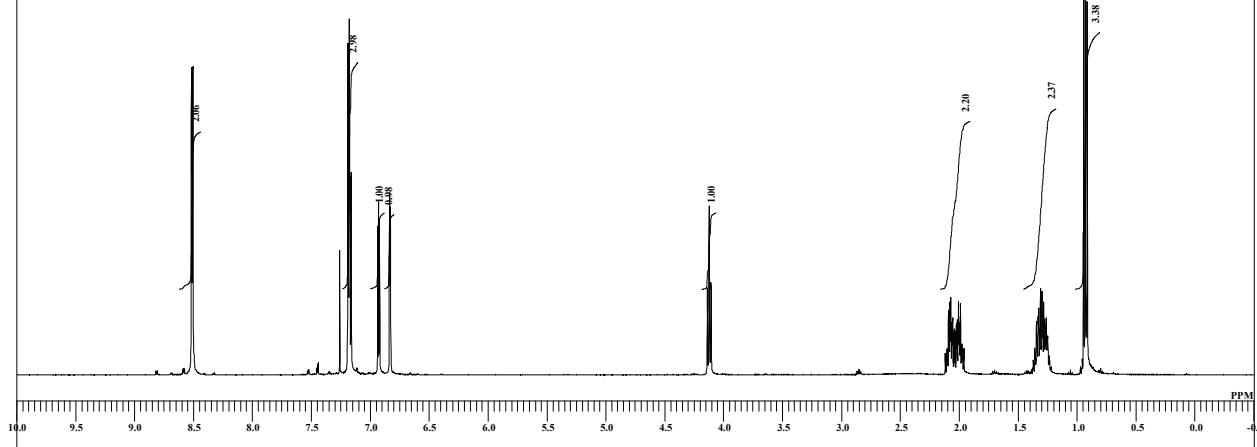
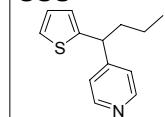
C:\Documents and Settings\PC-USER\My Documents\DATA\Hoshikawa\13\TH-13-199\f56-61C-2.als



TH-13-93-f20-30

C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\13\TH-13-93-f20-30.1

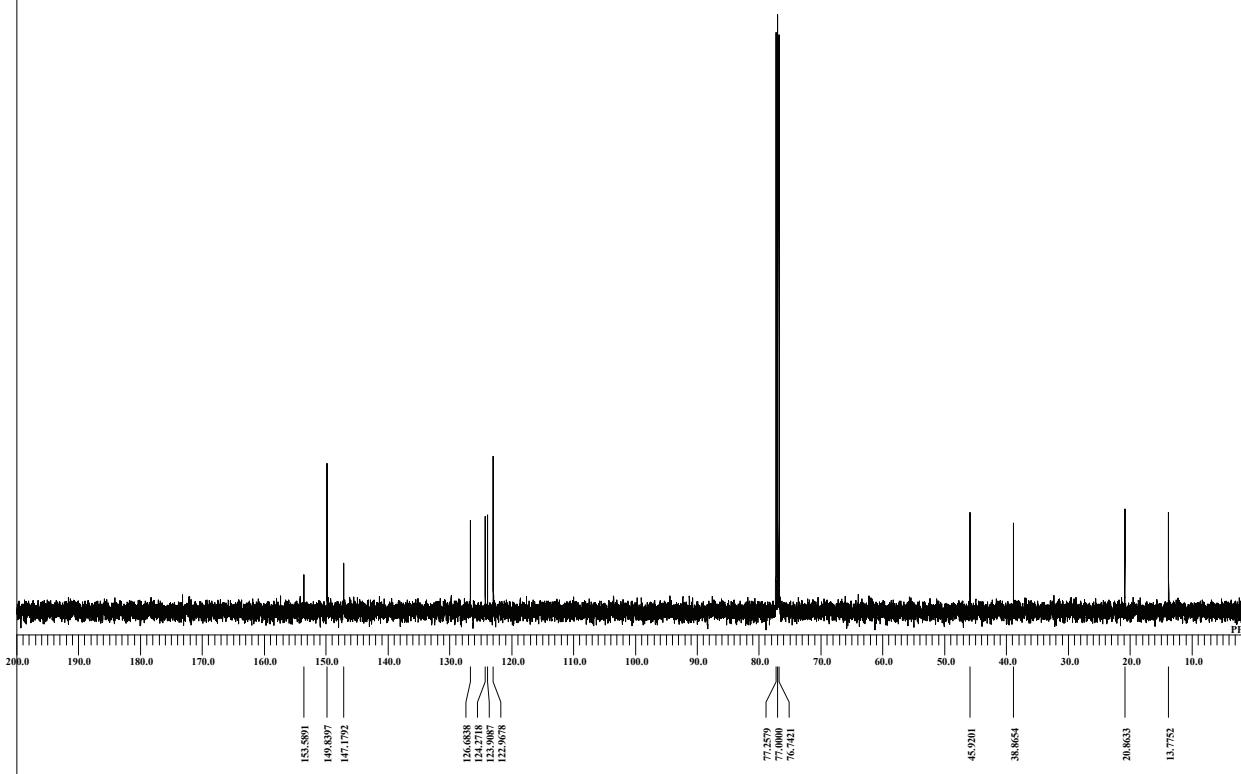
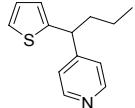
**3cc**



DFILE TH-13-93-f20-30.1  
 COMNT TH-13-93-f20-30  
 DATIM 07-10-2012 21:19:45  
 MENUF  
 OBNUC  
 OFR 490.15 MHz  
 OBFRQ 9.16 kHz  
 OBSET 6.71 Hz  
 OBFIN 32.5 Hz  
 PWR 6.50 sec  
 PREADT 0.0000 sec  
 PREDL 0.0000 msec  
 IWT 1.0000 sec  
 POINT 16384  
 SPO 16384  
 TIMES 8  
 DUMMY 1  
 FREQU 9191.18 Hz  
 FLT 37000 Hz  
 DELAY 13.52 usec  
 ACQTM 1.7826 sec  
 PD 5.0000 sec  
 SCANS 8  
 ADBIT 16  
 RGAIN 36  
 BF 0.01 Hz  
 T1 0.00  
 T2 0.00  
 T3 90.00  
 T4 100.00  
 EXMOD single\_pulse.ex2  
 EXPDM  
 IRNUC 1H  
 IRSET 490.15 MHz  
 IRFIN 9.16 kHz  
 IRRIN 7.60 Hz  
 IRRPW 92 usec  
 IRATN 79  
 DFILM TH-13-93-f20-30.1  
 LKSET 70.30 kHz  
 LKFIN 32.5 Hz  
 LKLEV 0  
 LGAIN 0  
 LKPHS 0  
 LKSIG 0  
 CSPED 0 Hz  
 FILDC  
 FILDF  
 CTEMP 25.3 c  
 SLVNT CDCL3  
 EXREF 7.26 ppm

TH-13-93-f20-30C

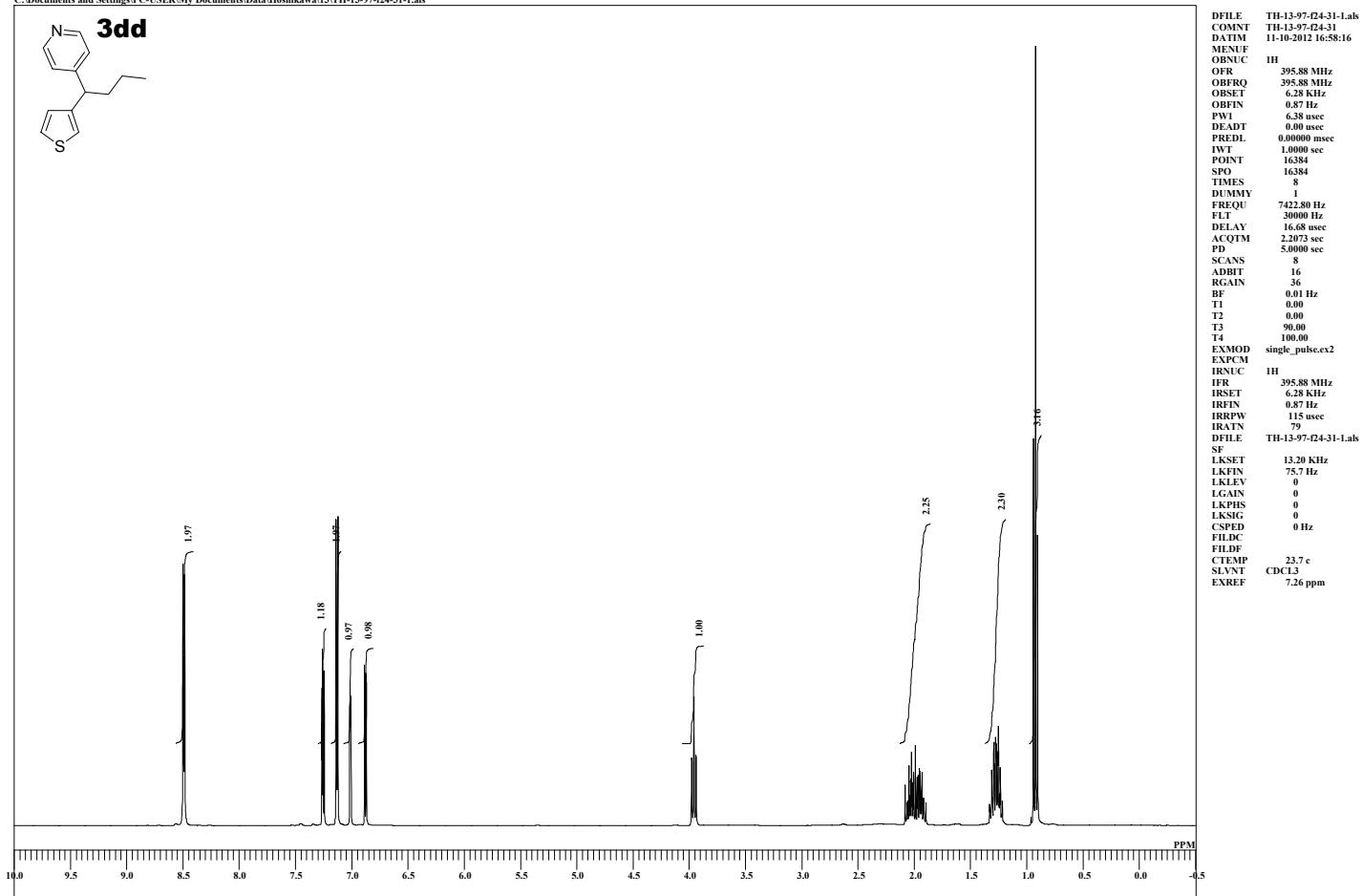
C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\13\TH-13-93-f20-30C.1



DFILE TH-13-93-f20-30C.1  
 COMNT TH-13-93-f20-30C  
 DATIM 07-10-2012 21:26:57  
 MENUF  
 OBNUC 13C  
 OFR 123.26 MHz  
 OBFRQ 123.26 MHz  
 OBSET 2.31 kHz  
 OBFIN 6.71 Hz  
 PWR 3.12 sec  
 PREADT 0.00 usec  
 PREDL 0.00000 msec  
 IWT 1.0000 sec  
 POINT 65536  
 ACQTM 0.8493 sec  
 PD 8.0000 sec  
 SCANS 34  
 ADBIT 16  
 RGAIN 60  
 BF 1.00 Hz  
 T1 0.00  
 T2 0.00  
 T3 90.00  
 T4 100.00  
 EXMOD single\_pulse\_dec  
 EXPDM  
 IRNUC 1H  
 IFR 490.15 MHz  
 IRSET 9.16 kHz  
 IRFIN 7.60 Hz  
 IRRPW 92 usec  
 IRATN 79  
 DFILM TH-13-93-f20-30C.1  
 SF  
 LKSET 70.30 kHz  
 LKFIN 32.5 Hz  
 LKLEV 0  
 LGAIN 0  
 LKPHS 0  
 LKSIG 0  
 CSPED 0 Hz  
 FILDC  
 FILDF  
 CTEMP 25.9 c  
 SLVNT CDCL3  
 EXREF 77.00 ppm

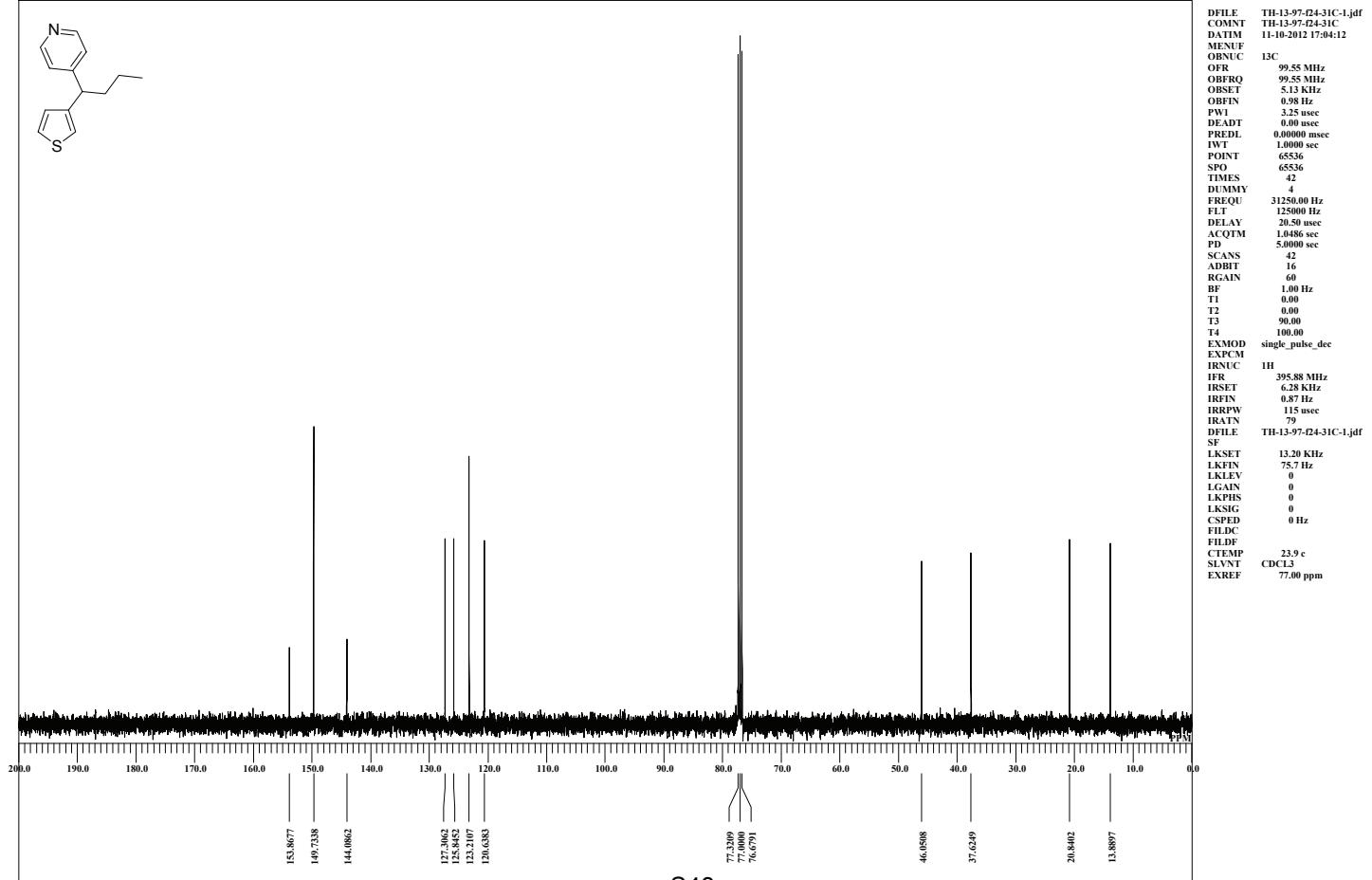
TH-13-97-f24-31

C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\13\TH-13-97-f24-31-1.xls



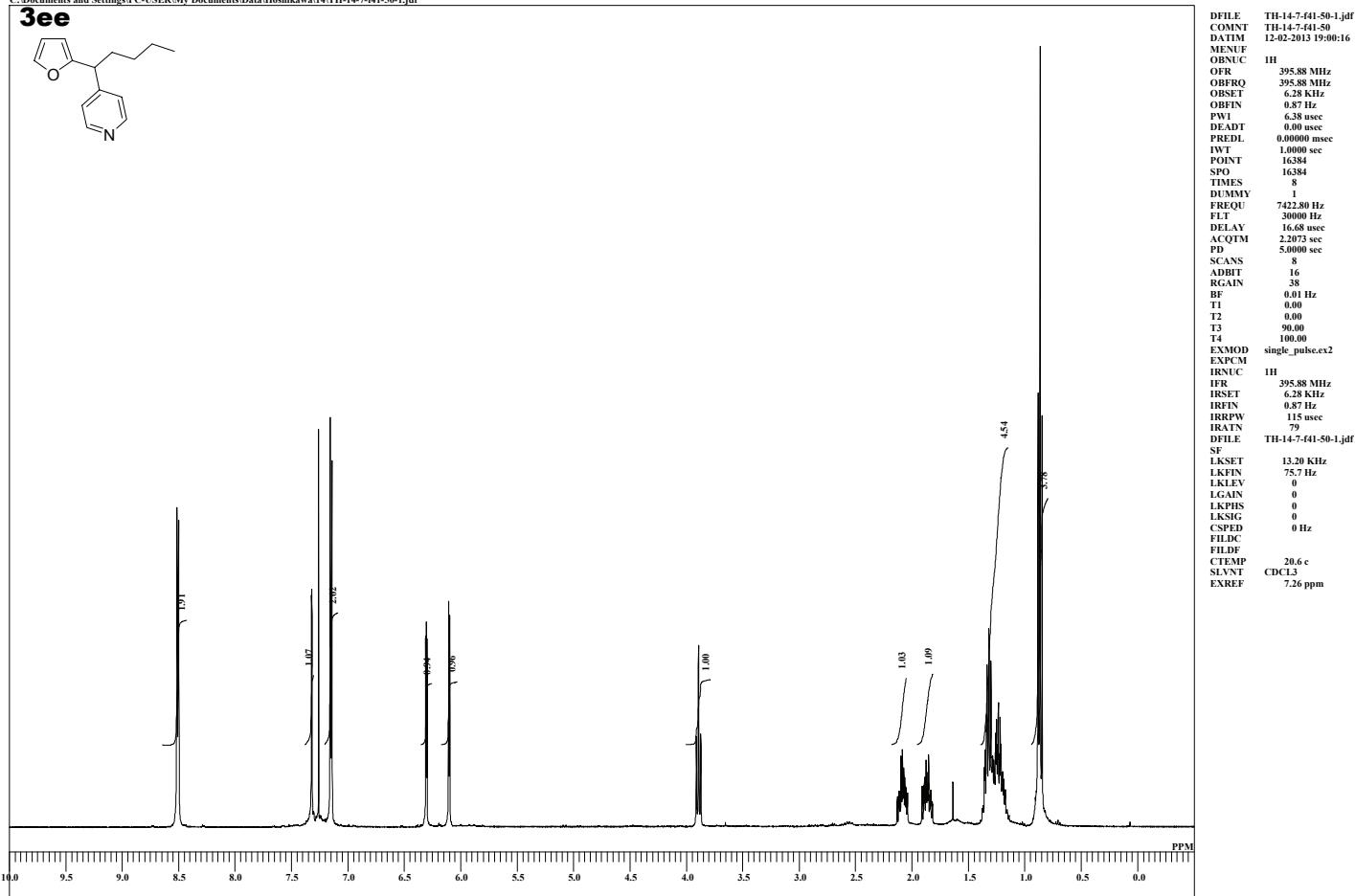
TH-13-97-f24-31C

C:\Documents and Settings\PC-USER\My Documents\Data\Hoshikawa\13\TH-13-97-f24-31C-1.jdf

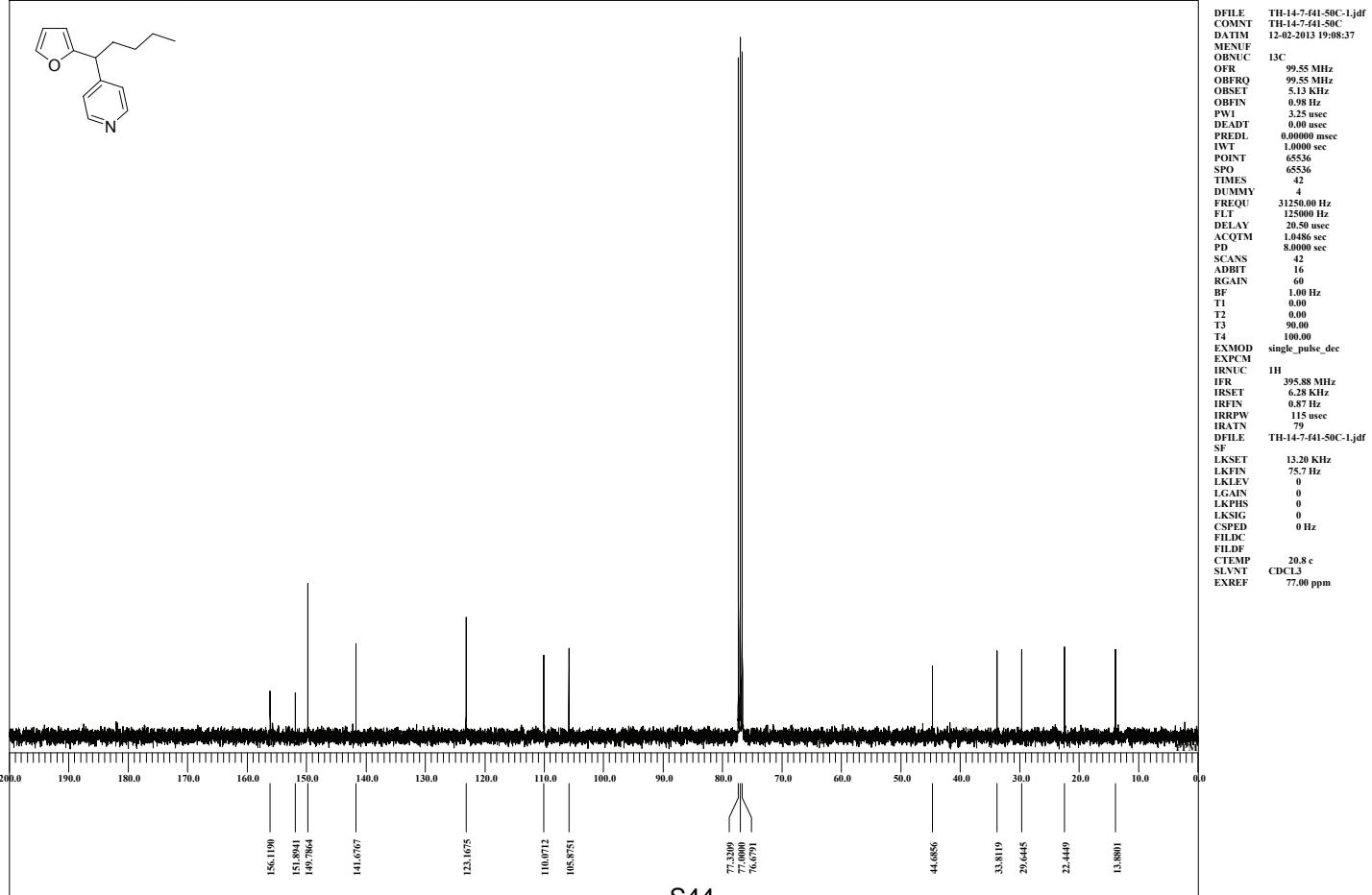


TH-14-7-f41-50

C:\Documents and Settings\PC-USER\My Documents\DATA\Hoshikawa\14\TH-14-7-f41-50-1.jdf



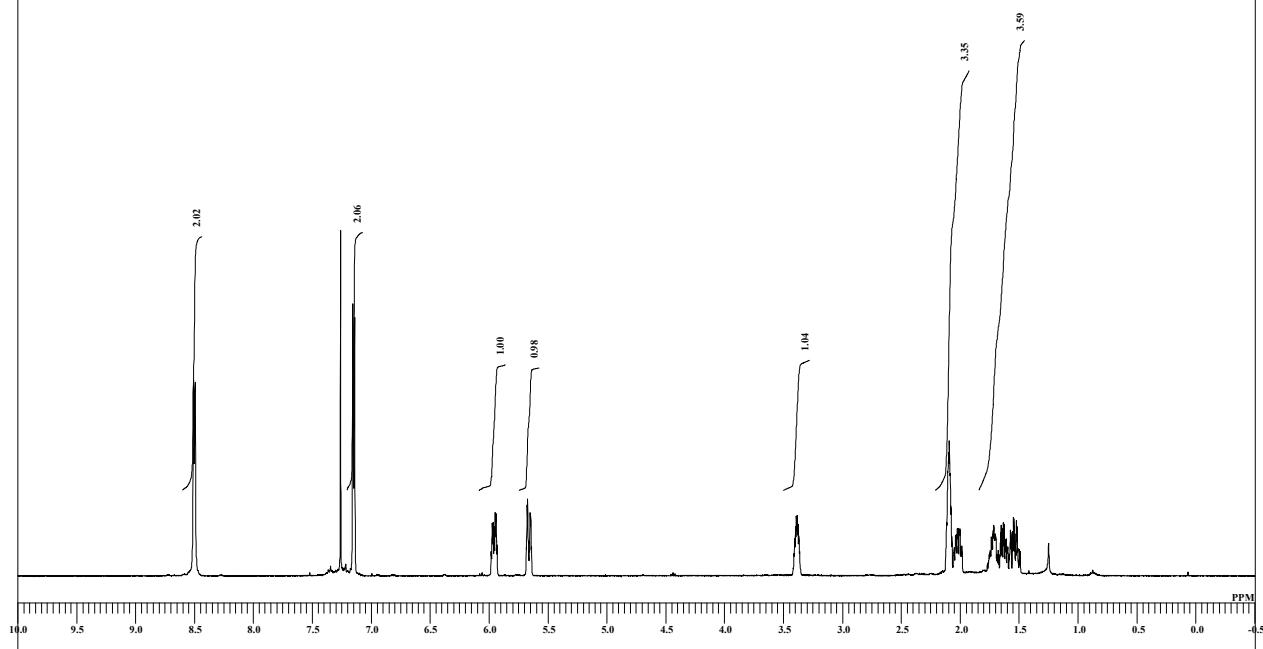
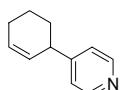
C:\Documents and Settings\PC-USER\My Documents\DATA\Hoshikawa\14\TH-14-7-f41-50C-1.jdf



TH-13-189-f47-54

C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\13\TH-13-189-f47-54-1.als

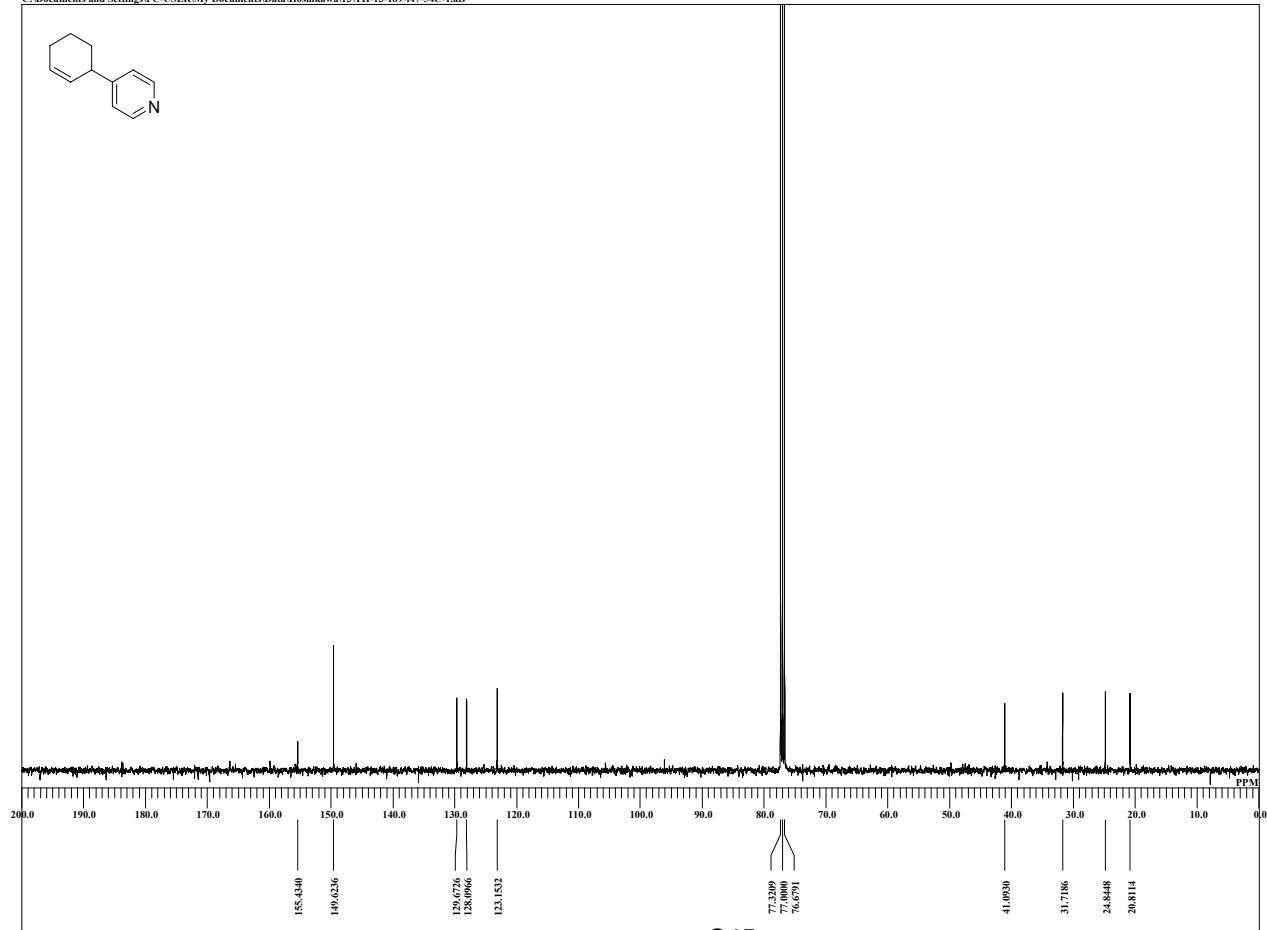
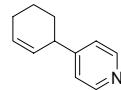
**3ff**



DFILE TH-13-189-f47-54-1.als  
 COMNT TH-13-189-f47-54  
 DATIM 13-02-2013 20:40:30  
 MENUF  
 OBNUC  
 OFR 395.88 MHz  
 OBFRQ 395.88 MHz  
 OBSET 6.28 kHz  
 OBFIN 0.87 Hz  
 PW1 6.38 sec  
 DE1 0.00 usec  
 PREDL 0.00000 msec  
 IWT 1.0000 sec  
 POINT 16384  
 SPO 16384  
 TIMES 8  
 DUMMY 1  
 FREQU 7422.80 Hz  
 FLT 30000 Hz  
 DELAY 16.68 usec  
 ACQTM 2.2072 sec  
 PD 5.0000 sec  
 SCANS 8  
 ADBIT 16  
 RGAIN 42  
 BF 0.01 Hz  
 T1 0.00  
 T2 0.00  
 T3 90.00  
 T4 100.00  
 EXMOD single\_pulse.ex2  
 EXPDM  
 IRNUC IH  
 IRSET 395.88 MHz  
 IRFIN 6.28 kHz  
 IRFIN 0.87 Hz  
 IRRPW 115 usec  
 IRATN 79  
 DFILM TH-13-189-f47-54-1.als  
 LKSET 13.20 kHz  
 LKFIN 75.7 Hz  
 LKLEV 0  
 LGAIN 0  
 LKPHS 0  
 LKSIG 0  
 CSPED 0 Hz  
 FILDC  
 FILDF  
 CTEMP 20.4 c  
 SLVNT CDCl<sub>3</sub>  
 EXREF 7.26 ppm

TH-13-189-f47-54C

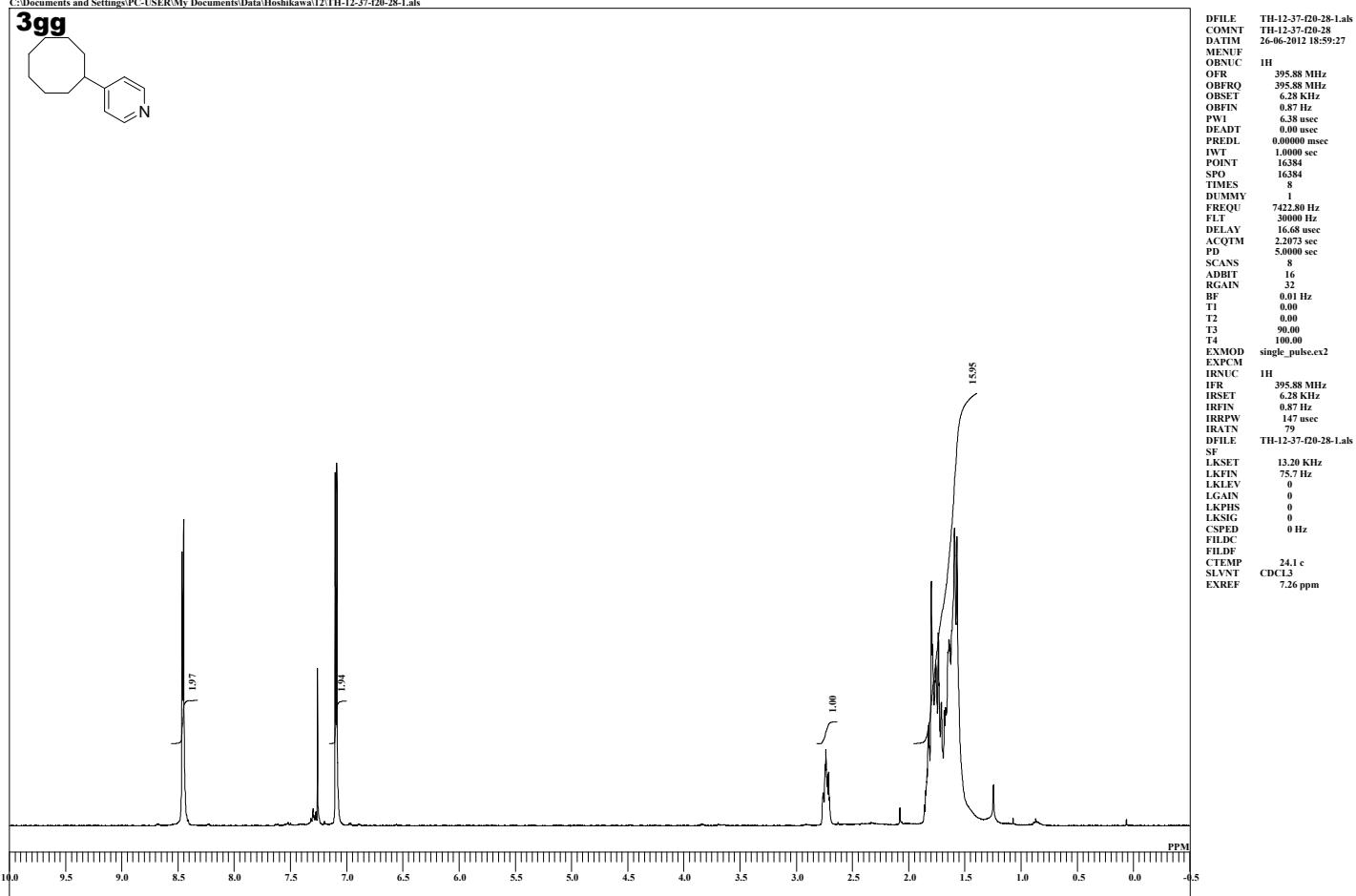
C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\13\TH-13-189-f47-54C-1.als



DFILE TH-13-189-f47-54C-1.als  
 COMNT TH-13-189-f47-54C  
 DATIM 13-02-2013 20:49:03  
 MENUF  
 OBNUC 99.55 MHz  
 OBFRQ 99.55 MHz  
 OBSET 5.13 kHz  
 OBFIN 0.98 Hz  
 PW1 3.24 sec  
 DE1 0.00 usec  
 PREDL 0.00000 msec  
 IWT 1.0000 sec  
 POINT 52428  
 TIMES 42  
 DUMMY 42  
 FREQU 24999.62 Hz  
 FLT 125000 Hz  
 DELAY 20.50 usec  
 ACQTM 1.0486 sec  
 PD 8.0000 sec  
 SCANS 42  
 ADBIT 16  
 RGAIN 60  
 BF 1.01 Hz  
 T1 0.00  
 T2 0.00  
 T3 90.00  
 T4 100.00  
 EXMOD single\_pulse\_dec  
 EXPDM  
 IRNUC IH  
 IRSET 395.88 MHz  
 IRFIN 6.28 kHz  
 IRFIN 0.87 Hz  
 IRRPW 115 usec  
 IRATN 79  
 DFILM TH-13-189-f47-54C-1.als  
 SF  
 LKSET 13.20 kHz  
 LKFIN 75.7 Hz  
 LKLEV 0  
 LGAIN 0  
 LKPHS 0  
 LKSIG 0  
 CSPED 0 Hz  
 FILDC  
 FILDF  
 CTEMP 20.7 c  
 SLVNT CDCl<sub>3</sub>  
 EXREF 77.00 ppm

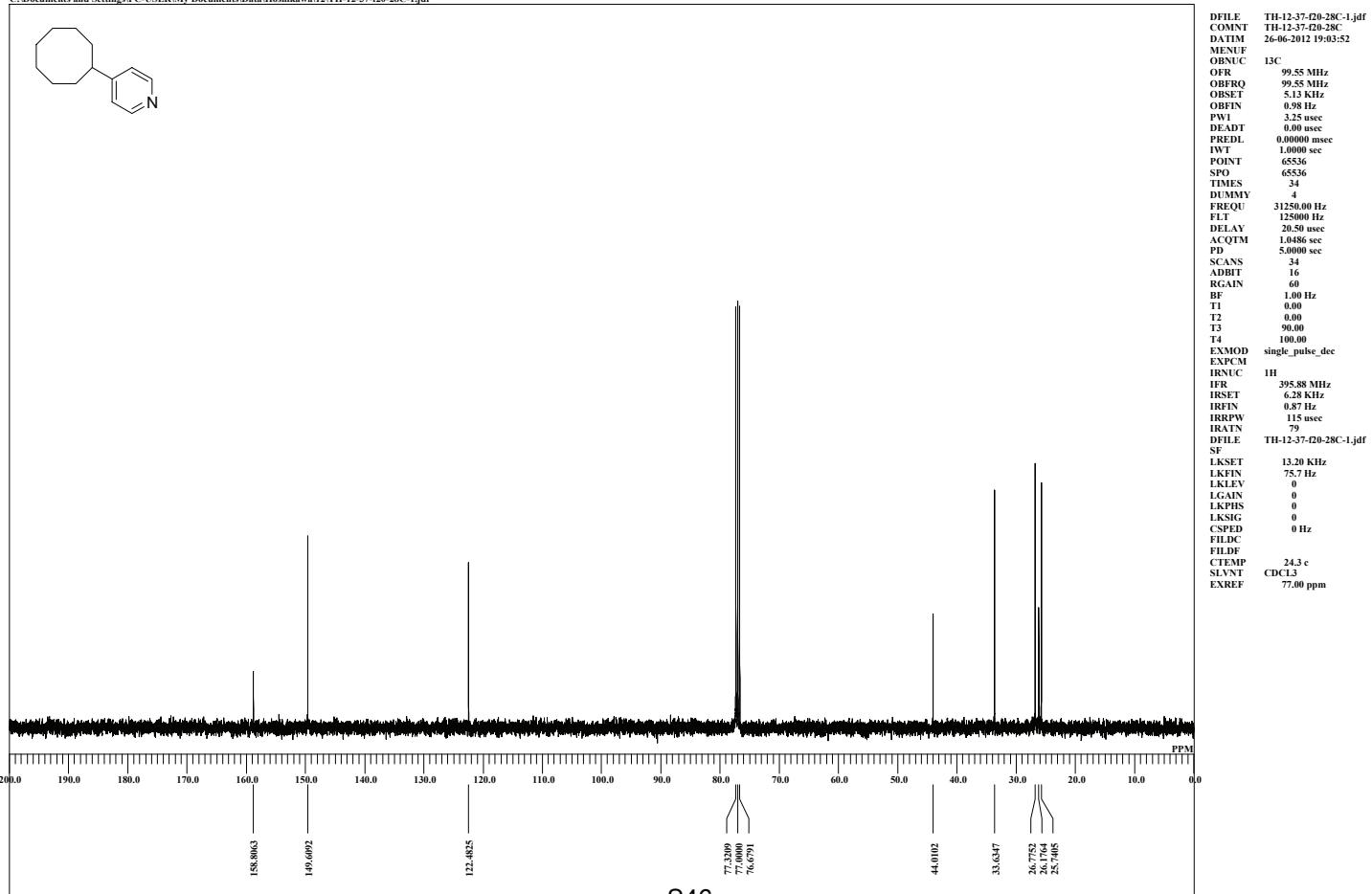
TH-12-37-f20-28

C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\12\TH-12-37-f20-28-1.als



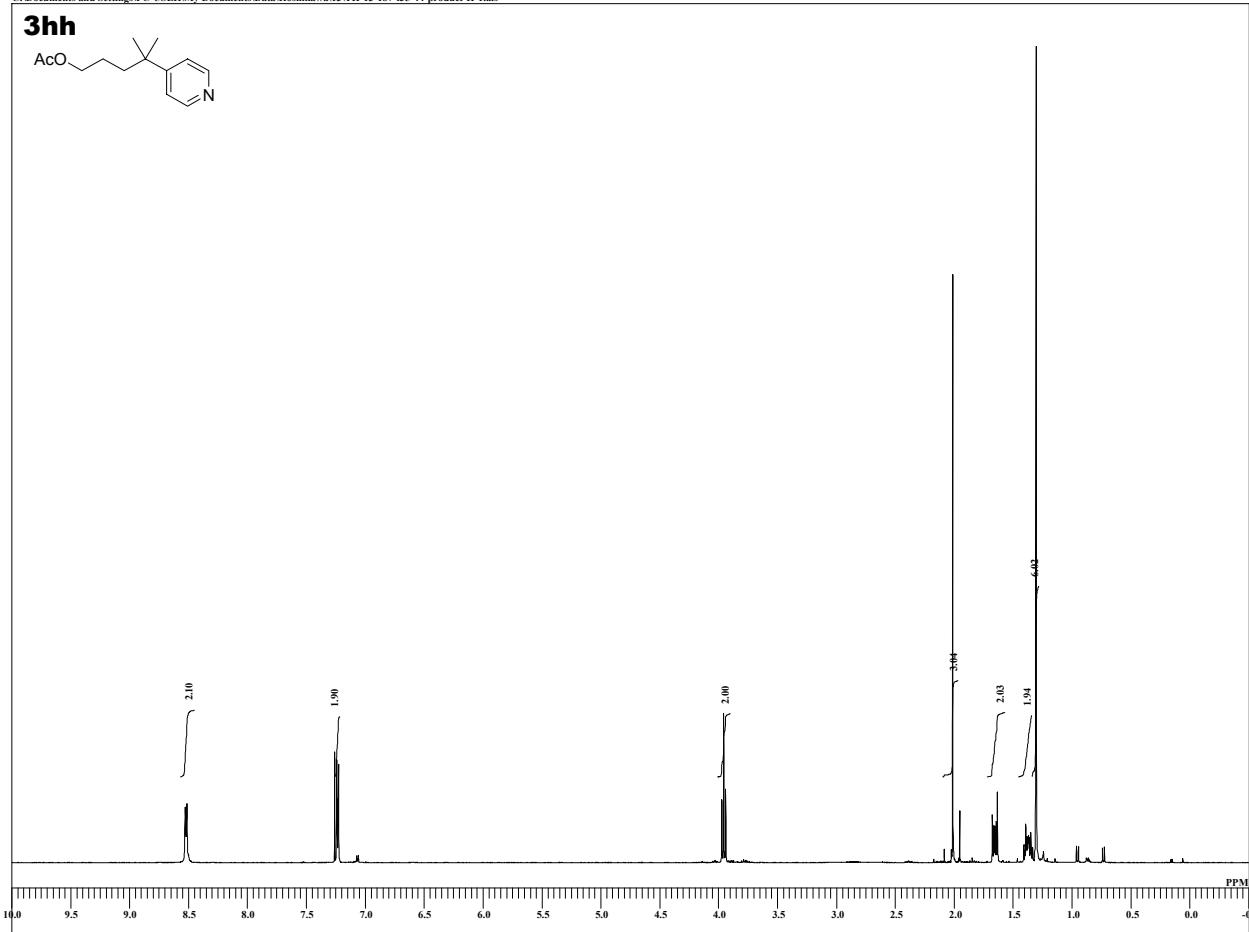
TH-12-37-f20-28C

C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\12\TH-12-37-f20-28C-1.jdf



TH-13-187-f35-44-product-H

C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\13\TH-13-187-f35-44-product-H-1.als



TH-13-187-f35-44-product-C

C:\Documents and Settings\PC-USER\My Documents\Hoshikawa\13\TH-13-187-f35-44-product-C-1.jdf

