

Supporting Information

Cu-Catalyzed Mild and Efficient Oxidation of TH β Cs Using Air: Application in Practical Total Syntheses of Perlolyrine and Flazin

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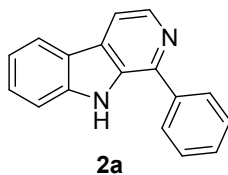
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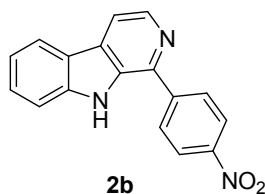
1. Characterization Data of β -Carbolines 2a-2x:

1-Phenyl-9H-pyrido[3,4-b]indole 2a:



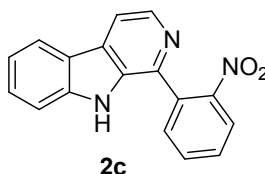
Pale yellow solid, mp 240-241 °C. ^1H NMR (DMSO- d_6 , 400 MHz) δ 11.54 (s, 1H, NH in indole), 8.48 (d, $J = 5.2$ Hz, 1H), 8.27 (d, $J = 7.9$ Hz, 1H), 8.13 (d, $J = 5.2$ Hz, 1H), 8.05 (d, $J = 7.8$ Hz, 2H), 7.47-7.72 (m, 5H), 7.28 (t, $J = 7.6$ Hz, 1H). ^{13}C NMR (DMSO- d_6 , 100 MHz) δ 142.51, 141.44, 138.75, 138.69, 133.34, 129.49, 129.03 (2C), 128.80, 128.71 (2C), 128.46, 121.88, 121.16, 119.80, 114.17, 112.75. HRMS (ESI) m/z calcd for $\text{C}_{17}\text{H}_{13}\text{N}_2$ $[\text{M}+\text{H}]^+$: 245.1079, found: 245.1075. IR (KBr film) 3061, 2954, 2877, 1623, 1560, 1496, 1415, 1322, 1234, 737, 698 cm^{-1} .

1-(4-Nitrophenyl)-9H-pyrido[3,4-b]indole 2b:



Pale yellow solid, mp. 242-243 °C. ^1H NMR (DMSO- d_6 , 400 MHz) δ 11.74 (s, 1H, NH in indole), 8.53 (d, $J = 5.1$ Hz, 1H), 8.44 (d, $J = 8.5$ Hz, 2H), 8.28-8.35 (m, 3H), 8.23 (d, $J = 5.1$ Hz, 1H), 7.66 (d, $J = 8.0$ Hz, 1H), 7.59 (dd, $J_1 = 8.0$ Hz, $J_2 = 7.9$ Hz, 1H), 7.30 (dd, $J_1 = 7.8$ Hz, $J_2 = 7.9$ Hz, 1H). ^{13}C NMR (DMSO- d_6 , 100 MHz) δ 147.02, 144.54, 141.20, 139.37, 138.63, 133.35, 129.86, 129.50 (2C), 128.53, 123.74 (2C), 121.70, 120.65, 119.77, 115.09, 112.35. HRMS (ESI) m/z calcd for $\text{C}_{17}\text{H}_{12}\text{N}_3\text{O}_2$ $[\text{M}+\text{H}]^+$: 290.0930, found: 290.0942. IR (KBr film) 3368, 3060, 2922, 2847, 1624, 1599, 1510, 1455, 1425, 1348, 1226, 1103, 855, 831, 757, 740, 531 cm^{-1} .

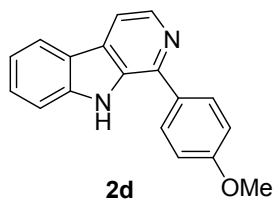
1-(2-Nitrophenyl)-9H-pyrido[3,4-b]indole 2c:



Pale yellow solid, mp. 241-242 °C. ^1H NMR (CDCl_3 , 400 MHz) δ 8.47 (s, 1H, NH in indole), 8.45 (d, $J = 5.2$ Hz, 1H), 8.13 (d, $J = 8.0$ Hz, 1H), 8.02 (d, $J = 8.1$ Hz, 1H), 7.95 (d, $J = 5.2$ Hz, 1H), 7.72 (d, $J = 8.0$ Hz, 1H), 7.68 (dd, $J_1 = 8.1$ Hz, $J_2 = 8.0$ Hz, 1H), 7.57 (dd, $J_1 = 8.1$ Hz, $J_2 = 8.0$ Hz, 1H), 7.52 (dd, $J_1 = 8.1$ Hz, $J_2 = 8.0$ Hz, 1H), 7.38 (d, $J = 8.1$ Hz, 1H), 7.30 (dd, $J_1 = 8.1$ Hz, $J_2 = 8.0$ Hz, 1H). ^{13}C NMR (DMSO-

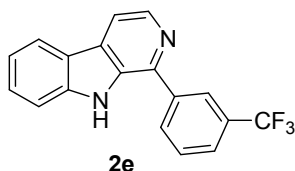
d_6 , 100 MHz) δ 149.16, 141.04, 139.34, 138.08, 133.51, 133.17, 132.59, 131.58, 129.76, 128.97, 128.43, 124.70, 121.81, 120.79, 119.65, 114.59, 112.21. HRMS (ESI) m/z calcd for $C_{17}H_{12}N_3O_2$ $[M+H]^+$: 290.0930, found: 290.0938. IR (KBr film) 3359, 3068, 2923, 2855, 1626, 1566, 1524, 1420, 1320, 1235, 738 cm^{-1} .

1-(4-Methoxyphenyl)-9H-pyrido[3,4-b]indole 2d:



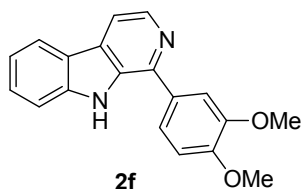
White solid, mp 158-159°C. 1H NMR (DMSO- d_6 , 400 MHz) δ 11.48 (s, 1H, NH in indole), 8.43 (d, $J = 5.2$ Hz, 1H), 8.23 (d, $J = 7.9$ Hz, 1H), 8.05 (d, $J = 5.2$ Hz, 1H), 8.02 (d, $J = 8.2$ Hz, 2H), 7.67 (d, $J = 8.1$ Hz, 1H), 7.55 (dd, $J_1 = 8.1$ Hz, $J_2 = 8.0$ Hz, 1H), 7.25 (dd, $J_1 = 7.9$ Hz, $J_2 = 8.0$ Hz, 1H), 7.17 (d, $J = 8.2$ Hz, 2H), 3.87 (s, 3H). ^{13}C NMR (DMSO- d_6 , 100 MHz) δ 160.06, 142.60, 141.53, 138.72, 133.24, 131.39, 130.14 (2C), 129.46, 128.46, 121.97, 121.37, 119.88, 114.57 (2C), 113.74, 112.88, 55.73. HRMS (ESI) m/z calcd for $C_{18}H_{15}N_2O$ $[M+H]^+$: 275.1184, found: 275.1180. IR (KBr film) 3141, 3064, 2980, 1626, 1610, 1514, 1470, 1430, 1322, 1254, 1238, 1175, 1038, 742 cm^{-1} .

1-(3-(Trifluoromethyl)-phenyl)-9H-pyrido[3,4-b]indole 2e:



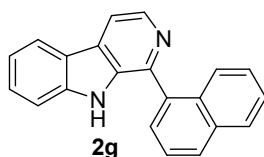
White solid, mp. 140-141°C. 1H NMR ($CDCl_3$, 400 MHz) δ 8.76 (s, 1H, NH in indole), 8.58 (d, $J = 5.2$ Hz, 1H), 8.21 (s, 1H), 8.17 (d, $J = 7.9$ Hz, 1H), 8.10 (d, $J = 7.7$ Hz, 1H), 7.98 (d, $J = 5.2$ Hz, 1H), 7.70 (d, $J = 7.8$ Hz, 1H), 7.64 (dd, $J_1 = 7.9$ Hz, $J_2 = 7.8$ Hz, 1H), 7.57 (dd, $J_1 = 7.8$ Hz, $J_2 = 7.7$ Hz, 1H), 7.51 (d, $J = 8.0$ Hz, 1H), 7.33 (dd, $J_1 = 7.8$ Hz, $J_2 = 8.0$ Hz, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 141.18, 140.82, 139.18, 139.10, 133.64, 131.33, 131.28 (q, $^2J_{CF} = 32.5$ Hz), 130.45, 129.37, 128.76, 125.30 (q, $^3J_{CF} = 4.1$ Hz), 124.98 (q, $^3J_{CF} = 4.1$ Hz), 123.95 (q, $^1J_{CF} = 272.6$ Hz), 121.79, 121.68, 120.40, 114.49, 111.77. HRMS (ESI) m/z calcd for $C_{18}H_{12}F_3N_2$ $[M+H]^+$: 313.0953, found: 313.0950. IR (KBr film) 3127, 3062, 2955, 2876, 1626, 1566, 1499, 1456, 1338, 1276, 1234, 1167, 1125, 1071, 807, 744, 699 cm^{-1} .

1-(3,4-Dimethoxyphenyl)-9H-pyrido[3,4-b]indole 2f:



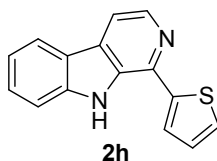
White solid, mp 106-107°C. ¹H NMR (CDCl₃, 400 MHz) δ 9.07 (s, 1H, NH in indole), 8.53 (d, *J* = 5.2 Hz, 1H), 8.15 (d, *J* = 8.0 Hz, 1H), 7.91 (d, *J* = 5.2 Hz, 1H), 7.43-7.57 (m, 5H), 7.30 (dd, *J*₁ = 7.9 Hz, *J*₂ = 8.0 Hz, 1H), 6.89 (d, *J* = 8.0 Hz, 1H), 3.88 (s, 3H), 3.85 (s, 3H). ¹³C NMR (DMSO-*d*₆, 100 MHz) δ 149.76, 149.38, 142.77, 141.51, 138.65, 133.31, 131.58, 129.44, 128.48, 122.00, 121.37, 121.24, 119.89, 113.81, 112.90, 112.29, 112.23, 56.14, 55.89. HRMS (ESI) *m/z* calcd for C₁₉H₁₇N₂O₂ [M+H]⁺: 305.1290, found: 305.1282. IR (KBr film) 3363, 2935, 1626, 1516, 1457, 1409, 1319, 1260, 1144, 1025, 747 cm⁻¹.

1-(Naphthalen-1-yl)-9H-pyrido[3,4-*b*]indole **2g**:



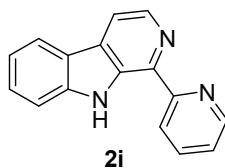
White solid, mp. 114-115 °C. ¹H NMR (DMSO-*d*₆, 400 MHz) δ 11.16 (s, 1H, NH in indole), 8.52 (d, *J* = 5.2 Hz, 1H), 8.30 (d, *J* = 7.9 Hz, 1H), 8.21 (d, *J* = 5.2 Hz, 1H), 8.12 (d, *J* = 7.8 Hz, 1H), 8.07 (d, *J* = 8.0 Hz, 1H), 7.68-7.77 (m, 3H), 7.48-7.58 (m, 3H), 7.44 (dd, *J*₁ = 7.8 Hz, *J*₂ = 7.9 Hz, 1H), 7.26 (dd, *J*₁ = 7.9 Hz, *J*₂ = 8.0 Hz, 1H). ¹³C NMR (DMSO-*d*₆, 100 MHz) δ 143.22, 141.40, 138.47, 136.09, 135.15, 134.11, 131.69, 129.15, 128.98, 128.77, 128.60, 127.98, 126.82, 126.52, 126.13, 126.04, 122.19, 121.34, 119.86, 114.48, 112.72. HRMS (ESI) *m/z* calcd for C₂₁H₁₅N₂ [M+H]⁺: 295.1235, found: 295.1242. IR (KBr film) 3409, 3130, 3054, 2924, 2853, 1624, 1563, 1454, 1419, 1319, 1235, 777, 744 cm⁻¹.

1-(Thiophen-2-yl)-9H-pyrido[3,4-*b*]indole **2h**:



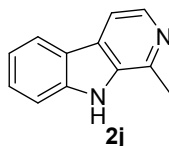
Pale yellow solid, mp. 165-166 °C. ¹H NMR (CDCl₃, 400 MHz) δ 8.90 (s, 1H, NH in indole), 8.48 (d, *J* = 5.2 Hz, 1H), 8.11 (d, *J* = 7.9 Hz, 1H), 7.86 (d, *J* = 5.2 Hz, 1H), 7.74 (d, *J* = 3.6 Hz, 1H), 7.50-7.58 (m, 2H), 7.46 (d, *J* = 5.0 Hz, 1H), 7.30 (dd, *J*₁ = 7.8 Hz, *J*₂ = 7.9 Hz, 1H), 7.18 (dd, *J*₁ = 5.0 Hz, *J*₂ = 3.6 Hz, 1H). ¹³C NMR (CDCl₃, 100 MHz) δ 142.83, 140.64, 139.18, 137.10, 132.27, 130.40, 128.64, 128.04, 127.20, 125.17, 121.80, 121.71, 120.49, 113.81, 111.83. HRMS (ESI) *m/z* calcd for C₁₅H₁₁N₂S [M+H]⁺: 251.0643, found: 251.0642. IR (KBr film) 3204, 3057, 2924, 2853, 1623, 1562, 1495, 1453, 1421, 1319, 1232, 743, 702 cm⁻¹.

1-(Pyridin-2-yl)-9H-pyrido[3,4-b]indole 2i:



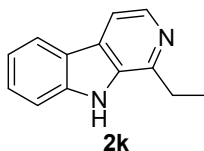
Pale yellow solid, mp 179-180 °C. ¹H NMR (CDCl₃, 400 MHz) δ 11.33 (s, 1H, NH in indole), 8.78 (d, *J* = 5.0 Hz, 1H), 8.75 (d, *J* = 8.0 Hz, 1H), 8.55 (d, *J* = 5.1 Hz, 1H), 8.16 (d, *J* = 7.9 Hz, 1H), 8.00 (d, *J* = 5.1 Hz, 1H), 7.89 (dd, *J*₁ = 7.9 Hz, *J*₂ = 8.0 Hz, 1H), 7.55-7.63 (m, 2H), 7.27-7.36 (m, 2H). ¹³C NMR (CDCl₃, 100 MHz) δ 157.98, 148.26, 140.63, 138.14, 138.09, 136.85, 134.77, 130.48, 128.44, 122.92, 121.69, 121.30, 121.15, 119.80, 115.47, 111.88. HRMS (ESI) *m/z* calcd for C₁₆H₁₂N₃ [M+H]⁺: 246.1031, found: 246.1032. IR (KBr film) 3397, 3296, 3060, 2922, 1627, 1590, 1566, 1438, 1448, 1417, 1318, 1236, 1149, 799, 743 cm⁻¹.

1-Methyl-9H-pyrido[3,4-b]indole 2j:



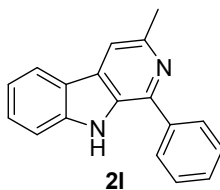
Pale yellow solid, mp 221-222 °C. ¹H NMR (CDCl₃, 400 MHz) δ 9.66 (s, 1H, NH in indole), 8.38 (d, *J* = 5.4 Hz, 1H), 8.12 (d, *J* = 7.9 Hz, 1H), 7.84 (d, *J* = 5.4 Hz, 1H), 7.46-7.54 (m, 2H), 7.25-7.30 (m, 1H), 2.83 (s, 3H). ¹³C NMR (CDCl₃, 100 MHz) δ 141.82, 140.24, 138.51, 134.68, 128.34, 128.23, 122.02, 121.84, 120.07, 112.96, 111.62, 20.31. HRMS (ESI) *m/z* calcd for C₁₂H₁₁N₂ [M+H]⁺: 183.0922, found: 183.0926. IR (KBr film) 3128, 3063, 2954, 2854, 1625, 1565, 1507, 1255, 1237, 741 cm⁻¹.

1-Ethyl-9H-pyrido[3,4-b]indole 2k:



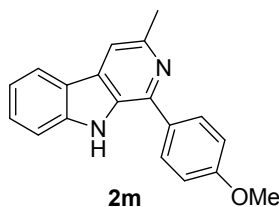
White solid, mp 195-196 °C. ¹H NMR (DMSO-*d*₆, 400 MHz) δ 11.62 (s, 1H, NH in indole), 8.27 (d, *J* = 5.3 Hz, 1H), 8.16 (d, *J* = 7.9 Hz, 1H), 7.90 (d, *J* = 5.3 Hz, 1H), 7.62 (d, *J* = 8.2 Hz, 1H), 7.53 (dd, *J*₁ = 8.2 Hz, *J*₂ = 8.0 Hz, 1H), 7.21 (dd, *J*₁ = 7.9 Hz, *J*₂ = 8.0 Hz, 1H), 3.17 (q, *J* = 7.5 Hz, 2H), 1.39 (t, *J* = 7.5 Hz, 3H). ¹³C NMR (DMSO-*d*₆, 100 MHz) δ 147.36, 140.86, 138.00, 134.26, 128.20, 127.59, 122.03, 121.61, 119.57, 113.05, 112.38, 27.14, 13.13. HRMS (ESI) *m/z* calcd for C₁₃H₁₃N₂ [M+H]⁺: 197.1079, found: 197.1082. IR (KBr film) 3171, 2926, 2855, 1626, 1458, 1430, 1401, 1324, 1240, 1155, 745 cm⁻¹.

3-Methyl-1-phenyl-9H-pyrido[3,4-*b*]indole 2l:



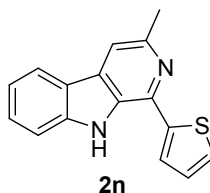
Pale yellow solid, mp. 128-129 °C. ¹H NMR (CDCl₃, 400 MHz) δ 8.51 (s, 1H, *NH* in indole), 8.08 (d, *J* = 7.9 Hz, 1H), 7.88 (d, *J* = 7.2 Hz, 2H), 7.74 (s, 1H), 7.45-7.55 (m, 3H), 7.36-7.42 (m, 2H), 7.23 (t, *J* = 7.2 Hz, 1H), 2.76 (s, 3H). ¹³C NMR (CDCl₃, 100 MHz) δ 147.85, 141.98, 140.83, 138.60, 131.91, 130.87, 129.15 (2C), 128.66, 128.38, 128.23 (2C), 121.76, 119.96, 112.71, 111.49, 24.39. HRMS (ESI) *m/z* calcd for C₁₈H₁₅N₂ [M+H]⁺: 259.1235, found: 259.1242. IR (KBr film) 3132, 3064, 2916, 1625, 1561, 1496, 1452, 1440, 1320, 1241, 742, 703 cm⁻¹.

1-(4-Methoxyphenyl)-3-methyl-9H-pyrido[3,4-*b*]indole 2m:



Pale yellow solid, mp. 131-132 °C. ¹H NMR (CDCl₃, 400 MHz) δ 8.82 (s, 1H, *NH* in indole), 8.05 (d, *J* = 7.9 Hz, 1H), 7.76 (d, *J* = 8.2 Hz, 2H), 7.68 (s, 1H), 7.46 (dd, *J*₁ = 7.9 Hz, *J*₂ = 8.0 Hz, 1H), 7.38 (d, *J* = 8.1 Hz, 1H), 7.22 (dd, *J*₁ = 8.0 Hz, *J*₂ = 8.1 Hz, 1H), 6.90 (d, *J* = 8.2 Hz, 2H), 3.72 (s, 3H), 2.73 (s, 3H). ¹³C NMR (CDCl₃, 100 MHz) δ 159.85, 147.56, 141.97, 140.96, 131.87, 131.15, 130.69, 129.46 (2C), 128.19, 121.80, 121.67, 119.78, 114.34 (2C), 112.21, 111.55, 55.29, 24.31. HRMS (EI) *m/z* calcd for C₁₉H₁₇N₂O [M+H]⁺: 289.1341, found: 289.1340. IR (KBr film) 3126, 3055, 2918, 1624, 1611, 1563, 1513, 1251, 1237, 744, 586 cm⁻¹.

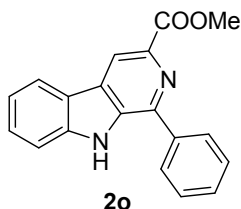
3-Methyl-1-(thiophen-2-yl)-9H-pyrido[3,4-*b*]indole 2n:



Pale yellow solid, mp. 161-162 °C. ¹H NMR (CDCl₃, 400 MHz) δ 8.54 (s, 1H, *NH* in indole), 8.08 (d, *J* = 7.9 Hz, 1H), 7.72 (s, 1H), 7.71 (d, *J* = 4.8 Hz, 1H), 7.45-7.55 (m, 3H), 7.29 (d, *J* = 7.8 Hz, 1H), 7.20 (dd, *J*₁ = 4.8 Hz, *J*₂ = 3.7 Hz, 1H), 2.75 (s, 3H). ¹³C NMR (CDCl₃, 100 MHz) δ 147.89, 142.84, 140.94, 135.89, 131.44, 130.76, 128.52, 127.98, 126.95, 125.11, 121.79, 121.71, 120.27, 112.82, 111.69, 24.29.

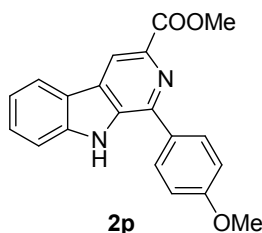
HRMS (ESI) m/z calcd for $C_{16}H_{13}N_2S$ $[M+H]^+$: 265.0799, found: 265.0796. IR (KBr film) 3344, 3062, 2911, 1624, 1558, 1493, 1438, 1323, 1236, 740, 711 cm^{-1} .

Methyl 1-phenyl-9H-pyrido[3,4-*b*]indole-3-carboxylate 2o:



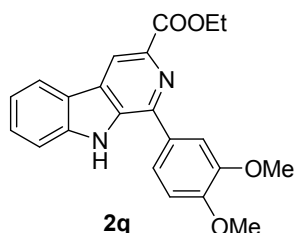
White solid, mp 256-257 °C. 1H NMR (DMSO- d_6 , 400 MHz) δ 11.92 (s, 1H, *NH* in indole), 8.92 (s, 1H), 8.42 (d, $J = 8.0$ Hz, 1H), 8.05 (d, $J = 7.9$ Hz, 2H), 7.70 (d, $J = 8.0$ Hz, 1H), 7.52-7.66 (m, 4H), 7.33 (dd, $J_1 = 8.0$ Hz, $J_2 = 7.9$ Hz, 1H), 3.94 (s, 3H). ^{13}C NMR (DMSO- d_6 , 100 MHz) δ 166.57, 142.56, 142.21, 138.08, 136.98, 135.29, 129.65, 129.38, 129.20 (2C), 129.11 (2C), 129.03, 122.39, 121.64, 120.75, 117.13, 113.35, 52.48. HRMS (ESI) m/z calcd for $C_{19}H_{15}N_2O_2$ $[M+H]^+$: 303.1134, found: 303.1136. IR (KBr film) 3319, 2952, 1723, 1626, 1352, 1253, 1217, 1103, 1049, 740 cm^{-1} .

Methyl 1-(4-methoxyphenyl)-9H-pyrido[3,4-*b*]indole-3-carboxylate 2p:



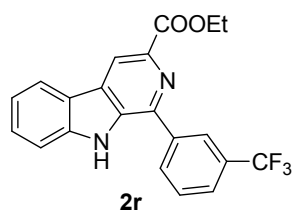
White solid, mp 229-230 °C. 1H NMR (DMSO- d_6 , 400 MHz) δ 11.89 (s, 1H, *NH* in indole), 8.88 (s, 1H), 8.41 (d, $J = 7.6$ Hz, 1H), 7.99 (d, $J = 8.6$ Hz, 2H), 7.70 (d, $J = 7.9$ Hz, 1H), 7.60 (dd, $J_1 = 7.9$ Hz, $J_2 = 7.8$ Hz, 1H), 7.32 (dd, $J_1 = 7.6$ Hz, $J_2 = 7.8$ Hz, 1H), 7.20 (d, $J = 8.6$ Hz, 2H), 3.93 (s, 3H), 3.88 (s, 3H). ^{13}C NMR (DMSO- d_6 , 100 MHz) δ 166.59, 160.41, 142.46, 141.90, 137.07, 134.84, 130.46, 130.43 (2C), 129.44, 128.99, 122.38, 121.66, 120.79, 116.64, 114.63 (2C), 113.24, 55.80, 52.46. HRMS (ESI) m/z calcd for $C_{20}H_{17}N_2O_3$ $[M+H]^+$: 333.1239, found: 333.1241. IR (KBr film) 3254, 2951, 2837, 1714, 1611, 1514, 1434, 1353, 1251, 1177, 1116, 1032, 746 cm^{-1} .

Ethyl 1-(3,4-dimethoxyphenyl)-9H-pyrido[3,4-*b*]indole-3-carboxylate 2q:



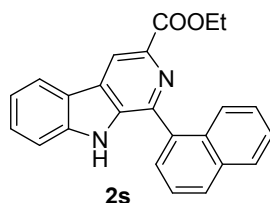
White solid, mp 217-218 °C. ¹H NMR (DMSO-*d*₆, 400 MHz) δ 11.89 (s, 1H, *NH* in indole), 8.87 (s, 1H), 8.41 (d, *J* = 7.9 Hz, 1H), 7.70 (d, *J* = 8.1 Hz, 1H), 7.54-7.64 (m, 3H), 7.32 (dd, *J*₁ = 7.9 Hz, *J*₂ = 8.0 Hz, 1H), 7.21 (d, *J* = 7.9 Hz, 1H), 4.41 (q, *J* = 7.1 Hz, 2H), 3.91 (s, 3H), 3.89 (s, 3H), 1.39 (t, *J* = 7.1 Hz, 3H). ¹³C NMR (DMSO-*d*₆, 100 MHz) δ 166.04, 150.10, 149.30, 142.73, 141.89, 137.28, 134.94, 130.69, 129.35, 128.95, 122.37, 121.70, 121.71, 120.76, 116.59, 113.24, 112.55, 112.31, 61.06, 56.16, 55.95, 14.83. HRMS (ESI) *m/z* calcd for C₂₂H₂₁N₂O₄ [M+H]⁺: 377.1501, found: 377.1502. IR (KBr film) 3423, 2937, 1709, 1626, 1516, 1371, 1348, 1257, 1143, 1025, 746 cm⁻¹.

Ethyl 1-(3-(trifluoromethyl)phenyl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate 2r:



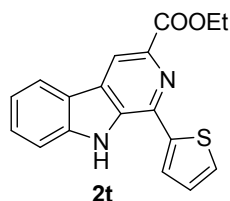
White solid, mp 209-210 °C. ¹H NMR (DMSO-*d*₆, 400 MHz) δ 12.06 (s, 1H, *NH* in indole), 8.96 (s, 1H), 8.45 (d, *J* = 7.9 Hz, 1H), 8.32 (d, *J* = 7.6 Hz, 1H), 8.27 (s, 1H), 7.86-7.94 (m, 2H), 7.70 (d, *J* = 8.0 Hz, 1H), 7.63 (dd, *J*₁ = 7.8 Hz, *J*₂ = 7.9 Hz, 1H), 7.35 (dd, *J*₁ = 7.9 Hz, *J*₂ = 8.0 Hz, 1H), 4.42 (q, *J* = 7.1 Hz, 2H), 1.39 (t, *J* = 7.1 Hz, 3H). ¹³C NMR (DMSO-*d*₆, 100 MHz) δ 165.77, 142.03, 140.77, 138.92, 137.47, 135.10, 132.99, 130.34, 130.07 (q, ²*J*_{CF} = 31.0 Hz), 130.05, 129.31, 126.00 (q, ³*J*_{CF} = 4.1 Hz), 125.61 (q, ³*J*_{CF} = 4.1 Hz), 124.68 (q, ¹*J*_{CF} = 271.0 Hz), 122.51, 121.56, 120.97, 117.55, 113.17, 61.17, 14.78. HRMS (ESI) *m/z* calcd for C₂₁H₁₆F₃N₂O₂ [M+H]⁺: 385.1164, found: 385.1160. IR (KBr film) 3260, 3058, 2988, 2908, 1724, 1624, 1567, 1454, 1373, 1330, 1255, 1160, 1120, 1095, 742, 541 cm⁻¹.

Ethyl 1-(naphthalen-1-yl)-9*H*-pyrido[3,4-*b*]indole-3-carboxylate 2s:



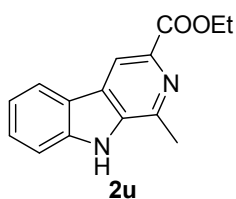
White solid, mp 275-276 °C. ¹H NMR (DMSO-*d*₆, 400 MHz) δ 11.63 (s, 1H, *NH* in indole), 9.03 (s, 1H), 8.47 (d, *J* = 7.9 Hz, 1H), 8.16 (d, *J* = 7.8 Hz, 1H), 8.10 (d, *J* = 8.0 Hz, 1H), 7.72-7.78 (m, 2H), 7.53-7.60 (m, 4H), 7.44 (dd, *J*₁ = 7.8 Hz, *J*₂ = 7.9 Hz, 1H), 7.32 (dd, *J*₁ = 7.9 Hz, *J*₂ = 8.0 Hz, 1H), 4.39 (q, *J* = 7.1 Hz, 2H), 1.35 (t, *J* = 7.1 Hz, 3H). ¹³C NMR (DMSO-*d*₆, 100 MHz) δ 166.12, 143.02, 141.89, 137.13, 136.84, 135.31, 134.02, 131.70, 129.50, 129.10, 128.95, 128.83, 128.16, 127.05, 126.62, 126.13, 125.80, 122.59, 121.67, 120.76, 117.44, 113.12, 61.12, 14.85. HRMS (ESI) *m/z* calcd for C₂₄H₁₉N₂O₂ [M+H]⁺: 367.1447, found: 367.1445. IR (KBr film) 3408, 3234, 2982, 1712, 1626, 1502, 1345, 1226, 752 cm⁻¹.

Ethyl 1-(thiophen-2-yl)-9H-pyrido[3,4-b]indole-3-carboxylate 2t:



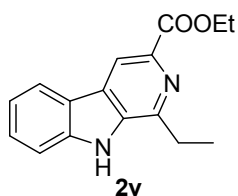
White solid, mp 162-163 °C. ¹H NMR (DMSO-*d*₆, 400 MHz) δ 11.90 (s, 1H, NH in indole), 8.85 (s, 1H), 8.42 (d, *J* = 7.9 Hz, 1H), 8.15 (d, *J* = 3.6 Hz, 1H), 7.75-7.81 (m, 2H), 7.63 (dd, *J*₁ = 7.9 Hz, *J*₂ = 8.0 Hz, 1H), 7.32-7.38 (m, 2H), 4.41 (q, *J* = 7.1 Hz, 2H), 1.40 (t, *J* = 7.1 Hz, 3H). ¹³C NMR (DMSO-*d*₆, 100 MHz) δ 165.55, 142.91, 142.01, 137.05, 136.80, 132.89, 130.20, 129.29, 129.28, 128.87, 127.09, 122.38, 121.54, 121.14, 116.81, 113.38, 61.18, 14.82. HRMS (ESI) *m/z* calcd for C₁₈H₁₅N₂O₂S [M+H]⁺: 323.0854, found: 323.0853. IR (KBr film) 3345, 3094, 2987, 2925, 1708, 1672, 1624, 1563, 1368, 1330, 1254, 1097, 1024, 740, 524 cm⁻¹.

Ethyl 1-methyl-9H-pyrido[3,4-b]indole-3-carboxylate 2u:



White solid, mp 217-218 °C. ¹H NMR (DMSO-*d*₆, 400 MHz) δ 12.04 (s, 1H, NH in indole), 8.77 (s, 1H), 8.36 (d, *J* = 7.9 Hz, 1H), 7.67 (d, *J* = 8.1 Hz, 1H), 7.60 (dd, *J*₁ = 7.9 Hz, *J*₂ = 8.0 Hz, 1H), 7.31 (dd, *J*₁ = 8.0 Hz, *J*₂ = 8.1 Hz, 1H), 4.38 (q, *J* = 7.1 Hz, 2H), 2.83 (s, 3H), 1.38 (t, *J* = 7.1 Hz, 3H). ¹³C NMR (DMSO-*d*₆, 100 MHz) δ 166.13, 142.59, 141.21, 136.68, 136.61, 128.75, 127.22, 122.48, 121.82, 120.54, 116.30, 112.75, 60.89, 20.83, 14.84. HRMS (ESI) *m/z* calcd for C₁₅H₁₅N₂O₂ [M+H]⁺: 255.1134, found: 255.1142. IR (KBr film) 3317, 3041, 2978, 1709, 1624, 1596, 1568, 1500, 1345, 1256, 1239, 741 cm⁻¹.

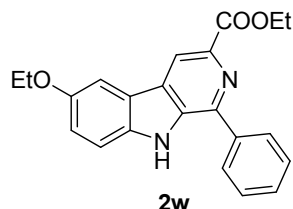
Ethyl 1-ethyl-9H-pyrido[3,4-b]indole-3-carboxylate 2v:



White solid, mp 210-211 °C. ¹H NMR (DMSO-*d*₆, 400 MHz) δ 12.02 (s, 1H, NH in indole), 8.76 (s, 1H), 8.35 (d, *J* = 7.9 Hz, 1H), 7.65 (d, *J* = 8.1 Hz, 1H), 7.58 (dd, *J*₁ = 7.9 Hz, *J*₂ = 8.0 Hz, 1H), 7.29 (dd, *J*₁ = 8.1 Hz, *J*₂ = 8.0 Hz, 1H), 4.37 (q, *J* = 7.1 Hz, 2H), 3.17 (q, *J* = 7.5 Hz, 2H), 1.37 (t, *J* = 7.1 Hz, 3H), 1.36 (t, *J* = 7.5 Hz, 3H). ¹³C NMR (DMSO-*d*₆, 100 MHz) δ 166.14, 147.40, 141.23, 136.80, 135.85, 128.75,

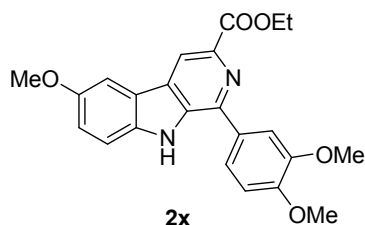
127.53, 122.42, 121.83, 120.51, 116.34, 112.77, 60.88, 27.27, 14.85, 13.19. HRMS (ESI) m/z calcd for $C_{16}H_{17}N_2O_2$ $[M+H]^+$: 269.1290, found: 269.1295. IR (KBr film) 3328, 2994, 2929, 1704, 1565, 1498, 1366, 1346, 1258, 1235, 1046, 749 cm^{-1} .

Ethyl 6-ethoxy-1-phenyl-9H-pyrido[3,4-b]indole-3-carboxylate 2w:



Pale yellow solid, m.p.: 228-229 °C. 1H NMR (400 MHz, $DMSO-d_6$) δ 11.75 (s, 1H, *NH* in indole), 8.92 (s, 1H), 8.06–7.96 (m, 3H), 7.64 (d, $J = 7.4$ Hz, 2H), 7.61–7.53 (m, 2H), 7.22 (d, $J = 8.9$ Hz, 1H), 4.41 (q, $J = 7.1$ Hz, 2H), 4.15 (q, $J = 6.9$ Hz, 2H), 1.40 (t, $J = 6.9$ Hz, 3H), 1.39 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (100 MHz, $DMSO-d_6$) δ 165.62, 153.35, 142.09, 137.69, 136.34, 136.21, 134.96, 129.00, 128.96, 128.81, 128.59, 121.65, 119.29, 116.94, 113.63, 104.29, 63.61, 60.61, 14.83, 14.45. IR (KBr film) 3448, 3227, 2980, 1712 (C=O), 1525, 1462, 1306, 1206, 1151, 1103, 1027, 725, 696 cm^{-1} . HRMS (ESI) m/z calcd for $C_{22}H_{21}N_2O_3$ $[M+H]^+$: 361.1552; found: 361.1549.

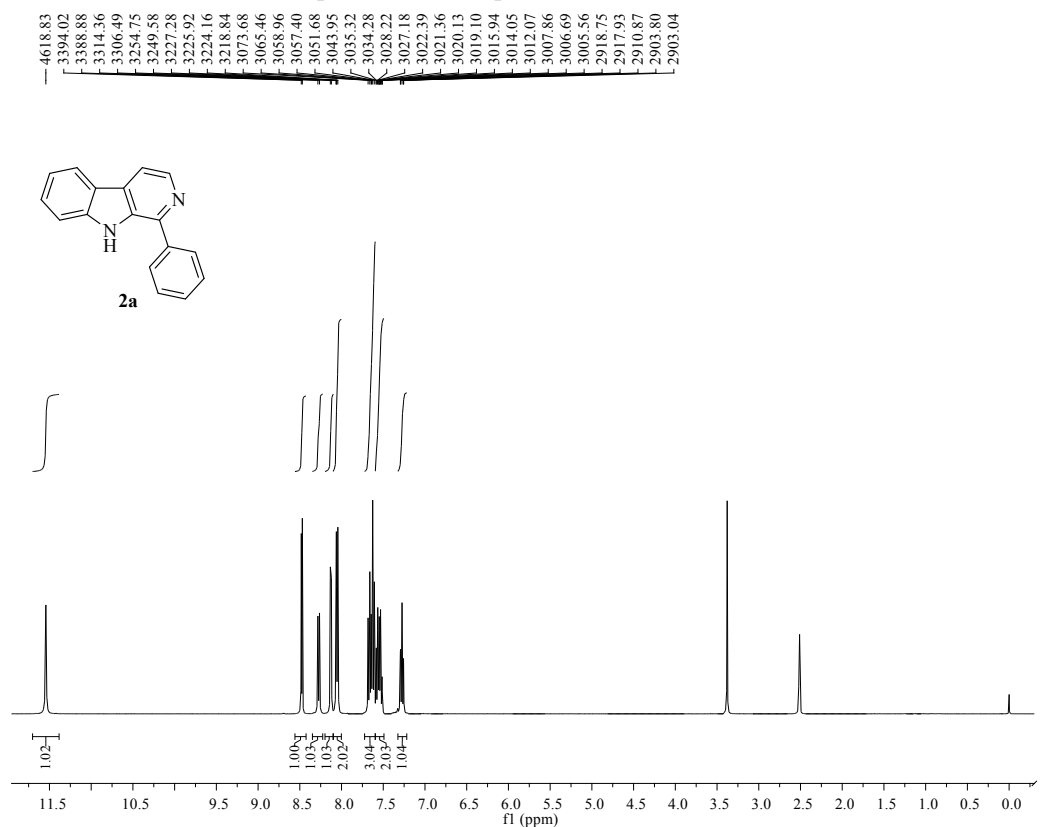
Ethyl 1-(3,4-dimethoxyphenyl)-6-methoxy-4,9-dihydro-3H-pyrido[3,4-b]indole-3-carboxylate 2x:



Pale yellow solid, m.p.: 207-208 °C. 1H NMR (400 MHz, $DMSO-d_6$) δ 11.72 (s, 1H, *NH* in indole), 8.88 (s, 1H), 8.00 (s, 1H), 7.69–7.42 (m, 3H), 7.23 (d, $J = 8.9$ Hz, 1H), 7.20 (d, $J = 8.9$ Hz, 1H), 4.40 (q, $J = 7.1$ Hz, 2H), 3.90 (s, 3H), 3.89 (s, 3H), 3.88 (s, 3H), 1.39 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (100 MHz, $DMSO-d_6$) δ 165.66, 154.14, 149.63, 148.84, 142.32, 136.22, 136.21, 134.90, 130.32, 128.75, 121.73, 121.20, 118.80, 116.48, 113.67, 112.02, 111.82, 103.50, 60.60, 55.71, 55.66, 55.51, 14.44. IR (KBr film) 3449, 3262, 2937, 1704 (C=O), 1514, 1495, 1463, 1303, 1266, 1240, 1219, 1104, 1033, 778, 759 cm^{-1} . HRMS (ESI) m/z calcd for $C_{23}H_{23}N_2O_5$ $[M+H]^+$: 407.1607; found: 407.1609.

2. ¹H and ¹³C NMR Spectra of All Compounds:

¹H NMR (DMSO-*d*₆, 400 MHz) spectrum of compound **2a**:



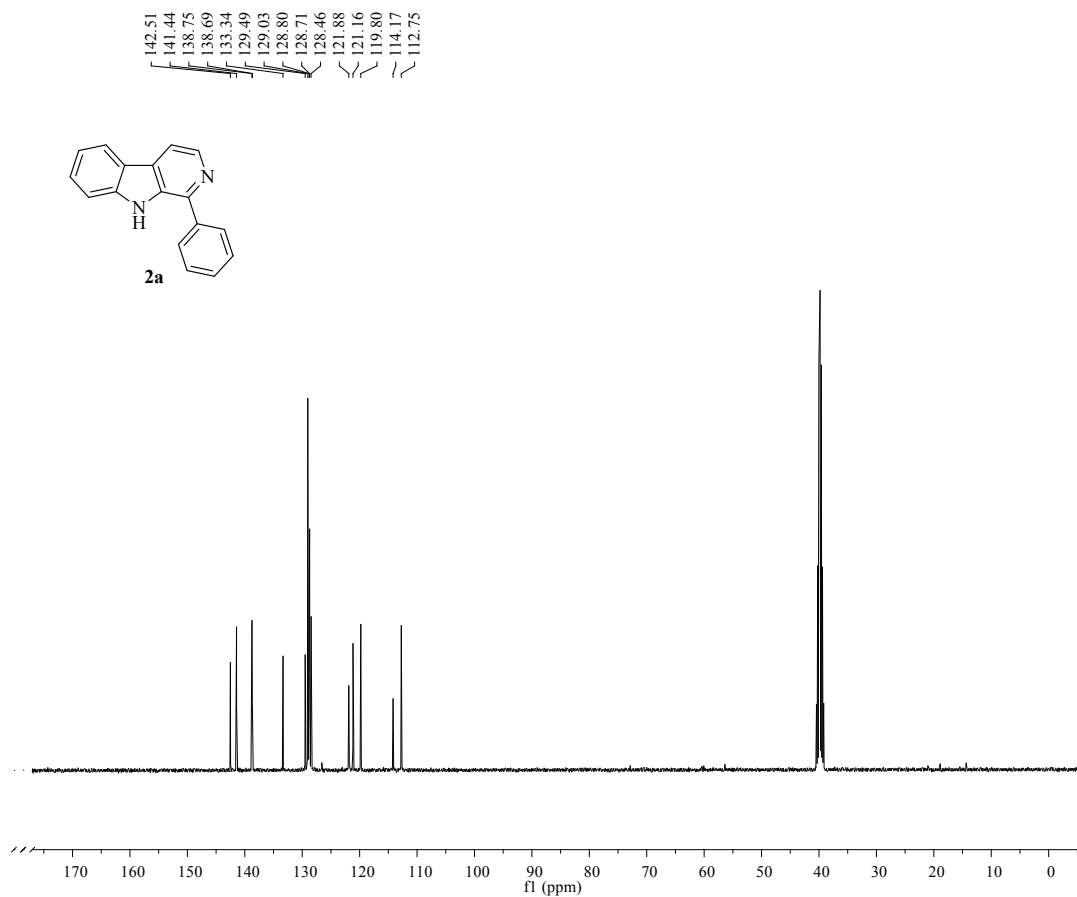
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PROCNO   1
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Time     11.01
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PROBHD   5 mm PABBO BB-
PULPROG  zg30
TD        65536
SOLVENT  DMSO
NS        16
DS         0
SWH       8223.685 Hz
FIDRES   0.125483 Hz
AQ        3.9846387 sec
RG         128
DW        60.800 usec
DE         6.50 usec
TE        295.0 K
D1        1.00000000 sec
TD0       1
    
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===== CHANNEL f1 =====
NUC1      1H
P1        14.90 usec
PL1       -1.00 dB
PL1W      13.37643433 W
SFO1      400.1324710 MHz
SI         32768
SF        400.1299987 MHz
WDW       EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
    
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¹³C NMR (DMSO-*d*₆, 100 MHz) spectrum of compound **2a**:



```

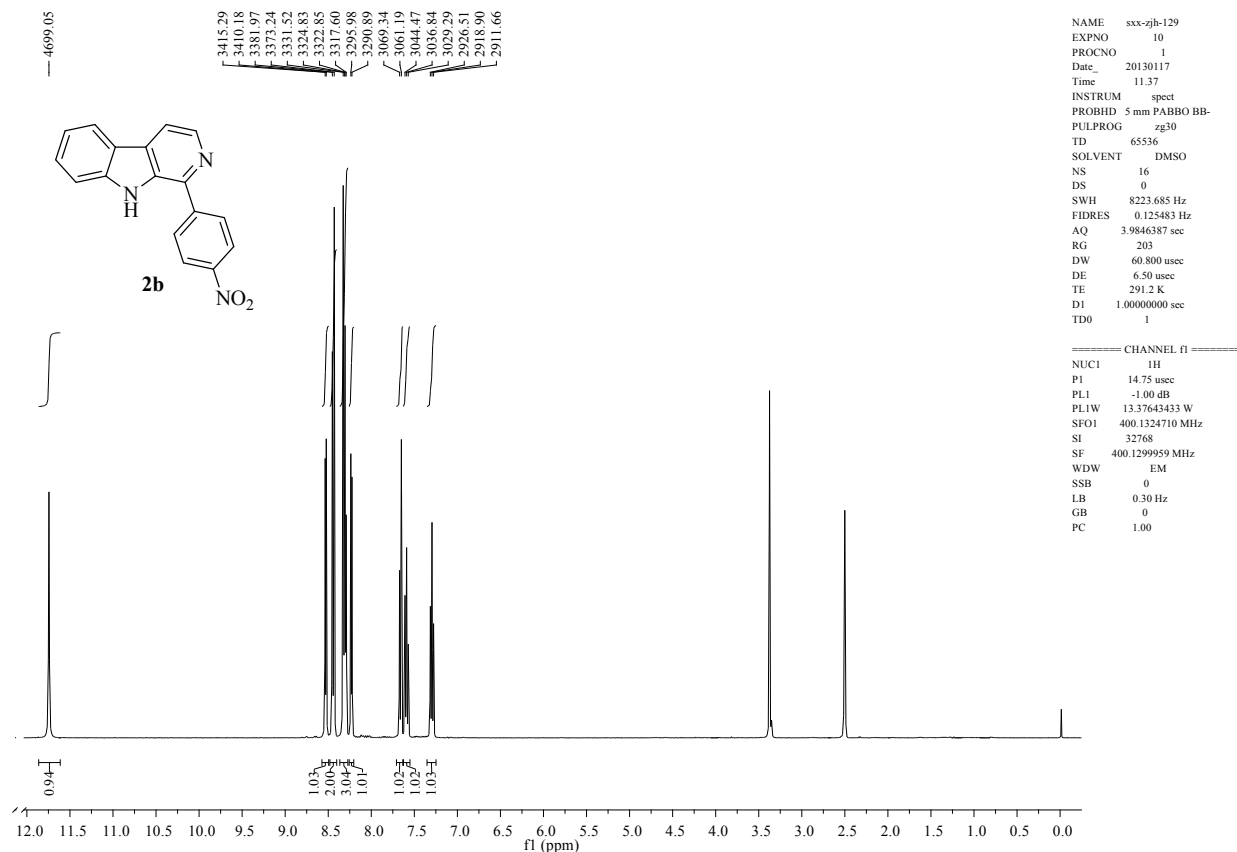
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PROCNO   1
Date_    20120520
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PULPROG  zgpg30
TD        65536
SOLVENT  DMSO
NS        400
DS         0
SWH       24038.461 Hz
FIDRES   0.366798 Hz
AQ        1.3631988 sec
RG         203
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DE         6.50 usec
TE        295.0 K
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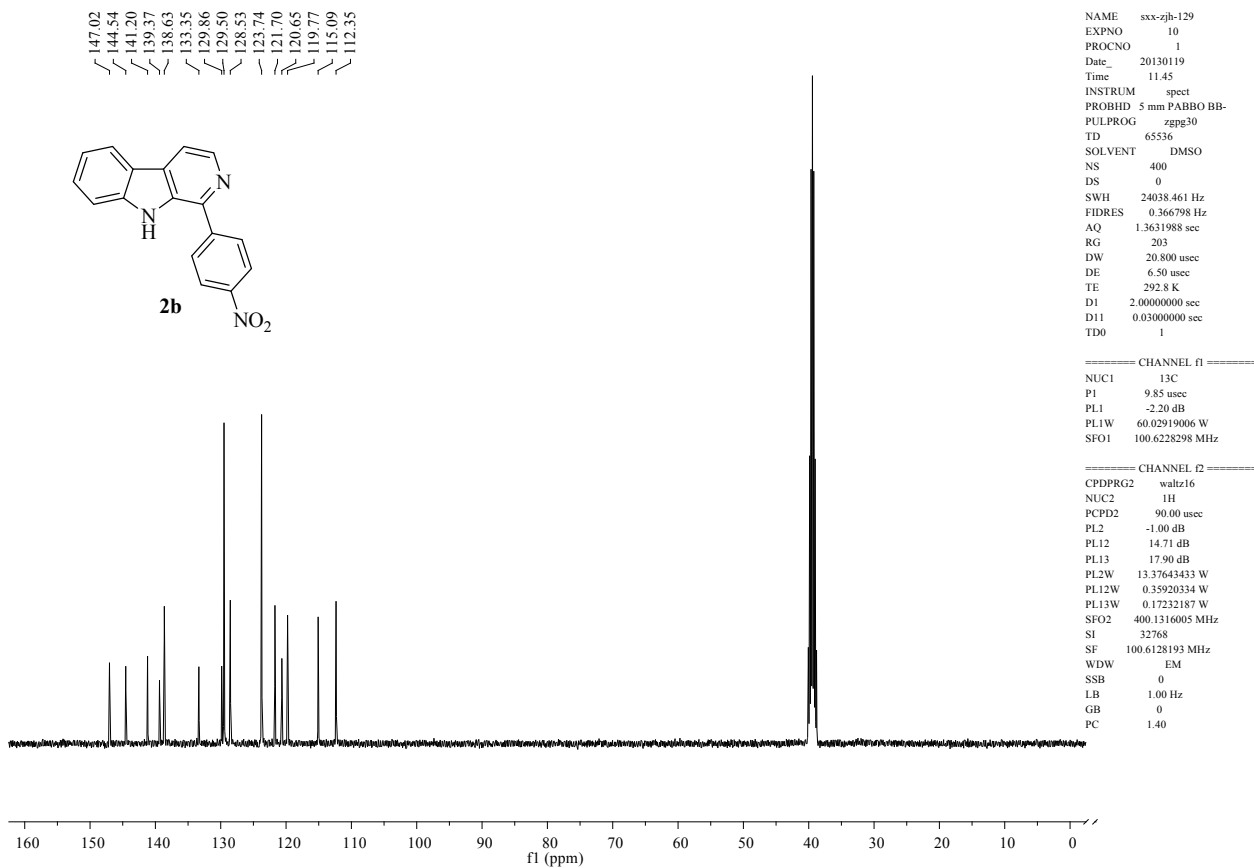
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===== CHANNEL f2 =====
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NUC2      1H
PCPD2     90.00 usec
PL2       -1.00 dB
PL12      14.90 dB
PL13      17.90 dB
PL2W      13.37643433 W
PL12W     0.34382734 W
PL13W     0.17232187 W
SFO2      400.1316005 MHz
SI         32768
SF        100.6127690 MHz
WDW       EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.00
    
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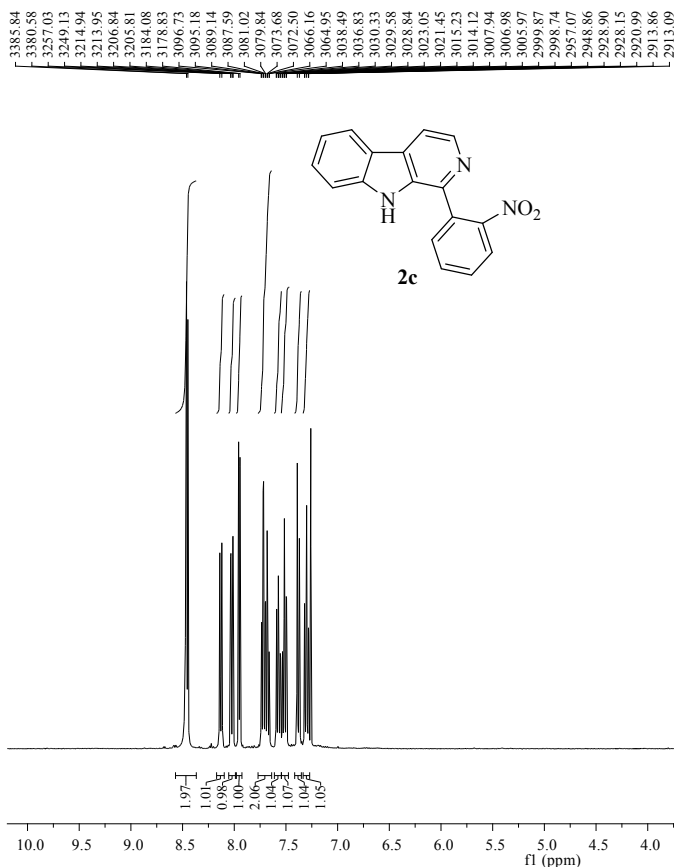
¹H NMR (DMSO-*d*₆, 400 MHz) spectrum of compound **2b**:



¹³C NMR (DMSO-*d*₆, 100 MHz) spectrum of compound **2b**:



¹H NMR (CDCl₃, 400 MHz) spectrum of compound **2c**:



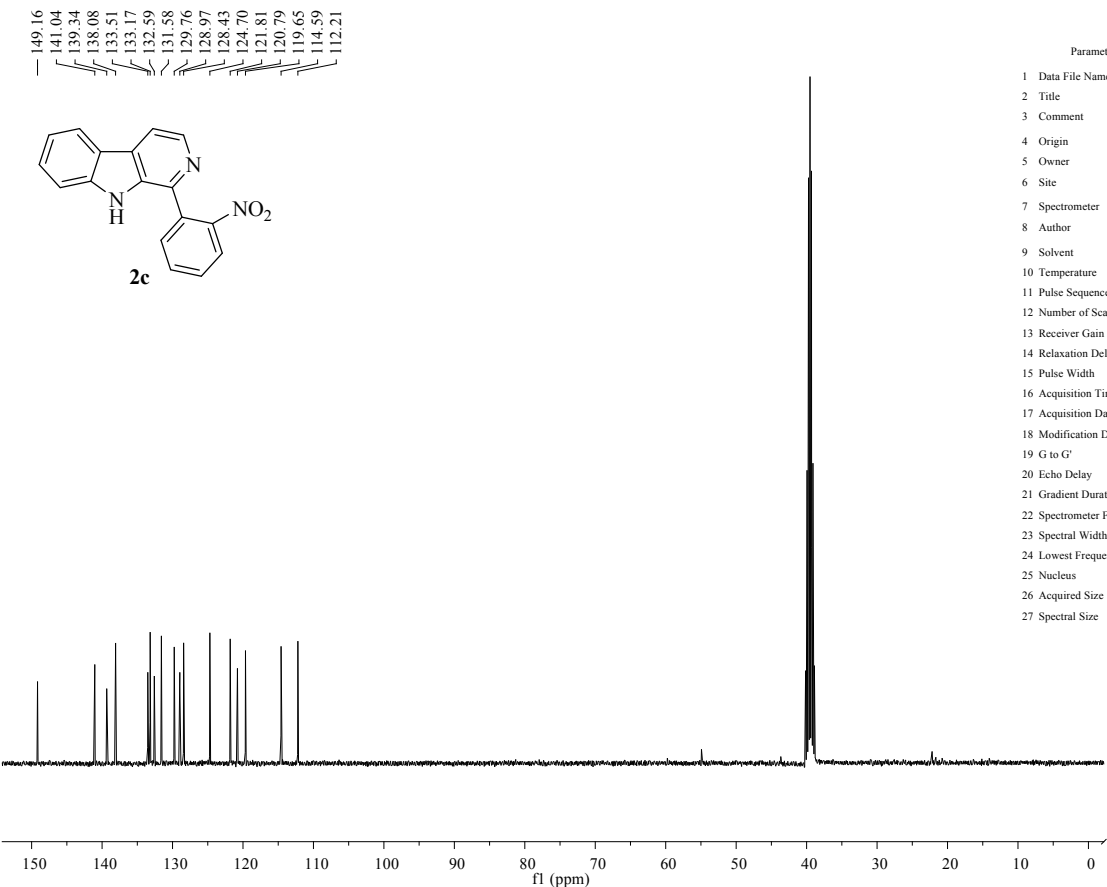
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PROCNO 1
Date_ 20130419
Time 7.47
INSTRUM spect
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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 406
DW 60.800 usec
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TD0 1
  
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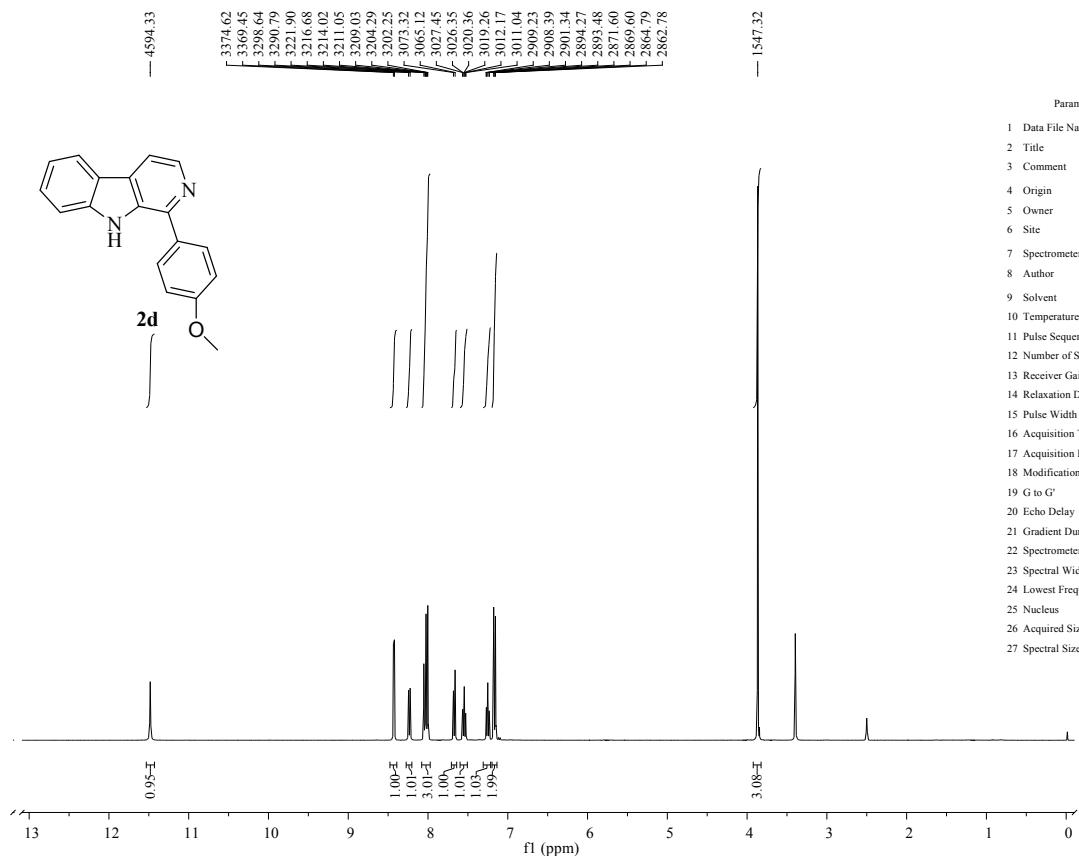
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SI 32768
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LB 0.30 Hz
GB 0
PC 1.00
  
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¹³C NMR (DMSO-*d*₆, 100 MHz) spectrum of compound **2c**:



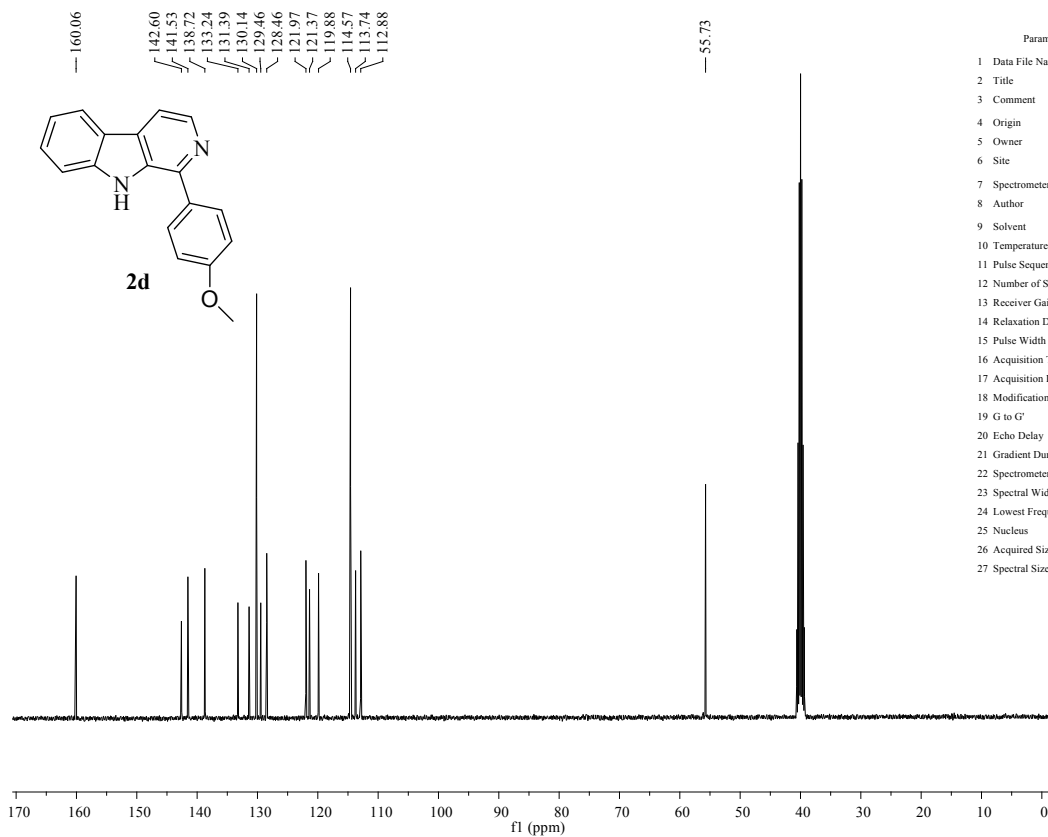
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1 Data File Name	SXX-ZJH-135
2 Title	SXX-ZJH-135
3 Comment	
4 Origin	Bruker BioSpin GmbH
5 Owner	nmr
6 Site	
7 Spectrometer	spect
8 Author	
9 Solvent	DMSO
10 Temperature	296.9
11 Pulse Sequence	zgpg30
12 Number of Scans	512
13 Receiver Gain	362
14 Relaxation Delay	2.0000
15 Pulse Width	9.4000
16 Acquisition Time	1.3631
17 Acquisition Date	2013-04-23T21:05:59
18 Modification Date	2013-04-23T21:06:00
19 G to G'	1.00000000
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21 Gradient Duration	0.0000
22 Spectrometer Frequency	100.62
23 Spectral Width	24038.5
24 Lowest Frequency	-2004.3
25 Nucleus	13C
26 Acquired Size	32768
27 Spectral Size	65536

¹H NMR (DMSO-*d*₆, 400 MHz) spectrum of compound **2d**:



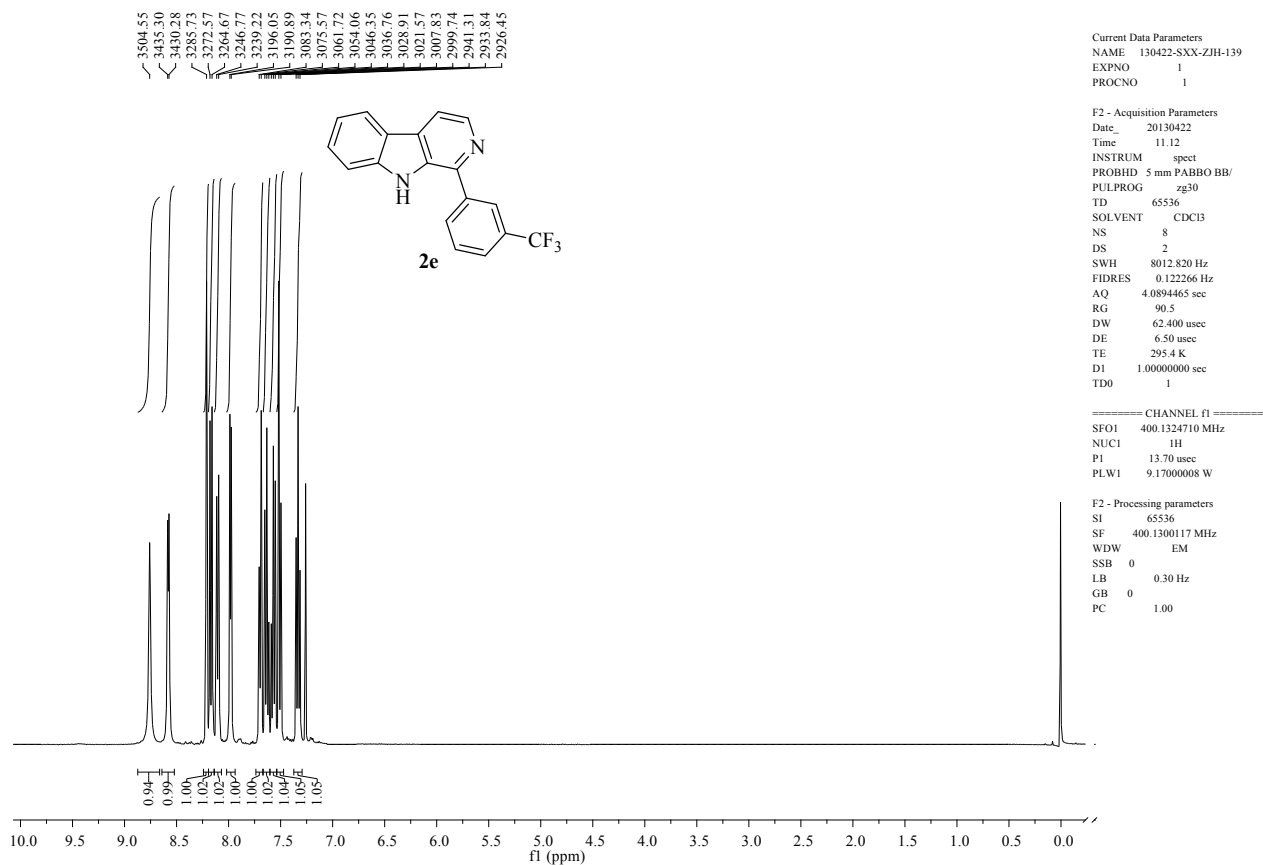
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3 Comment	
4 Origin	Bruker BioSpin GmbH
5 Owner	nmr
6 Site	
7 Spectrometer	spect
8 Author	
9 Solvent	DMSO
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12 Number of Scans	16
13 Receiver Gain	161
14 Relaxation Delay	1.0000
15 Pulse Width	13.3000
16 Acquisition Time	3.9846
17 Acquisition Date	2013-05-01T23:01:42
18 Modification Date	2013-05-01T23:01:00
19 G to G'	1.00000000
20 Echo Delay	0.0000
21 Gradient Duration	0.0000
22 Spectrometer Frequency	400.13
23 Spectral Width	8223.7
24 Lowest Frequency	-1642.9
25 Nucleus	1H
26 Acquired Size	32768
27 Spectral Size	65536

¹³C NMR (DMSO-*d*₆, 100 MHz) spectrum of compound **2d**:

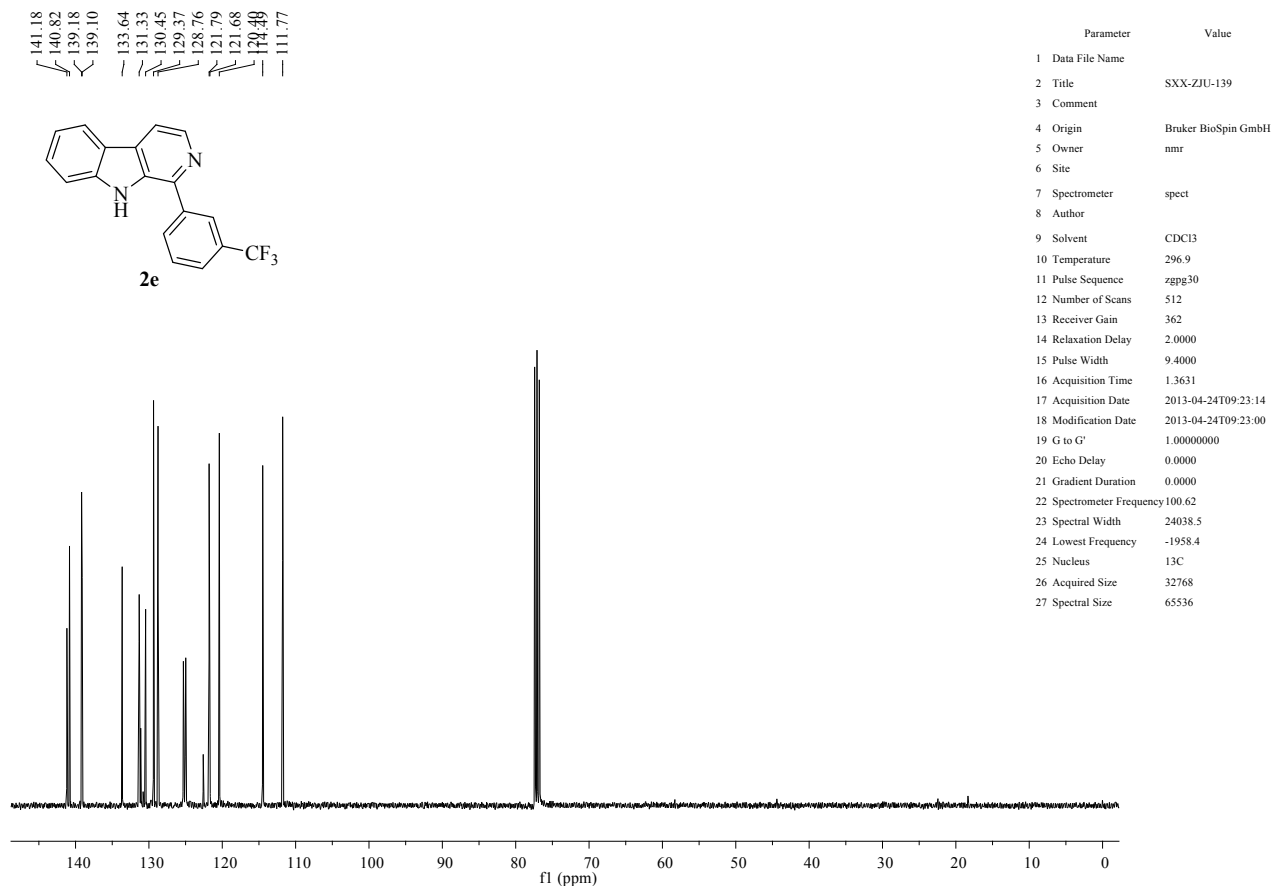


Parameter	Value
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2 Title	SXX-ZJH-157-C
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4 Origin	Bruker BioSpin GmbH
5 Owner	nmr
6 Site	
7 Spectrometer	spect
8 Author	
9 Solvent	DMSO
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17 Acquisition Date	2013-05-01T23:32:58
18 Modification Date	2013-05-01T23:32:00
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27 Spectral Size	65536

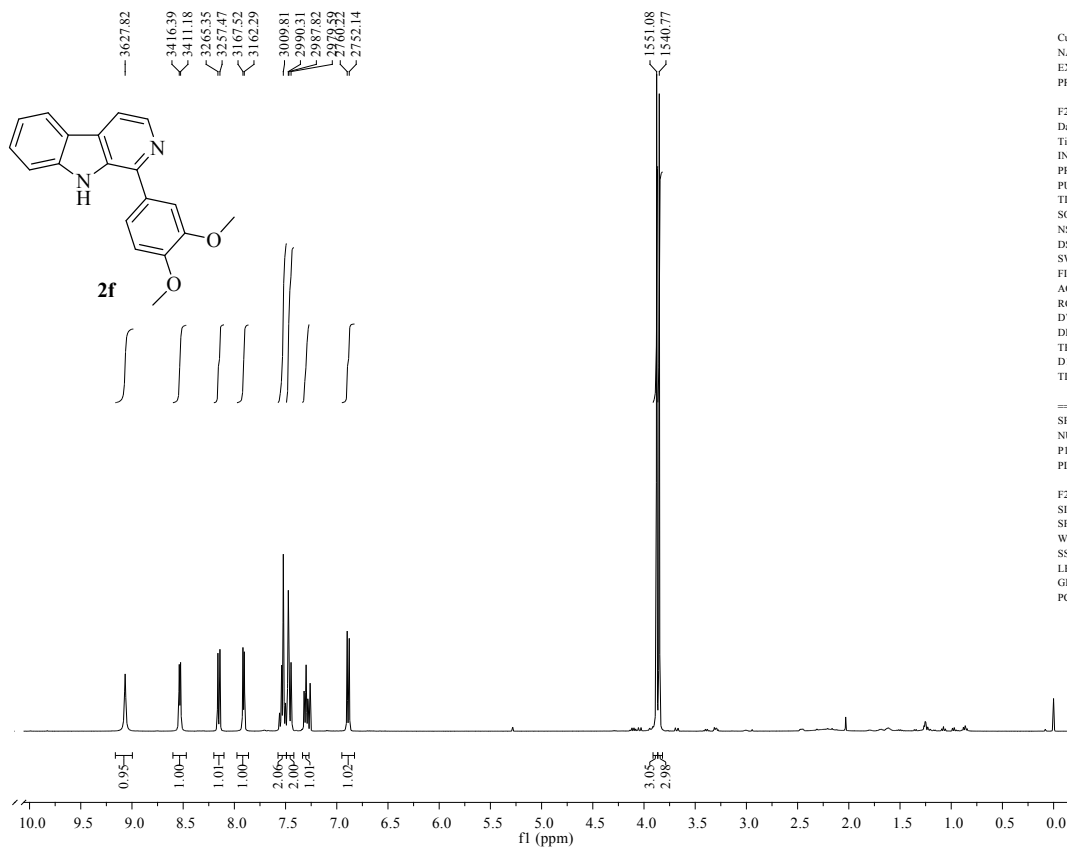
¹H NMR (CDCl₃, 400 MHz) spectrum of compound **2e**:



¹³C NMR (CDCl₃, 100 MHz) spectrum of compound **2e**:



¹H NMR (CDCl₃, 400 MHz) spectrum of compound **2f**:



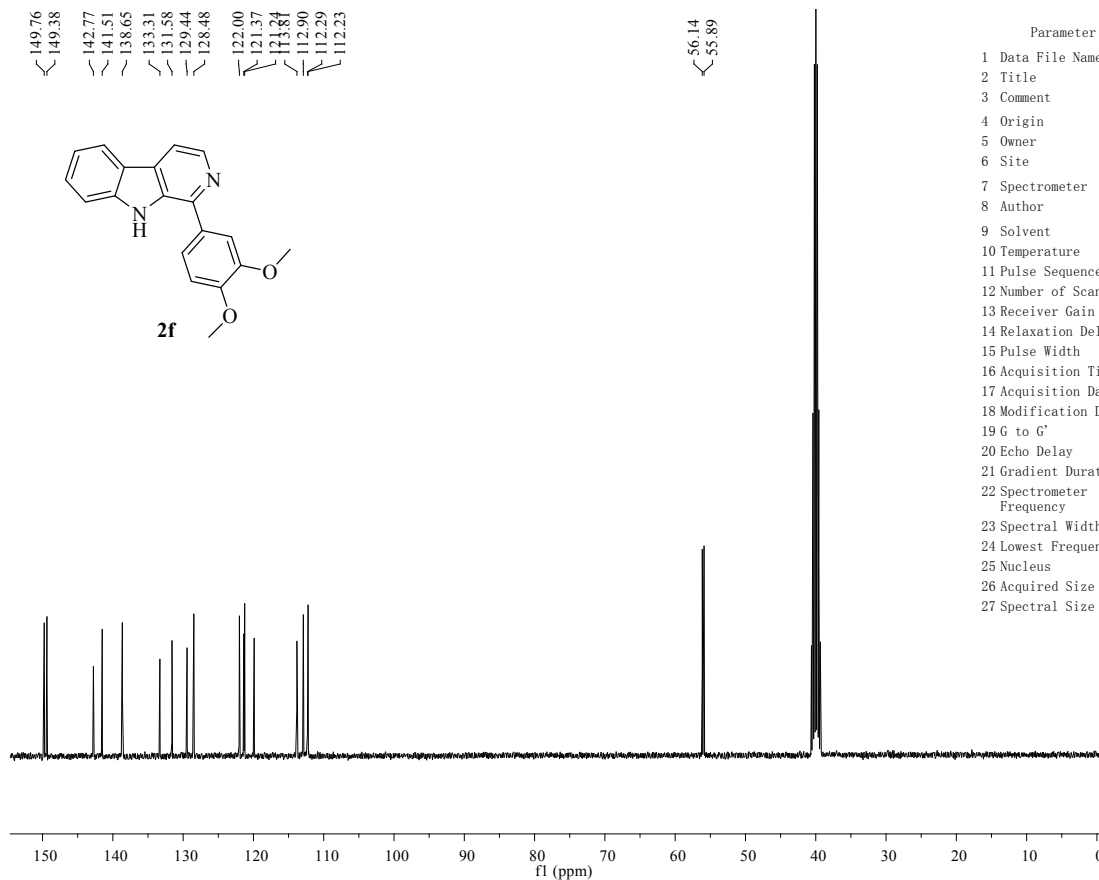
Current Data Parameters
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 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
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 Time 11.17
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 PULPROG zg30
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 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 64
 DW 62.400 usec
 DE 6.50 usec
 TE 296.0 K
 D1 1.0000000 sec
 TD0 1

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 PLW1 9.17000008 W

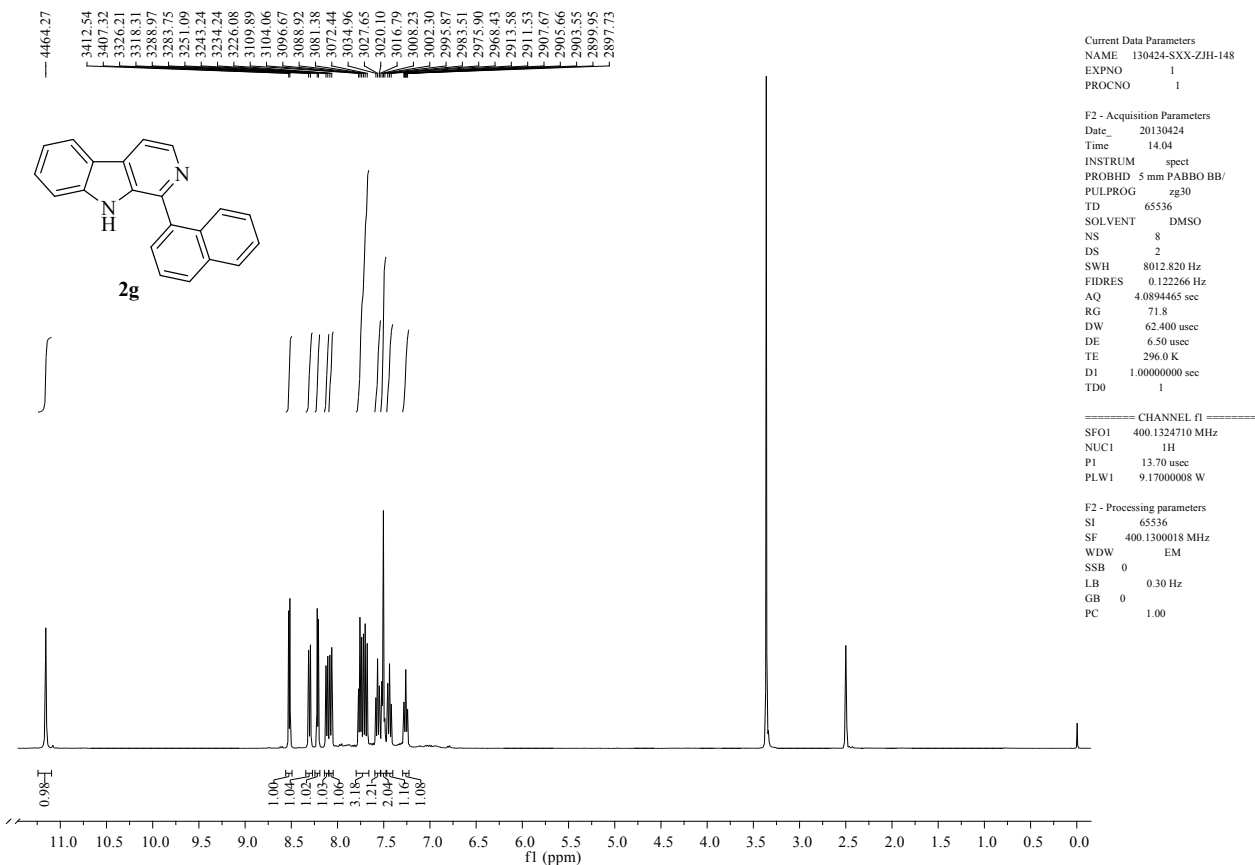
F2 - Processing parameters
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 PC 1.00

¹³C NMR (DMSO-*d*₆, 100 MHz) spectrum of compound **2f**:

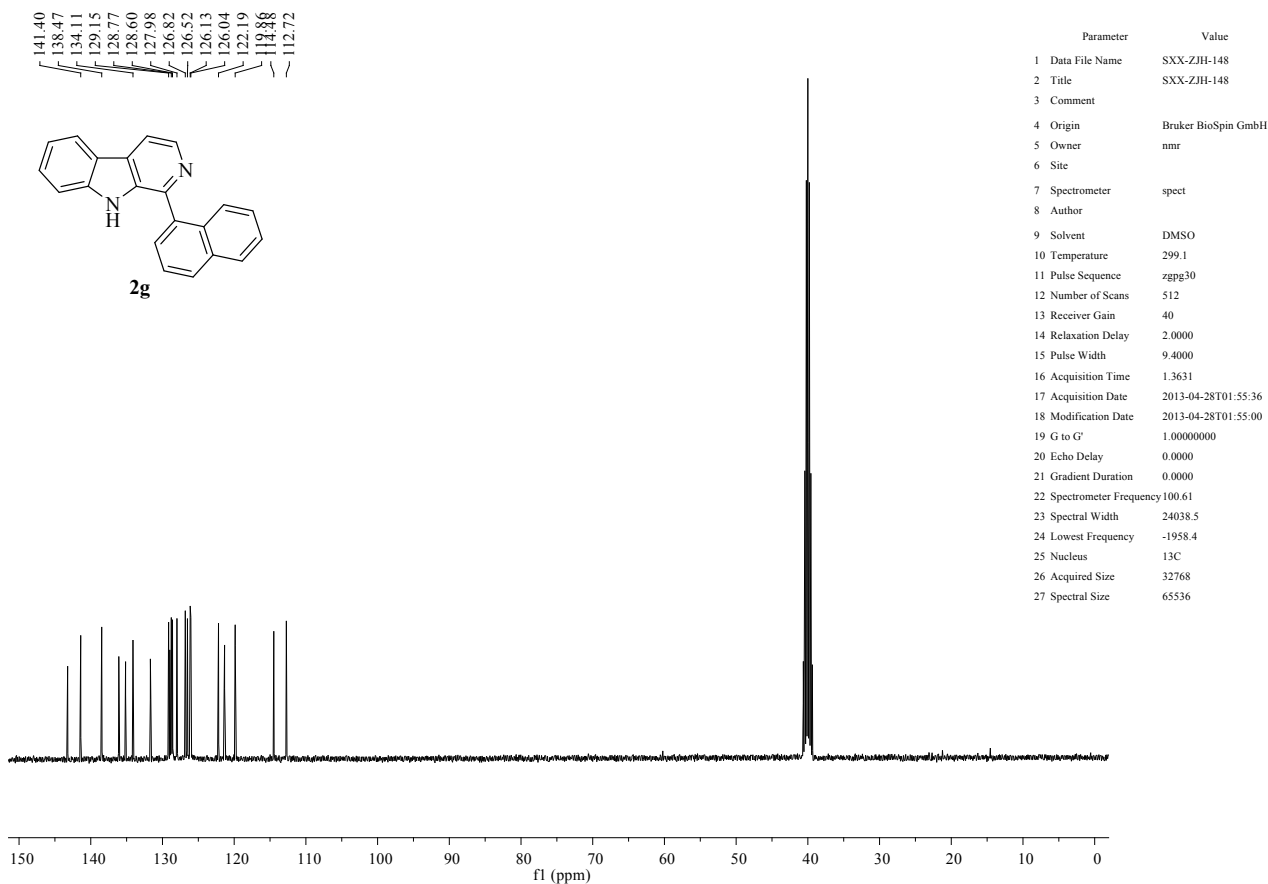


Parameter	Value
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4 Origin	Bruker BioSpin GmbH
5 Owner	nmr
6 Site	
7 Spectrometer	spect
8 Author	
9 Solvent	DMSO
10 Temperature	298.9
11 Pulse Sequence	zgpg30
12 Number of Scans	512
13 Receiver Gain	322
14 Relaxation Delay	2.0000
15 Pulse Width	9.4000
16 Acquisition Time	1.3631
17 Acquisition Date	2013-04-26T17:01:19
18 Modification Date	2013-04-26T17:01:00
19 G to G'	1.00000000
20 Echo Delay	0.0000
21 Gradient Duration	0.0000
22 Spectrometer	100.61
Frequency	
23 Spectral Width	24038.5
24 Lowest Frequency	-1958.4
25 Nucleus	13C
26 Acquired Size	32768
27 Spectral Size	65536

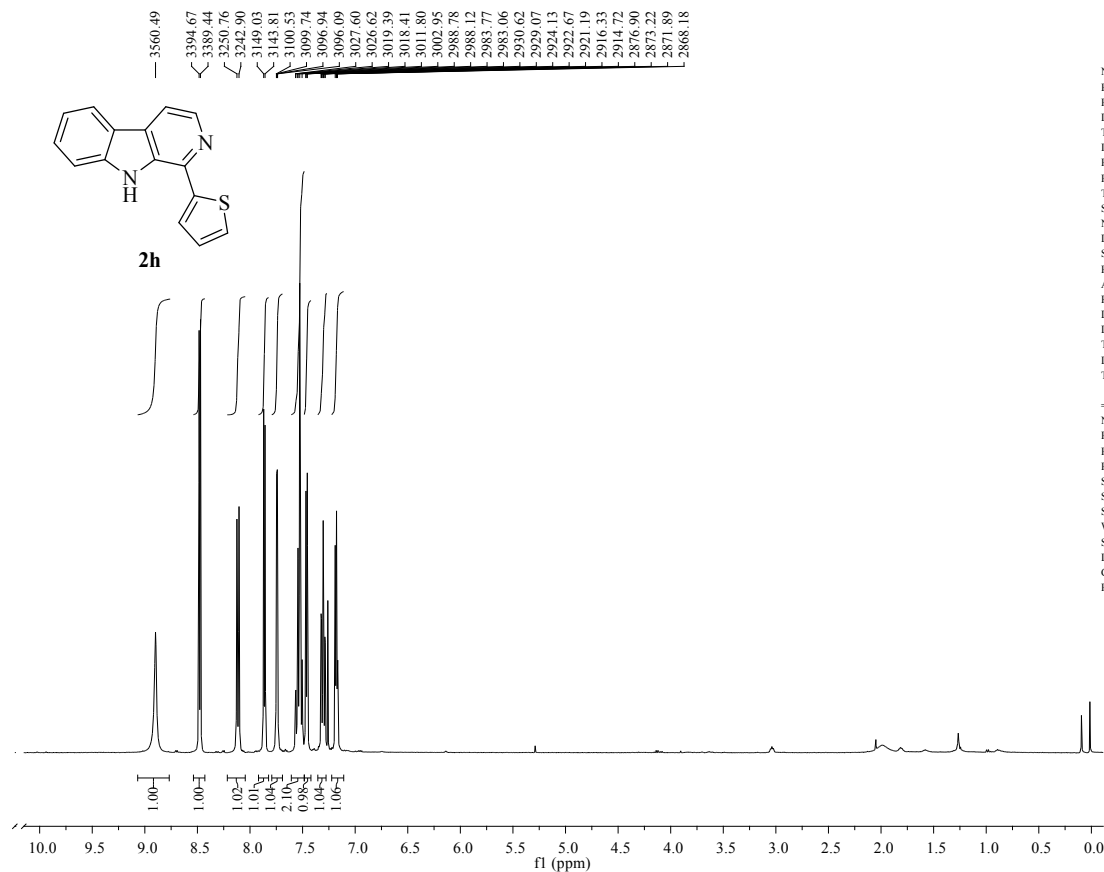
¹H NMR (DMSO-*d*₆, 400 MHz) spectrum of compound **2g**:



¹³C NMR (DMSO-*d*₆, 100 MHz) spectrum of compound **2g**:



¹H NMR (CDCl₃, 400 MHz) spectrum of compound **2h**:



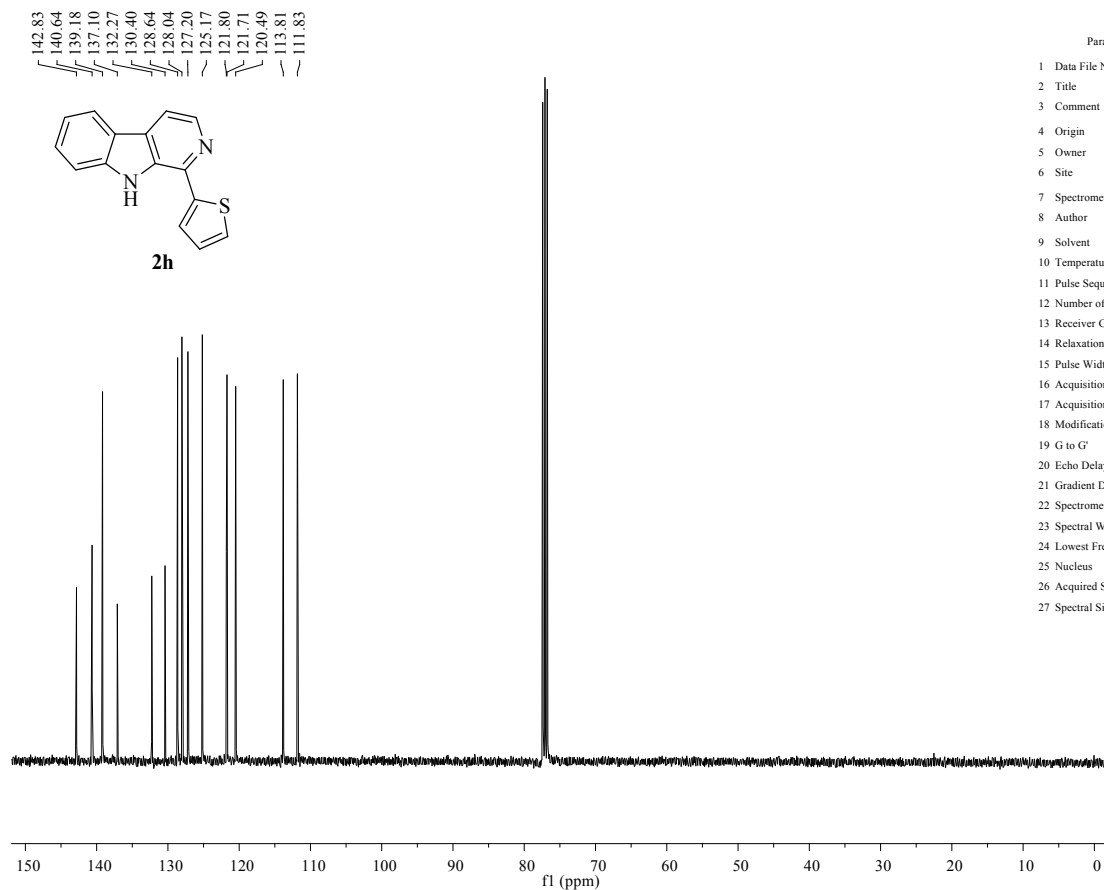
```

NAME SXX-ZJH-134
EXPNO 1
PROCNO 1
Date_ 20130419
Time 7.42
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 287
DW 60.800 usec
DE 6.50 usec
TE 298.3 K
D1 1.00000000 sec
TDO 1
    
```

```

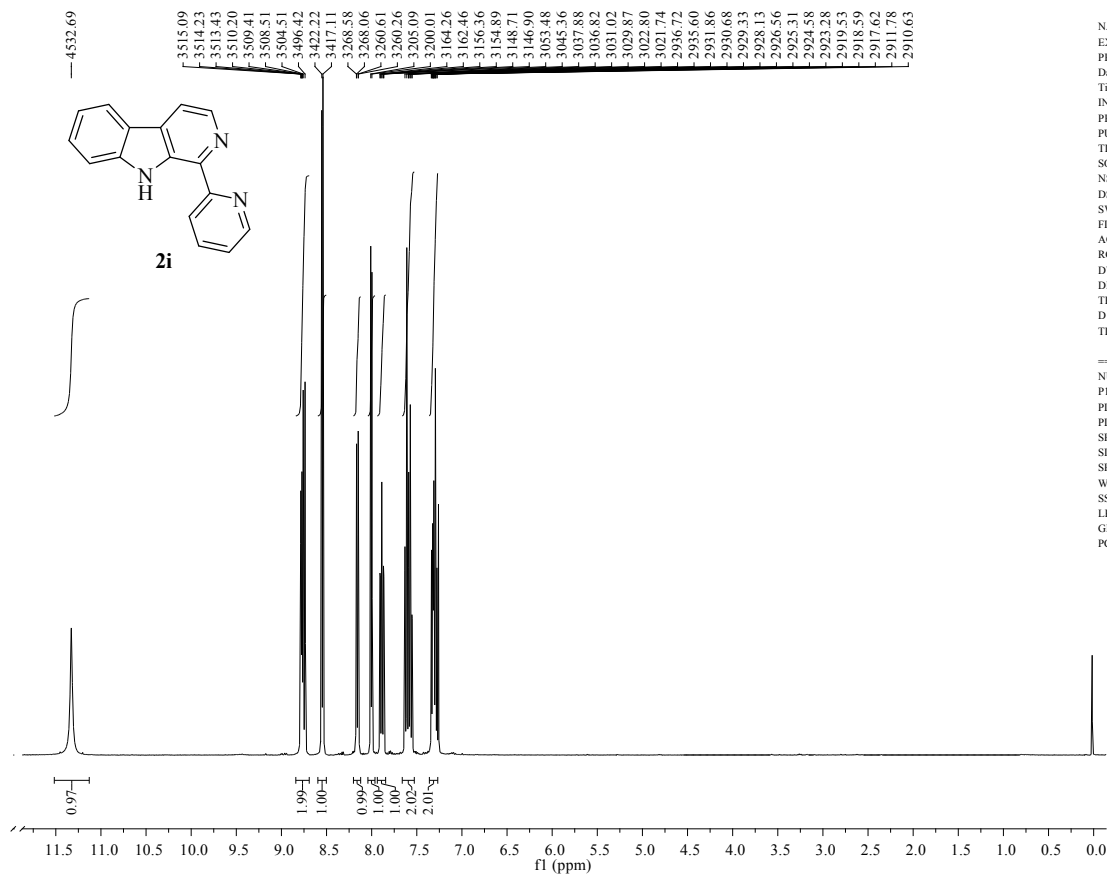
===== CHANNEL f1 =====
NUC1 1H
PI 13.30 usec
PL1 -2.00 dB
PL1W 15.28731537 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300151 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
    
```

¹³C NMR (CDCl₃, 100 MHz) spectrum of compound **2h**:



Parameter	Value
1 Data File Name	SXX-ZJH-134
2 Title	SXX-ZJH-134
3 Comment	
4 Origin	Bruker BioSpin GmbH
5 Owner	nmr
6 Site	
7 Spectrometer	spect
8 Author	
9 Solvent	CDCl3
10 Temperature	296.5
11 Pulse Sequence	zgpg30
12 Number of Scans	500
13 Receiver Gain	362
14 Relaxation Delay	2.0000
15 Pulse Width	9.4000
16 Acquisition Time	1.3631
17 Acquisition Date	2013-04-23T08:58:57
18 Modification Date	2013-04-23T08:59:00
19 G to G'	1.00000000
20 Echo Delay	0.0000
21 Gradient Duration	0.0000
22 Spectrometer Frequency	100.62
23 Spectral Width	24038.5
24 Lowest Frequency	-1958.4
25 Nucleus	13C
26 Acquired Size	32768
27 Spectral Size	65536

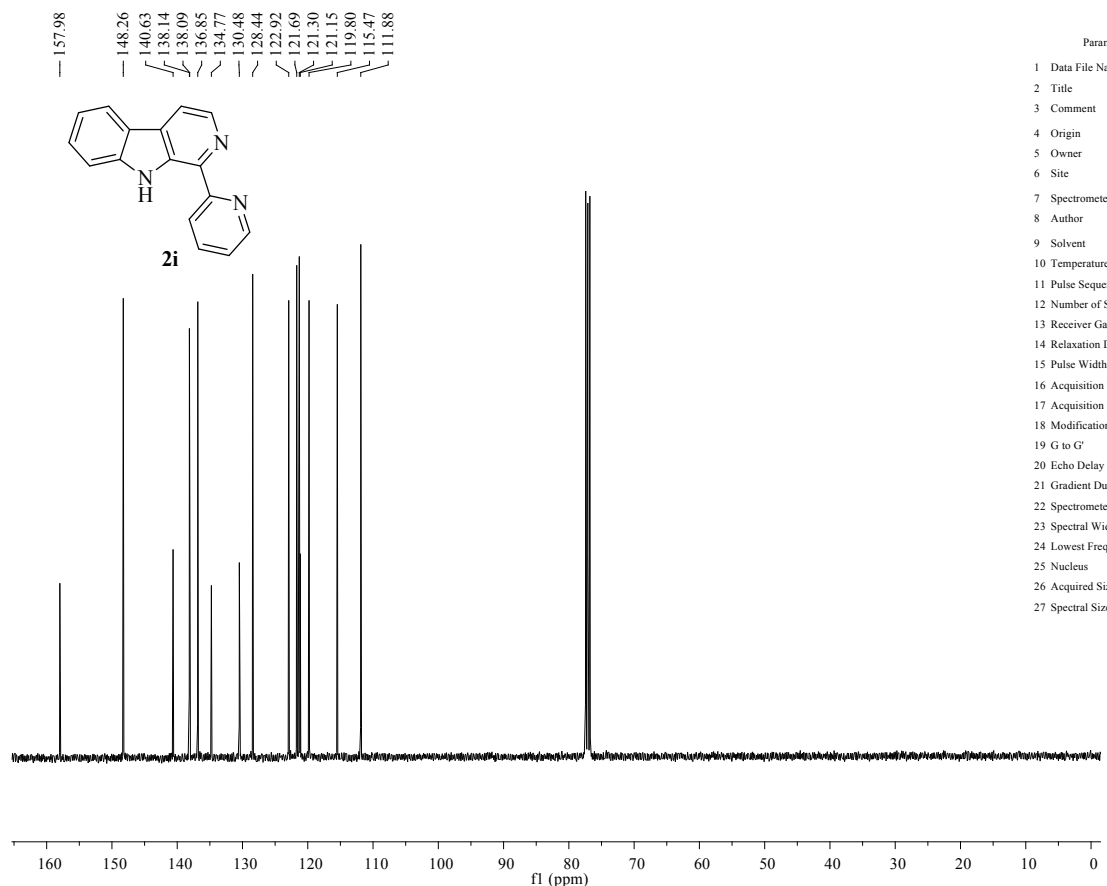
¹H NMR (CDCl₃, 400 MHz) spectrum of compound **2i**:



NAME SXX-ZJH-133
EXPNO 1
PROCNO 1
Date_ 20130419
Time 7.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 287
DW 60.800 usec
DE 6.50 usec
TE 298.3 K
D1 1.00000000 sec
TD0 1

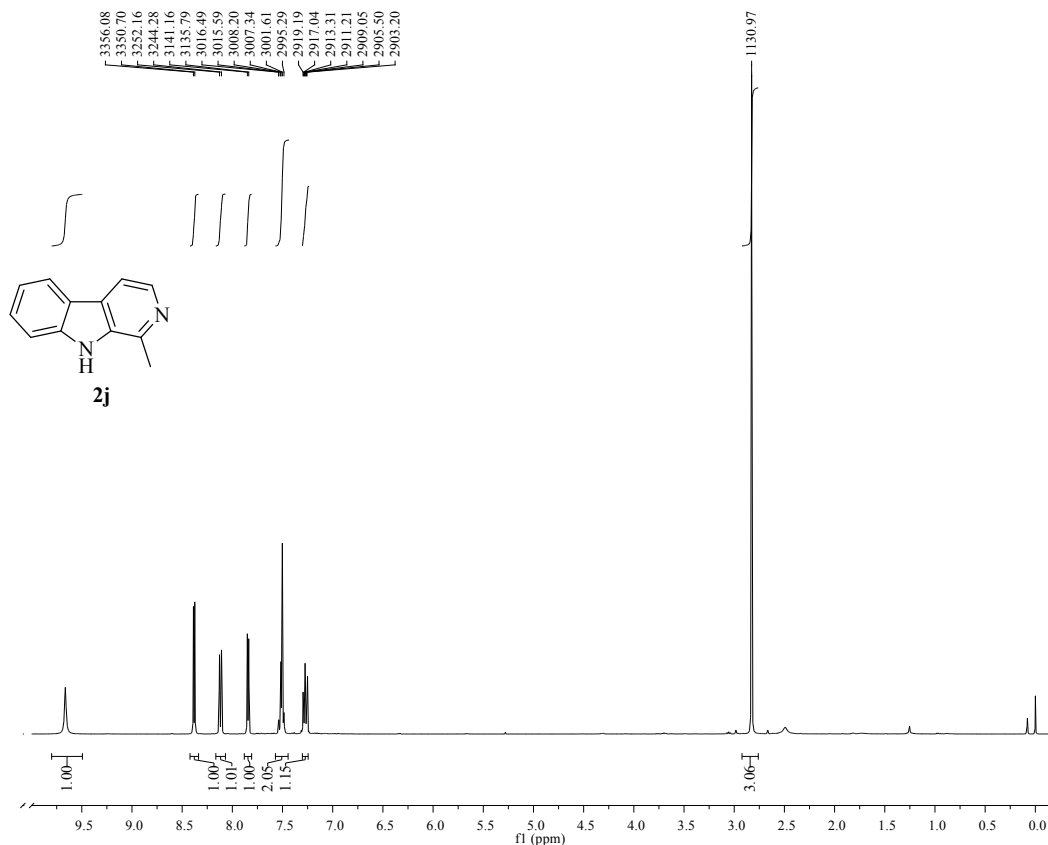
===== CHANNEL f1 =====
NUC1 1H
P1 13.30 usec
PL1 -2.00 dB
PL1W 15.28731537 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300161 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C NMR (CDCl₃, 100 MHz) spectrum of compound **2i**:



Parameter	Value
1 Data File Name	SXX-ZJH-133
2 Title	SXX-ZJH-133 (C)
3 Comment	
4 Origin	Bruker BioSpin GmbH
5 Owner	mnr
6 Site	
7 Spectrometer	spect
8 Author	
9 Solvent	CDCl3
10 Temperature	296.5
11 Pulse Sequence	zgpg30
12 Number of Scans	512
13 Receiver Gain	322
14 Relaxation Delay	2.0000
15 Pulse Width	9.4000
16 Acquisition Time	1.3631
17 Acquisition Date	2013-04-23T08:26:20
18 Modification Date	2013-04-23T08:26:00
19 G to G'	1.00000000
20 Echo Delay	0.0000
21 Gradient Duration	0.0000
22 Spectrometer Frequency	100.61
23 Spectral Width	24038.5
24 Lowest Frequency	-1958.4
25 Nucleus	13C
26 Acquired Size	32768
27 Spectral Size	32768

¹H NMR (CDCl₃, 400 MHz) spectrum of compound **2j**:



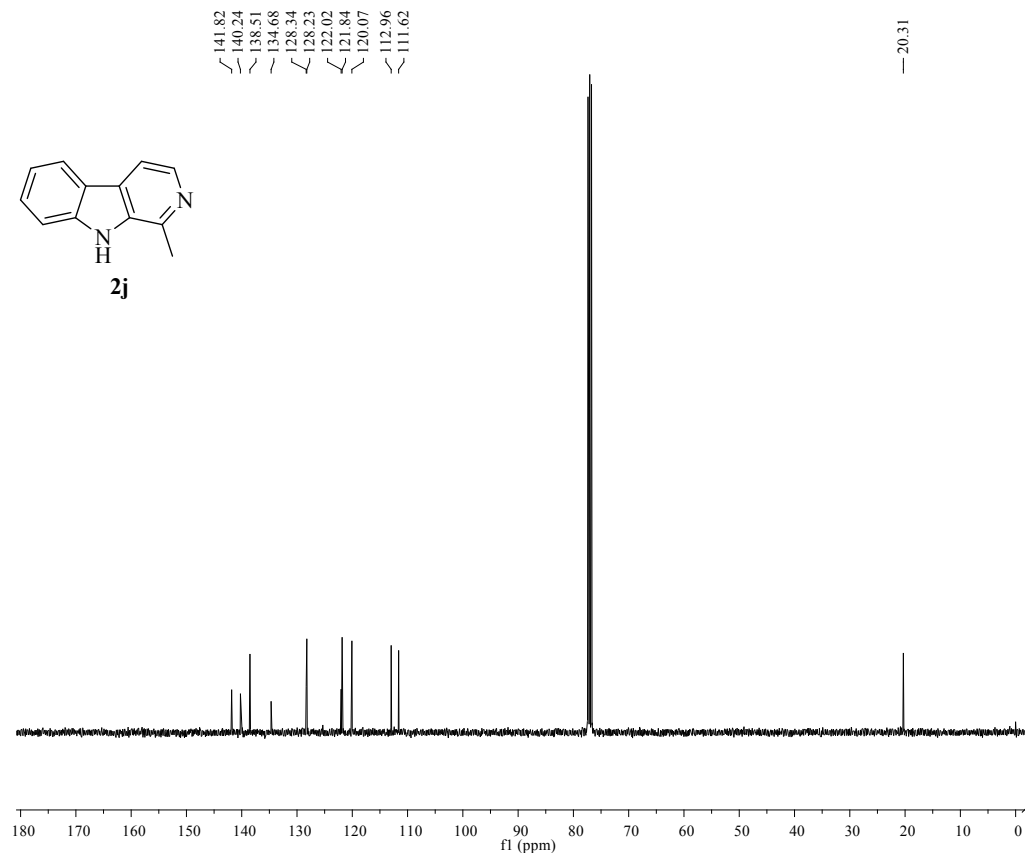
```

NAME      sxx-zjh-21
EXPNO     10
PROCNO    1
Date_     20120509
Time      16.50
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         0
SWH       8223.685 Hz
FIDRES    0.125483 Hz
AQ         3.9846387 sec
RG         128
DW         60.800 usec
DE         6.50 usec
TE         295.0 K
D1         1.00000000 sec
TD0        1
    
```

```

===== CHANNEL f1 =====
NUC1      1H
P1         14.90 usec
PL1        -1.00 dB
PL1W      13.37643433 W
SFO1      400.1324710 MHz
SI         32768
SF         400.1300135 MHz
WDW        EM
SSB         0
LB         0.30 Hz
GB         0
PC         1.00
    
```

¹³C NMR (CDCl₃, 100 MHz) spectrum of compound **2j**:



```

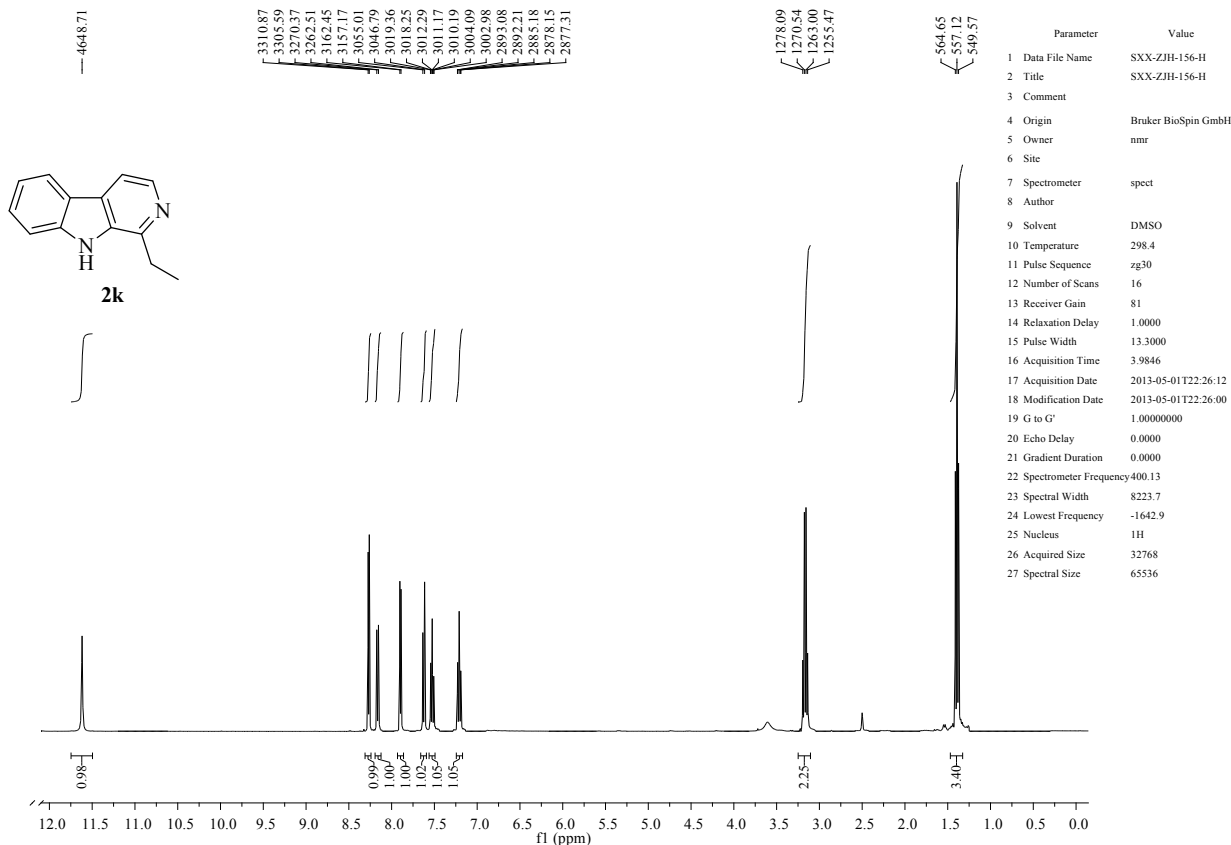
NAME      sxx-zjh-21
EXPNO     10
PROCNO    1
Date_     20120520
Time      8.11
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         400
DS         0
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ         1.3631988 sec
RG         203
DW         20.800 usec
DE         6.50 usec
TE         295.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
    
```

```

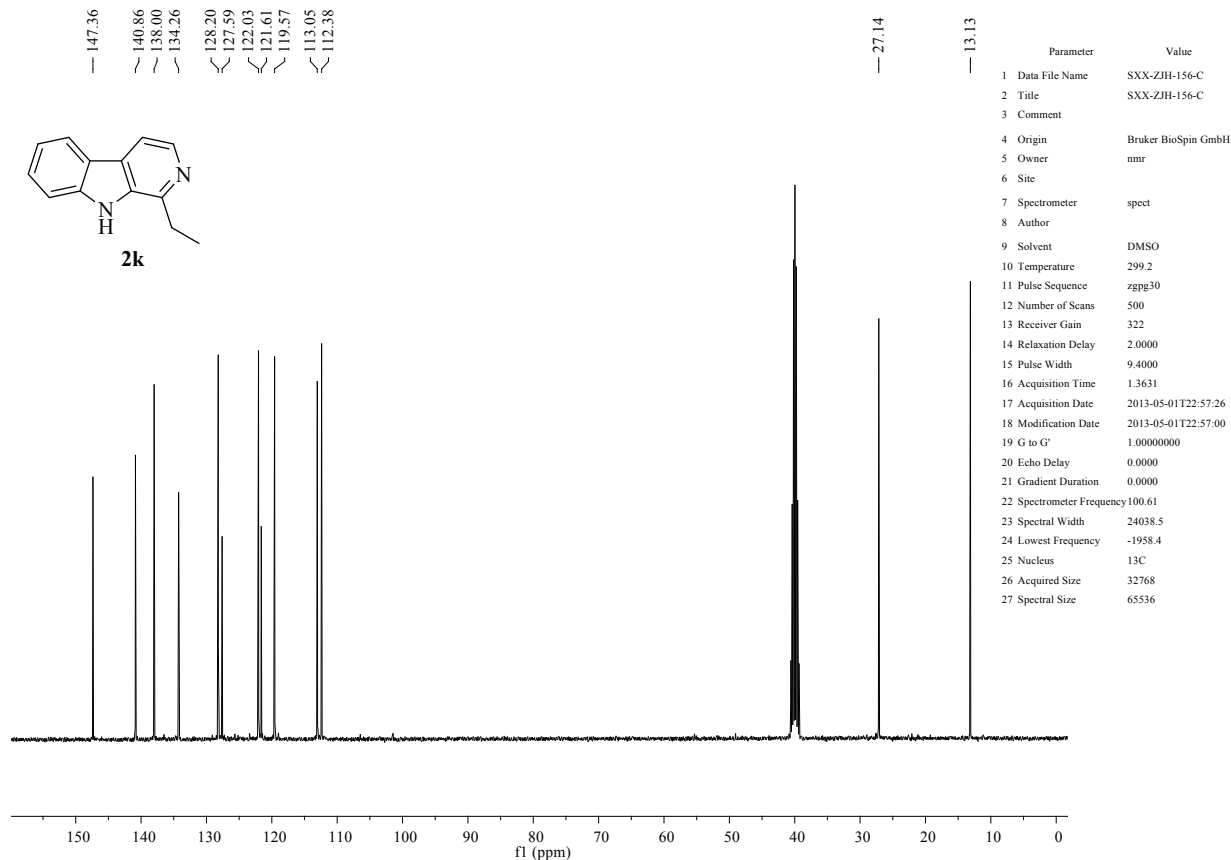
===== CHANNEL f1 =====
NUC1      13C
P1         9.00 usec
PL1        -2.00 dB
PL1W      57.32743073 W
SFO1      100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     90.00 usec
PL2        -1.00 dB
PL12       14.90 dB
PL13       17.90 dB
PL2W      13.37643433 W
PL12W     0.34382734 W
PL13W     0.17232187 W
SFO2      400.1316005 MHz
SI         32768
SF         100.6127690 MHz
WDW        EM
SSB         0
LB         1.00 Hz
GB         0
PC         1.00
    
```

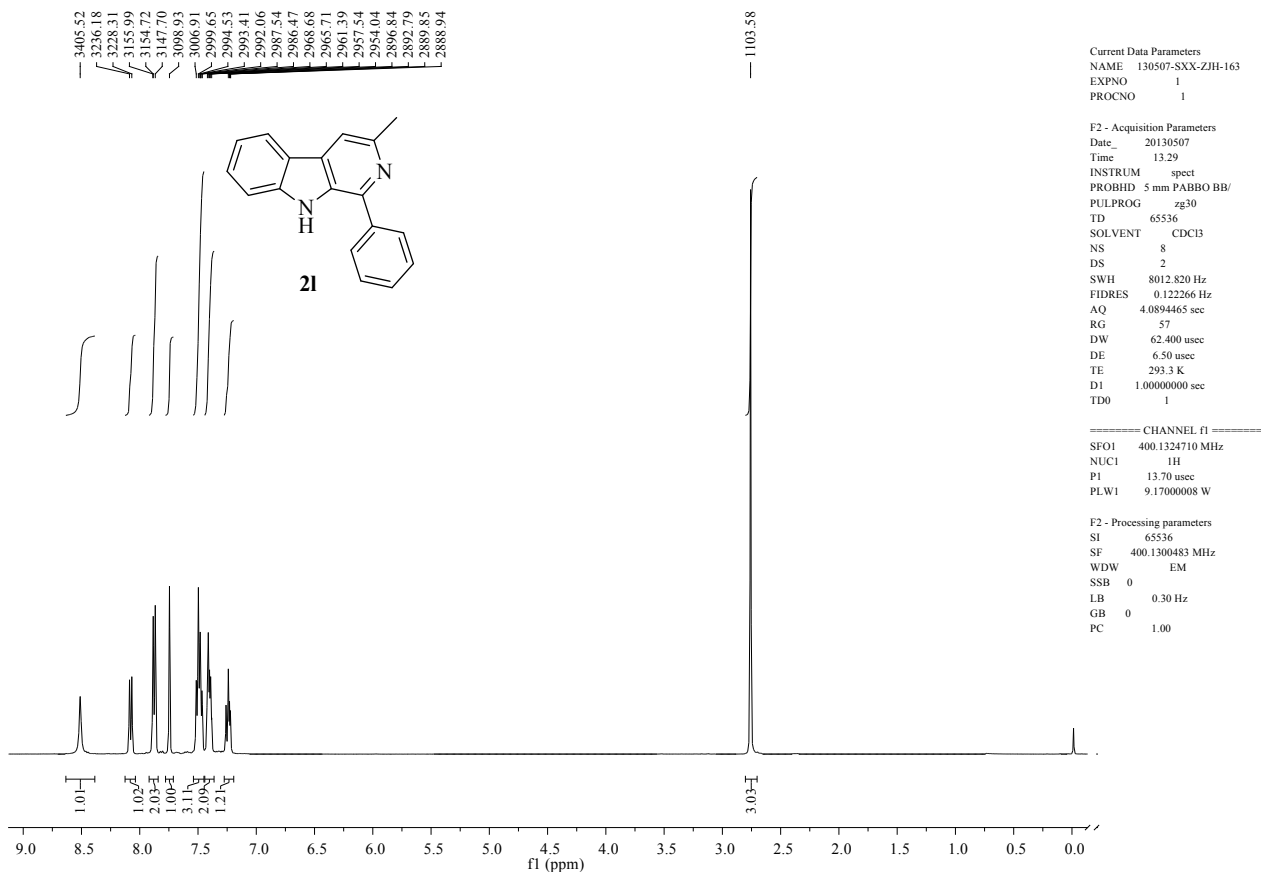
¹H NMR (DMSO-*d*₆, 400 MHz) spectrum of compound **2k**:



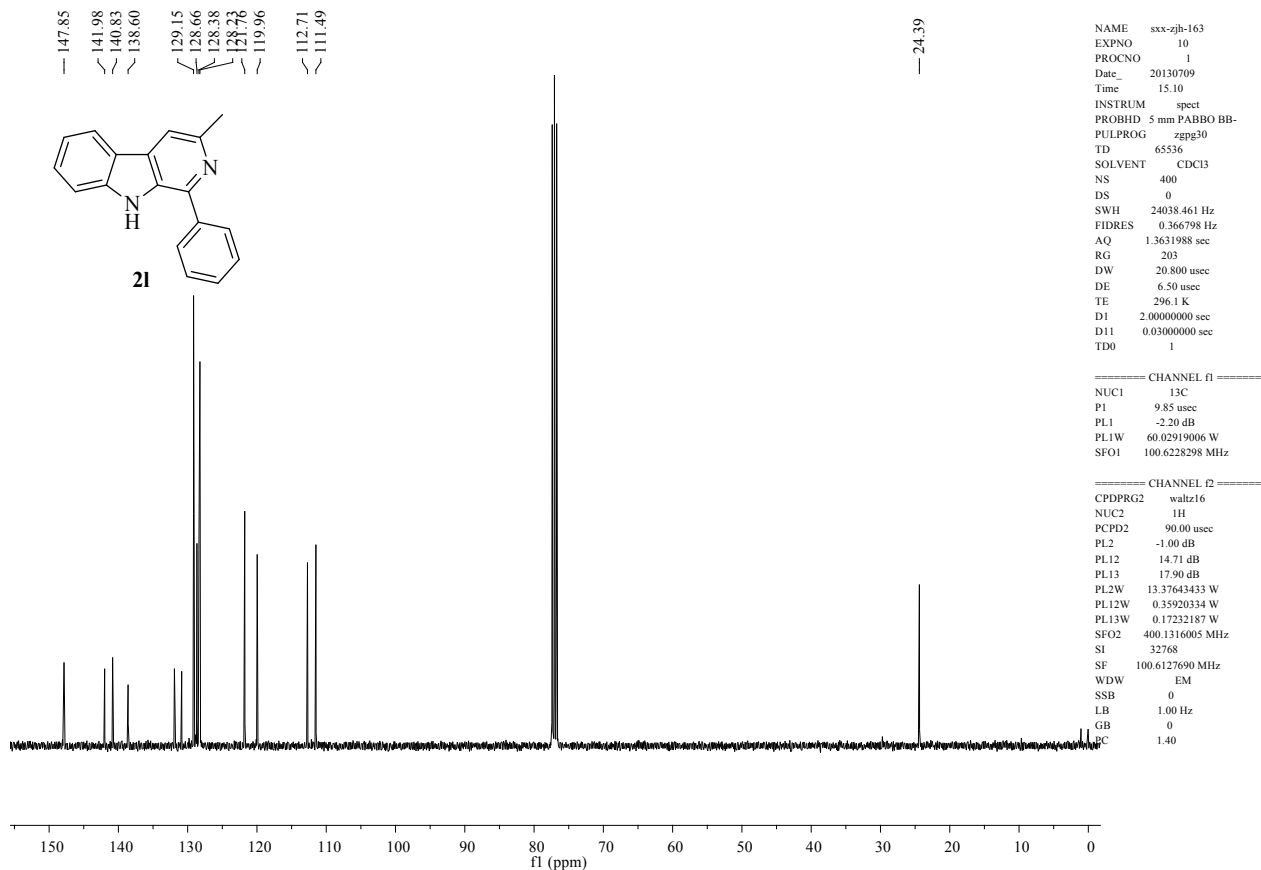
¹³C NMR (DMSO-*d*₆, 100 MHz) spectrum of compound **2k**:



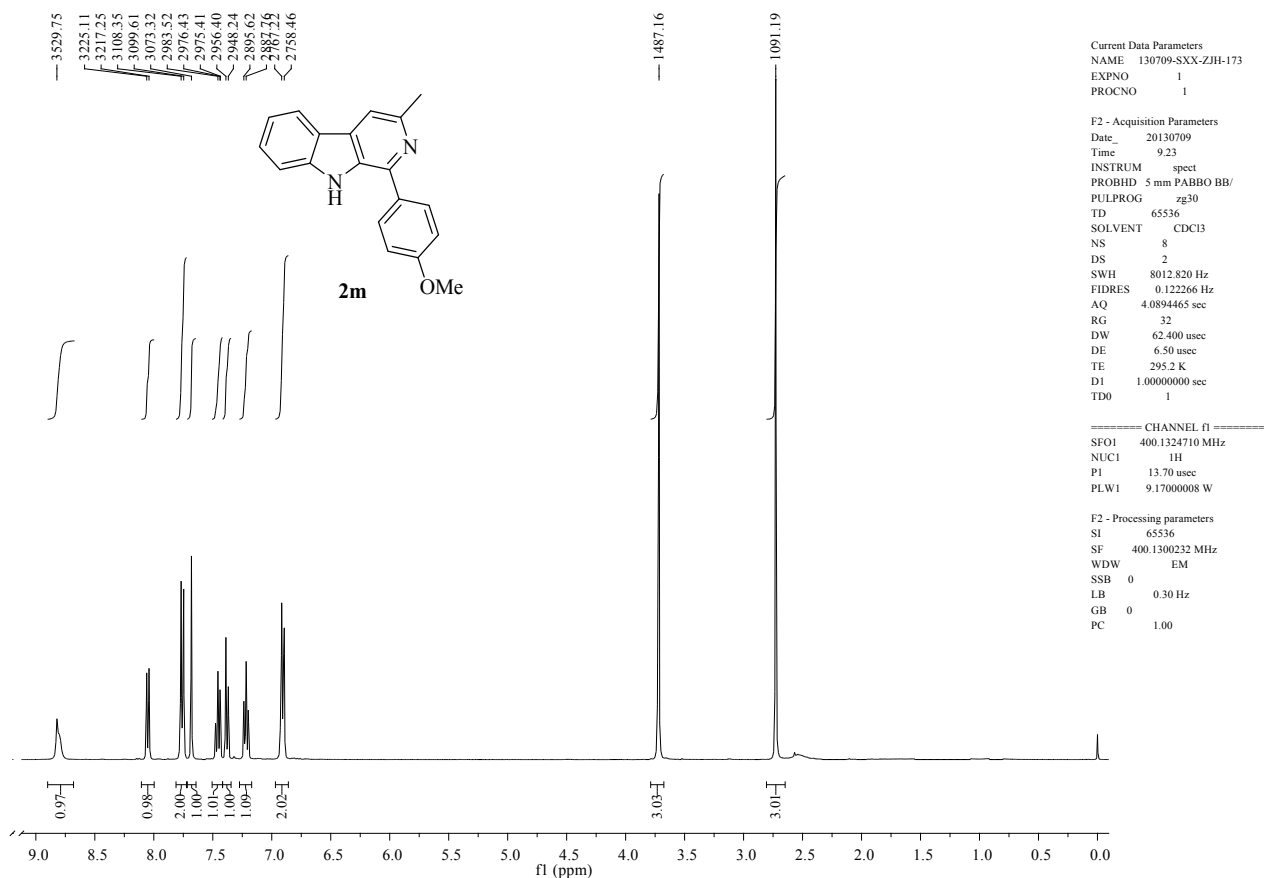
¹H NMR (CDCl₃, 400 MHz) spectrum of compound **21**:



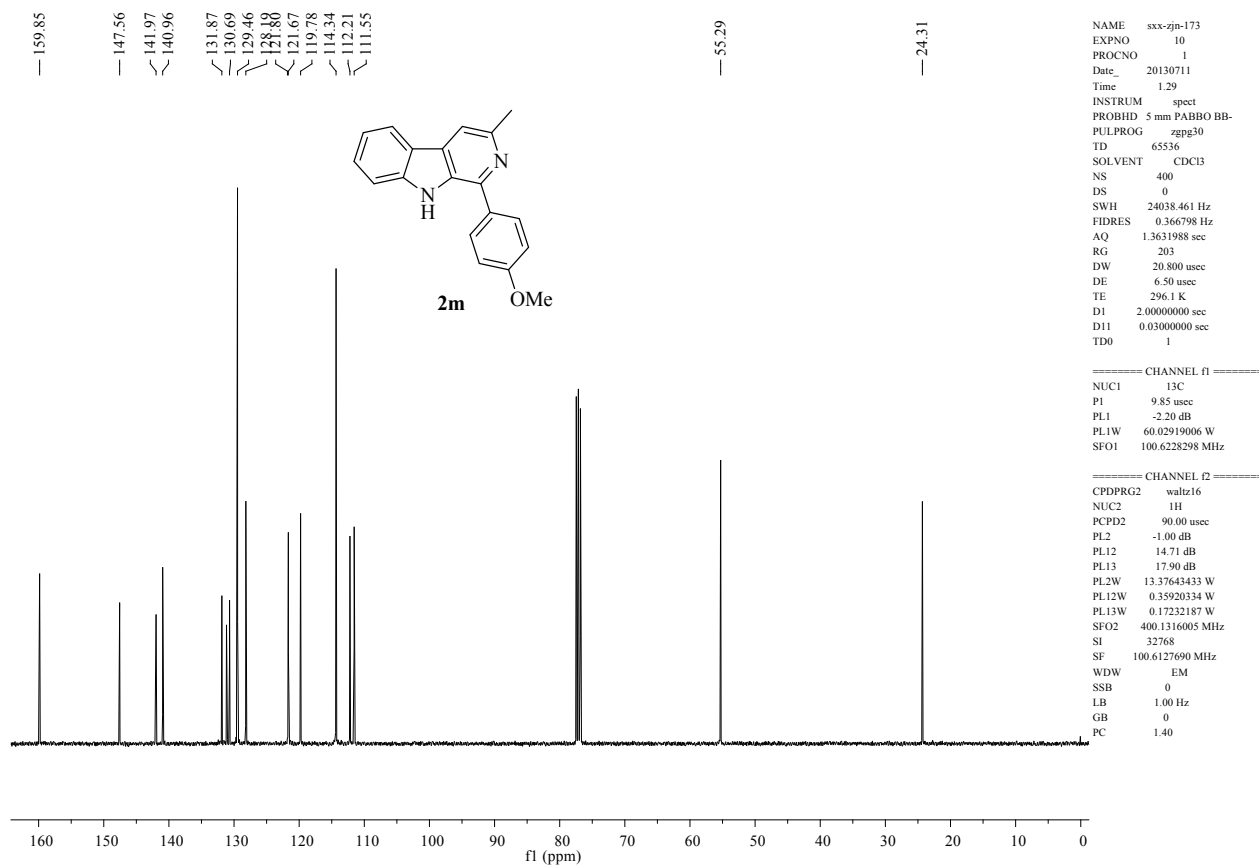
¹³C NMR (CDCl₃, 100 MHz) spectrum of compound **21**:



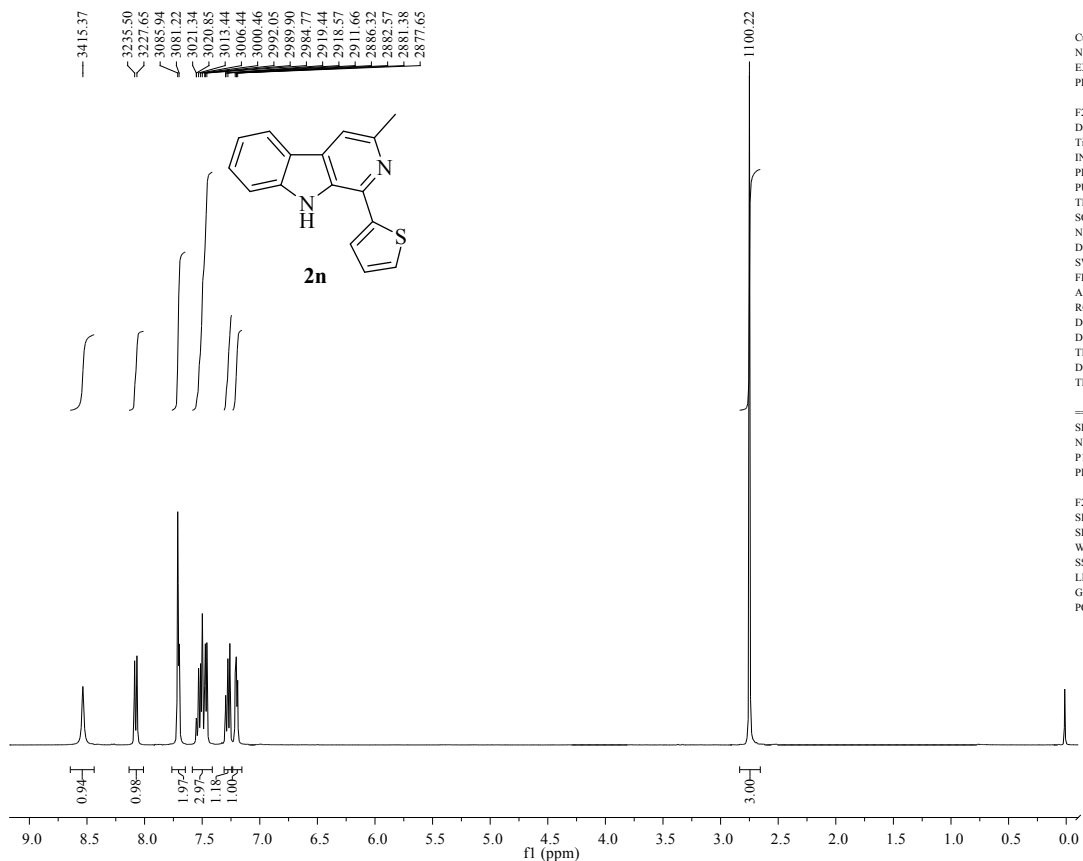
¹H NMR (CDCl₃, 400 MHz) spectrum of compound **2m**:



¹³C NMR (CDCl₃, 100 MHz) spectrum of compound **2m**:



¹H NMR (CDCl₃, 400 MHz) spectrum of compound **2n**:



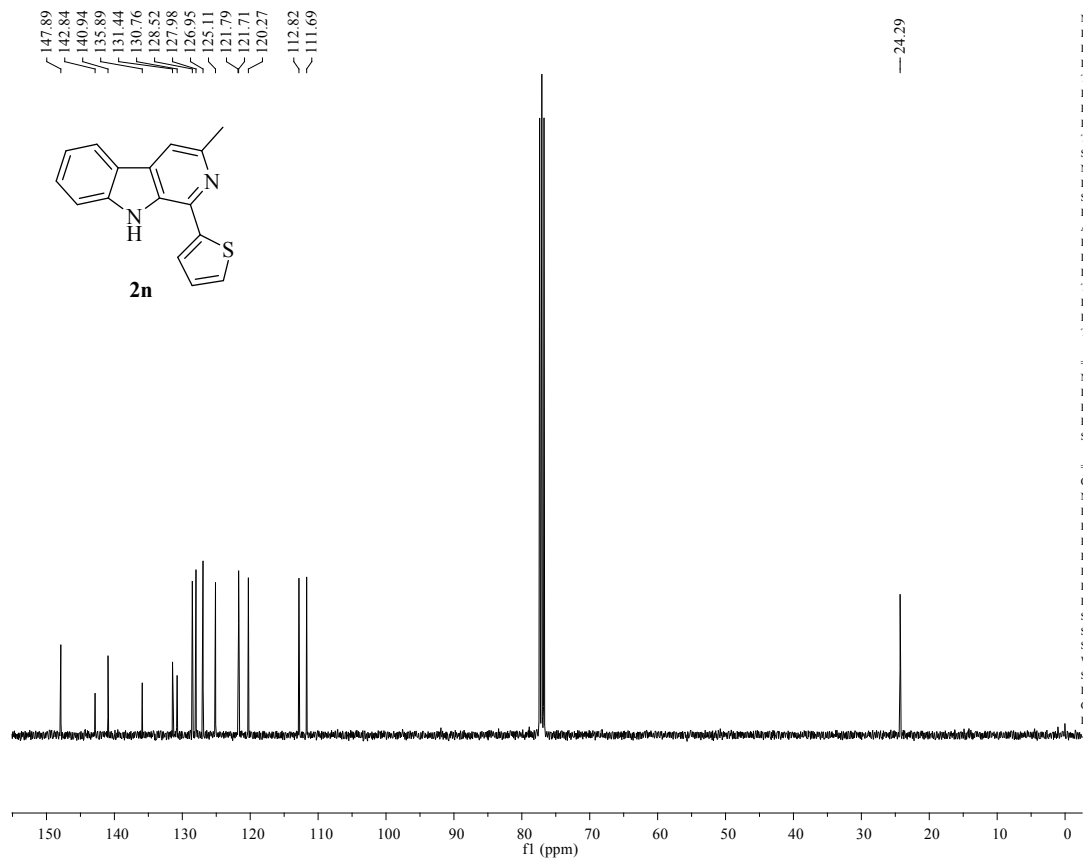
Current Data Parameters
 NAME 130709-SXX-ZH-172
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20130709
 Time 9.19
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 71.8
 DW 62.400 usec
 DE 6.50 usec
 TE 295.1 K
 D1 1.00000000 sec
 TDO 1

==== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 13.70 usec
 PLW1 9.17000008 W

F2 - Processing parameters
 SI 65536
 SF 400.1300152 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

¹³C NMR (CDCl₃, 100 MHz) spectrum of compound **2n**:

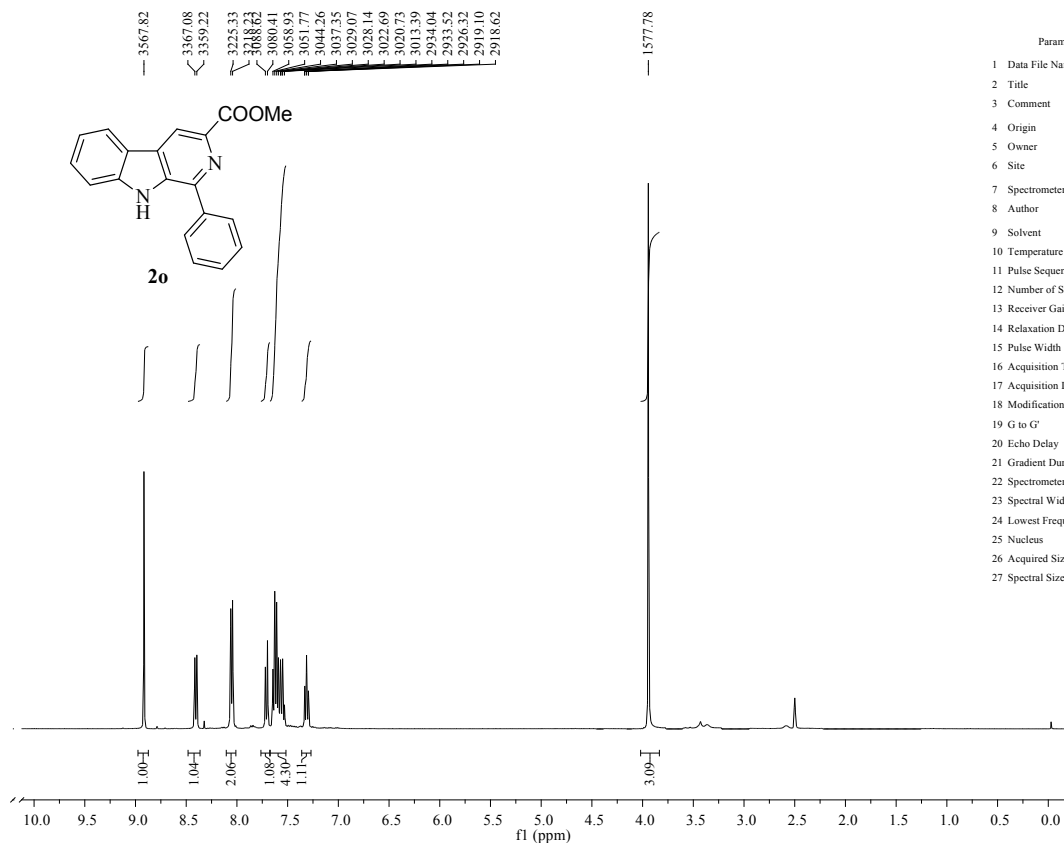


NAME sxx-zjn-172
 EXPNO 10
 PROCNO 1
 Date_ 20130711
 Time 1.01
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 400
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 203
 DW 20.800 usec
 DE 6.50 usec
 TE 296.3 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1

==== CHANNEL f1 =====
 NUC1 13C
 P1 9.85 usec
 PL1 -2.20 dB
 PL1W 60.02919006 W
 SFO1 100.6228298 MHz

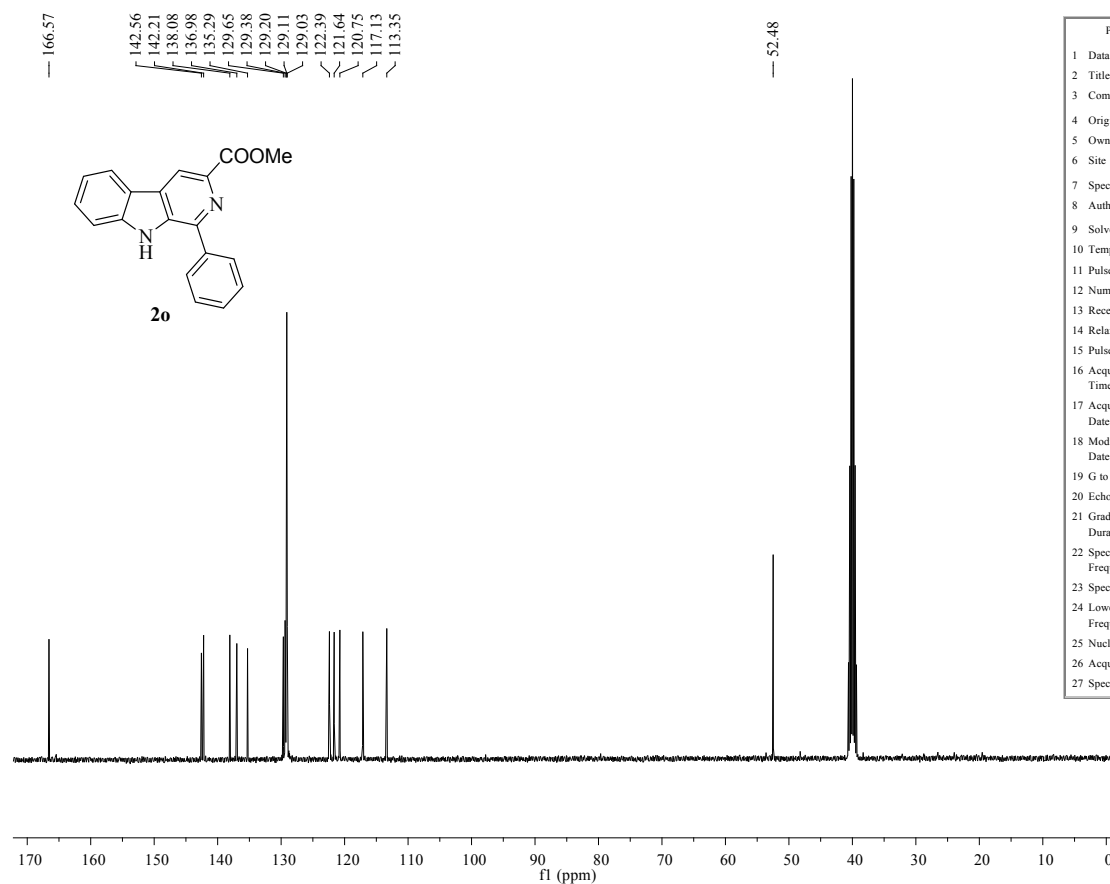
==== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 -1.00 dB
 PL12 14.71 dB
 PL13 17.90 dB
 PL2W 13.37643433 W
 PL12W 0.35920334 W
 PL13W 0.17232187 W
 SFO2 400.1316005 MHz
 SI 32768
 SF 100.6127690 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

¹H NMR (DMSO-*d*₆, 400 MHz) spectrum of compound **2o**:



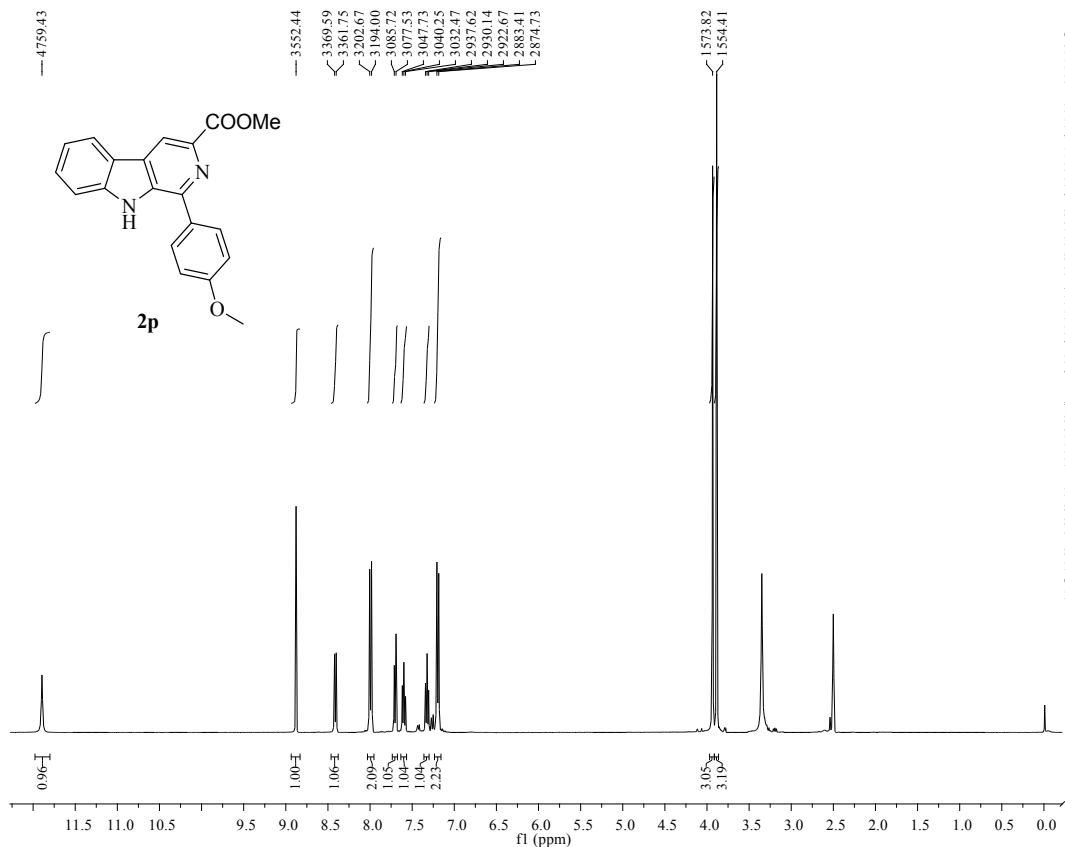
Parameter	Value
1 Data File Name	SXX-ZJH-160-H
2 Title	SXX-ZJH-160-H
3 Comment	
4 Origin	Bruker BioSpin GmbH
5 Owner	nmr
6 Site	
7 Spectrometer	spect
8 Author	
9 Solvent	DMSO
10 Temperature	298.3
11 Pulse Sequence	zg30
12 Number of Scans	16
13 Receiver Gain	161
14 Relaxation Delay	1.0000
15 Pulse Width	13.3000
16 Acquisition Time	3.9846
17 Acquisition Date	2013-05-02T00:12:54
18 Modification Date	2013-05-02T00:12:00
19 G to G'	1.00000000
20 Echo Delay	0.0000
21 Gradient Duration	0.0000
22 Spectrometer Frequency	400.13
23 Spectral Width	8223.7
24 Lowest Frequency	-1642.9
25 Nucleus	¹ H
26 Acquired Size	32768
27 Spectral Size	65536

¹³C NMR (DMSO-*d*₆, 100 MHz) spectrum of compound **2o**:



Parameter	Value
1 Data File Name	SXX-ZJH-160-C/1/ fid
2 Title	SXX-ZJH-160-C
3 Comment	
4 Origin	Bruker BioSpin GmbH
5 Owner	nmr
6 Site	
7 Spectrometer	spect
8 Author	
9 Solvent	DMSO
10 Temperature	299.1
11 Pulse Sequence	zgpg30
12 Number of Scans	500
13 Receiver Gain	362
14 Relaxation Delay	2.0000
15 Pulse Width	9.4000
16 Acquisition Time	1.3631
17 Acquisition Date	2013-05-02T00:44:24
18 Modification Date	2013-05-02T00:44:00
19 G to G'	1.00000000
20 Echo Delay	0.0000
21 Gradient Duration	0.0000
22 Spectrometer Frequency	100.61
23 Spectral Width	24038.5
24 Lowest Frequency	-1958.4
25 Nucleus	¹³ C
26 Acquired Size	32768
27 Spectral Size	65536

¹H NMR (DMSO-d₆, 400 MHz) spectrum of compound **2p**:



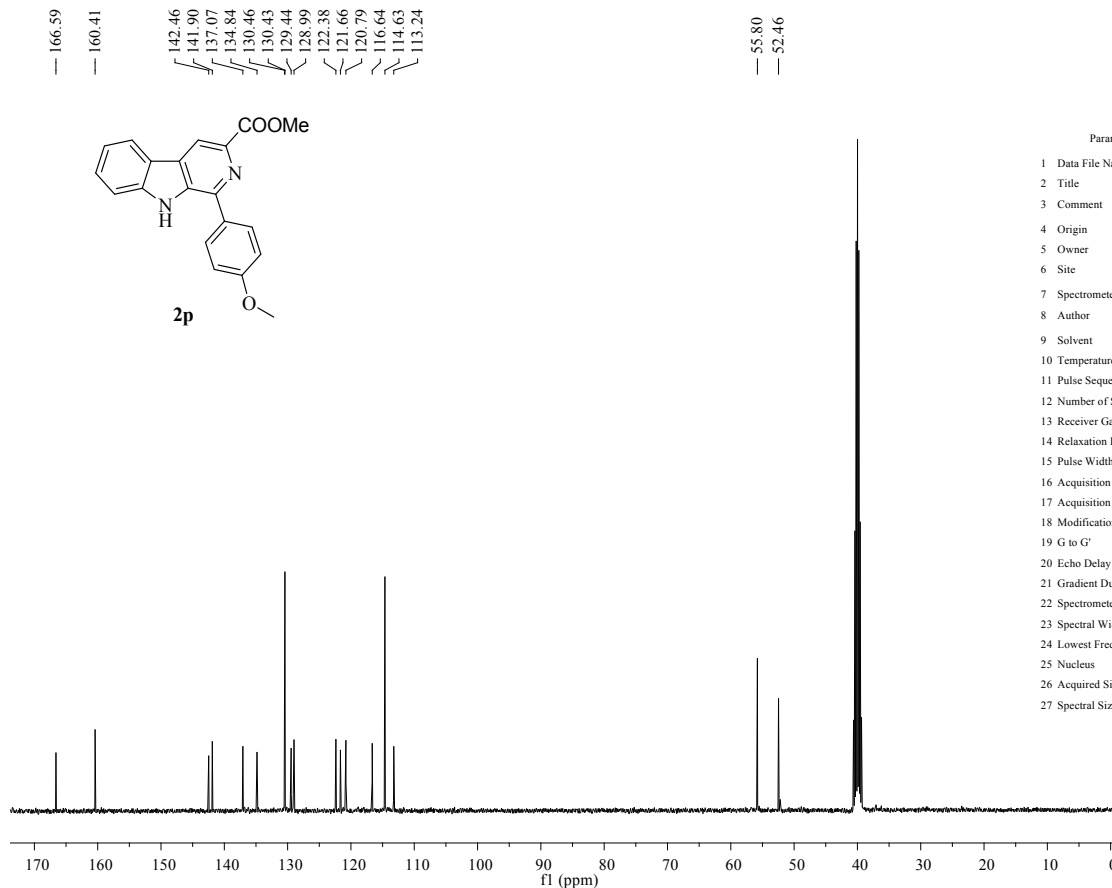
Current Data Parameters
 NAME 130428-SXX-ZJH-161
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20130428
 Time 10.07
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 8
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 71.8
 DW 62.400 usec
 DE 6.50 usec
 TE 296.1 K
 D1 1.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 13.70 usec
 PLW1 9.17000008 W

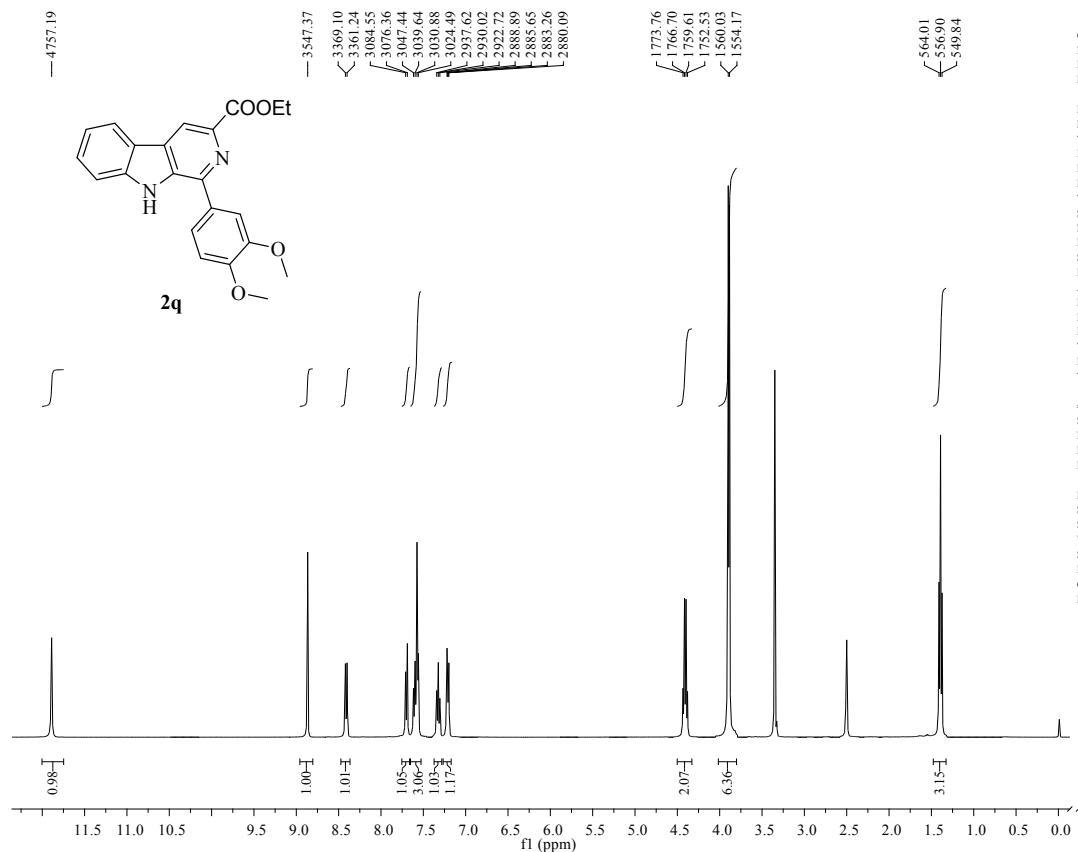
F2 - Processing parameters
 SI 65536
 SF 400.1299994 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

¹³C NMR (DMSO-d₆, 100 MHz) spectrum of compound **2p**:



Parameter	Value
1 Data File Name	SXX-XJN-161
2 Title	SXX-XJN-161
3 Comment	
4 Origin	Bruker BioSpin GmbH
5 Owner	nmr
6 Site	
7 Spectrometer	spect
8 Author	
9 Solvent	DMSO
10 Temperature	299.2
11 Pulse Sequence	zgpg30
12 Number of Scans	512
13 Receiver Gain	161
14 Relaxation Delay	2.0000
15 Pulse Width	9.4000
16 Acquisition Time	1.3631
17 Acquisition Date	2013-05-03T22:05:37
18 Modification Date	2013-05-03T22:05:00
19 G to G'	1.00000000
20 Echo Delay	0.0000
21 Gradient Duration	0.0000
22 Spectrometer Frequency	100.61
23 Spectral Width	24038.5
24 Lowest Frequency	-1958.4
25 Nucleus	13C
26 Acquired Size	32768
27 Spectral Size	65536

¹H NMR (DMSO-*d*₆, 400 MHz) spectrum of compound **2q**:



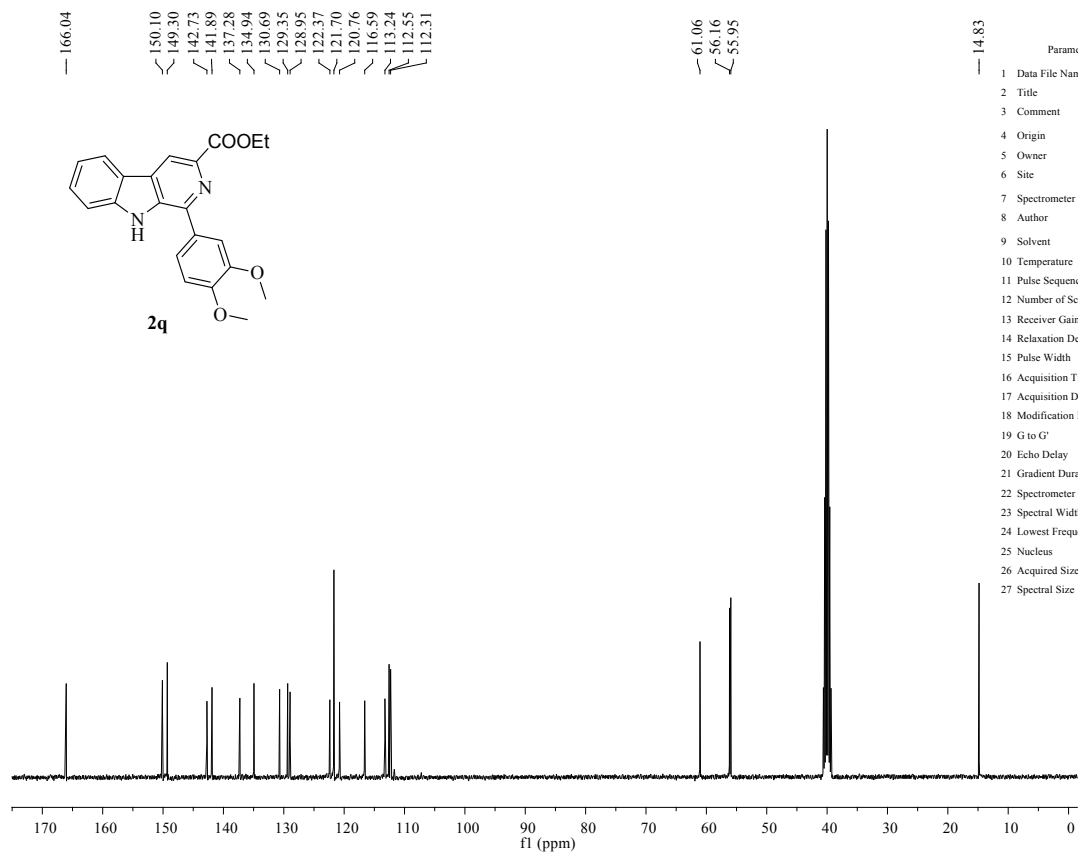
Current Data Parameters
 NAME 130428-SXX-ZJH-162
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20130428
 Time 10.10
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 8
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 64
 DW 62.400 usec
 DE 6.50 usec
 TE 296.0 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 13.70 usec
 PLW1 9.17000008 W

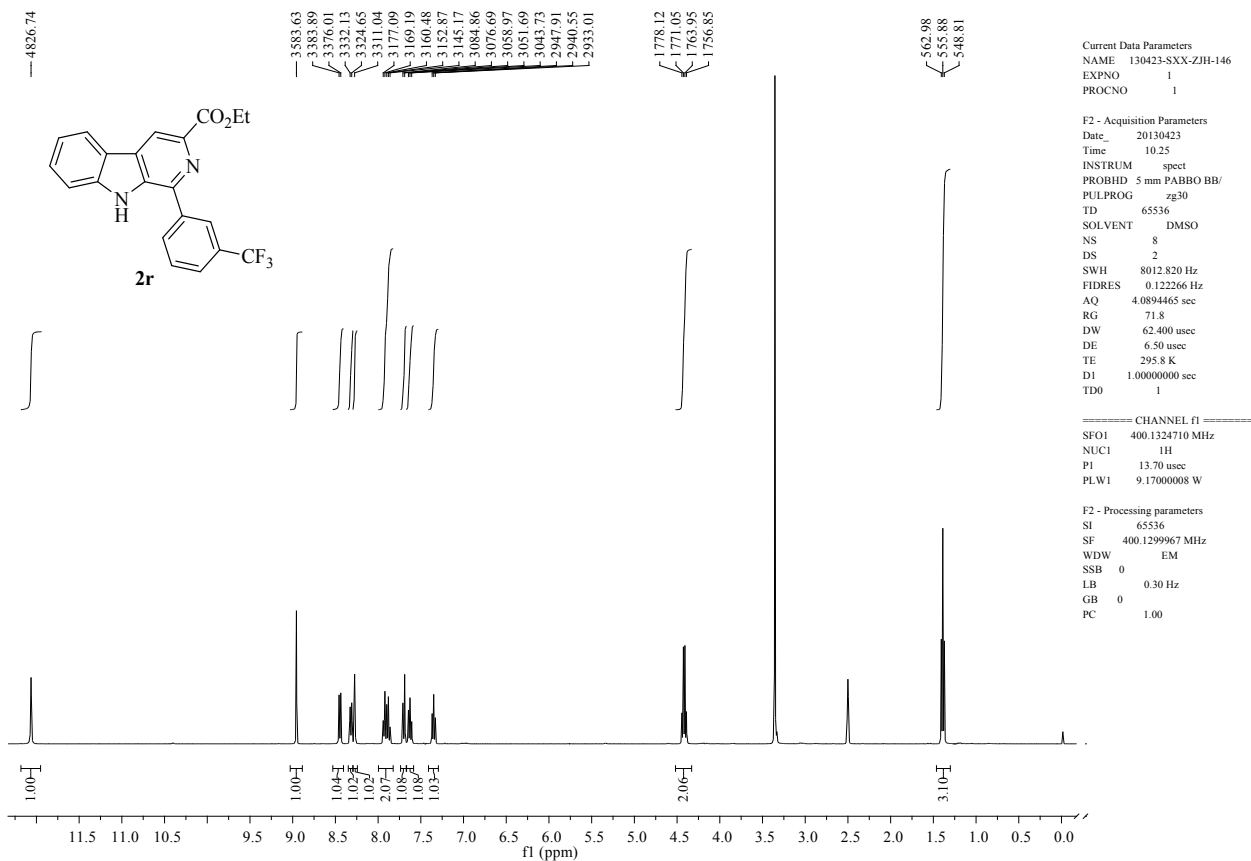
F2 - Processing parameters
 SI 65536
 SF 400.129985 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

¹³C NMR (DMSO-*d*₆, 100 MHz) spectrum of compound **2q**:

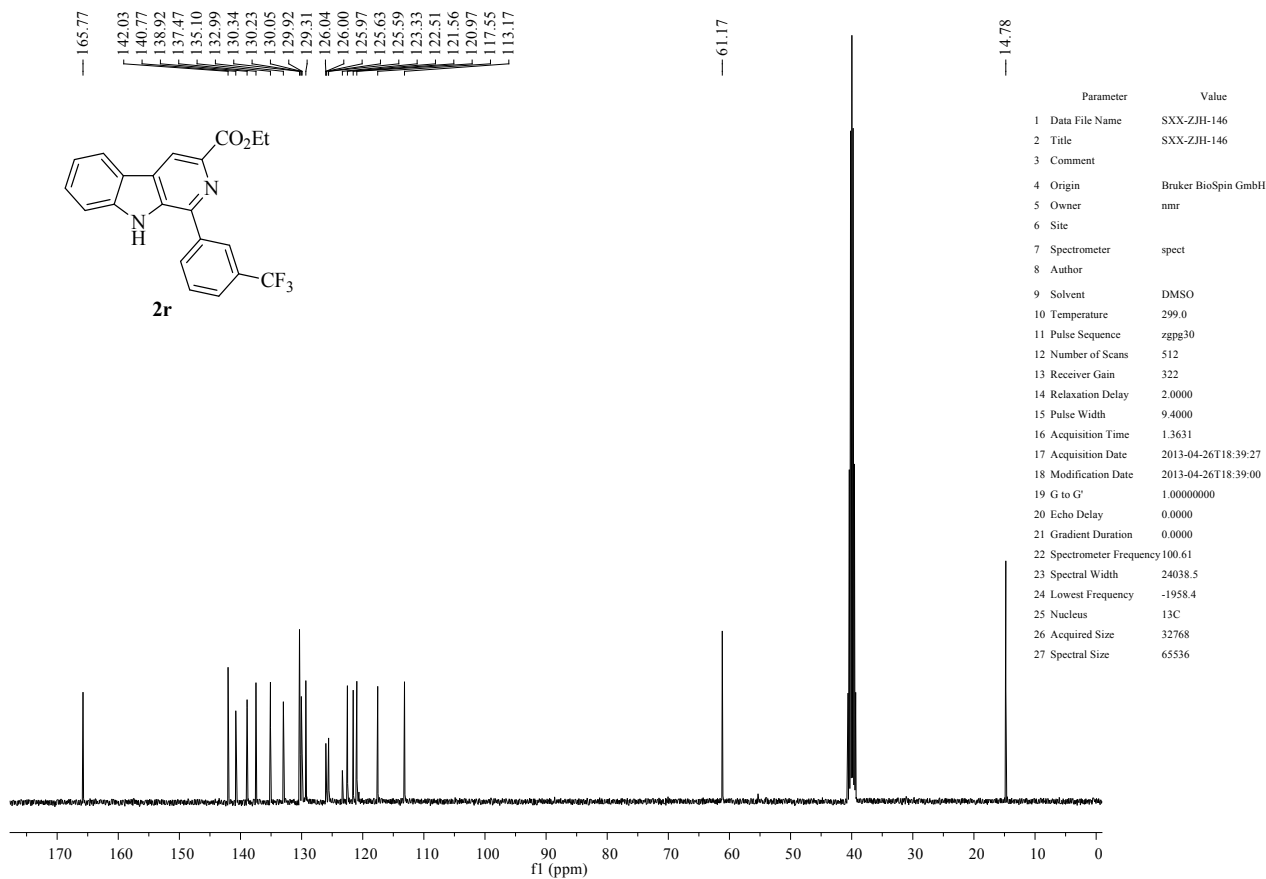


Parameter	Value
1 Data File Name	SXX-XJN-162 (C)
2 Title	SXX-XJN-162 (C)
3 Comment	
4 Origin	Bruker BioSpin GmbH
5 Owner	nmr
6 Site	
7 Spectrometer	spect
8 Author	
9 Solvent	DMSO
10 Temperature	299.2
11 Pulse Sequence	zgpg30
12 Number of Scans	512
13 Receiver Gain	181
14 Relaxation Delay	2.0000
15 Pulse Width	9.4000
16 Acquisition Time	1.3631
17 Acquisition Date	2013-05-03T22:37:58
18 Modification Date	2013-05-03T22:37:00
19 G to G'	1.00000000
20 Echo Delay	0.0000
21 Gradient Duration	0.0000
22 Spectrometer Frequency	100.61
23 Spectral Width	24038.5
24 Lowest Frequency	-1958.4
25 Nucleus	13C
26 Acquired Size	32768
27 Spectral Size	65536

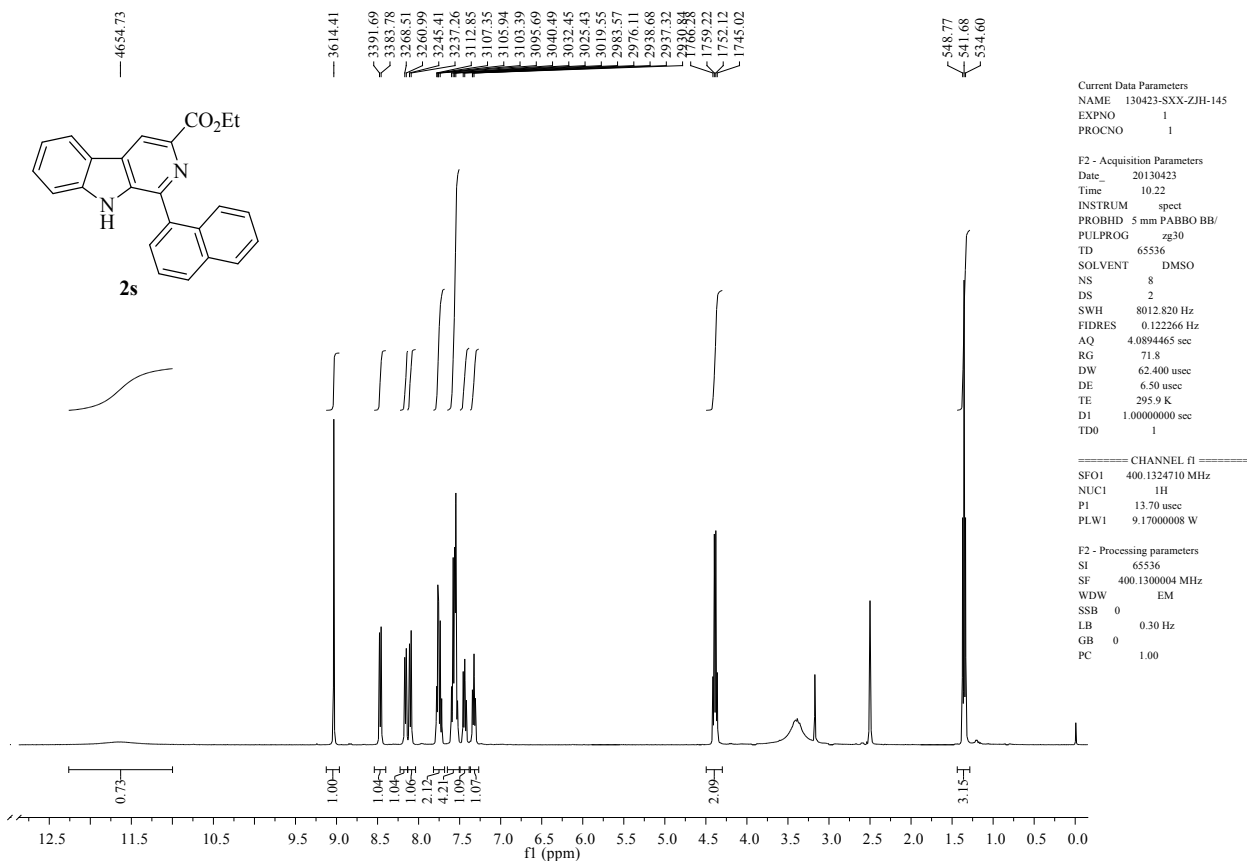
¹H NMR (DMSO-*d*₆, 400 MHz) spectrum of compound **2r**:



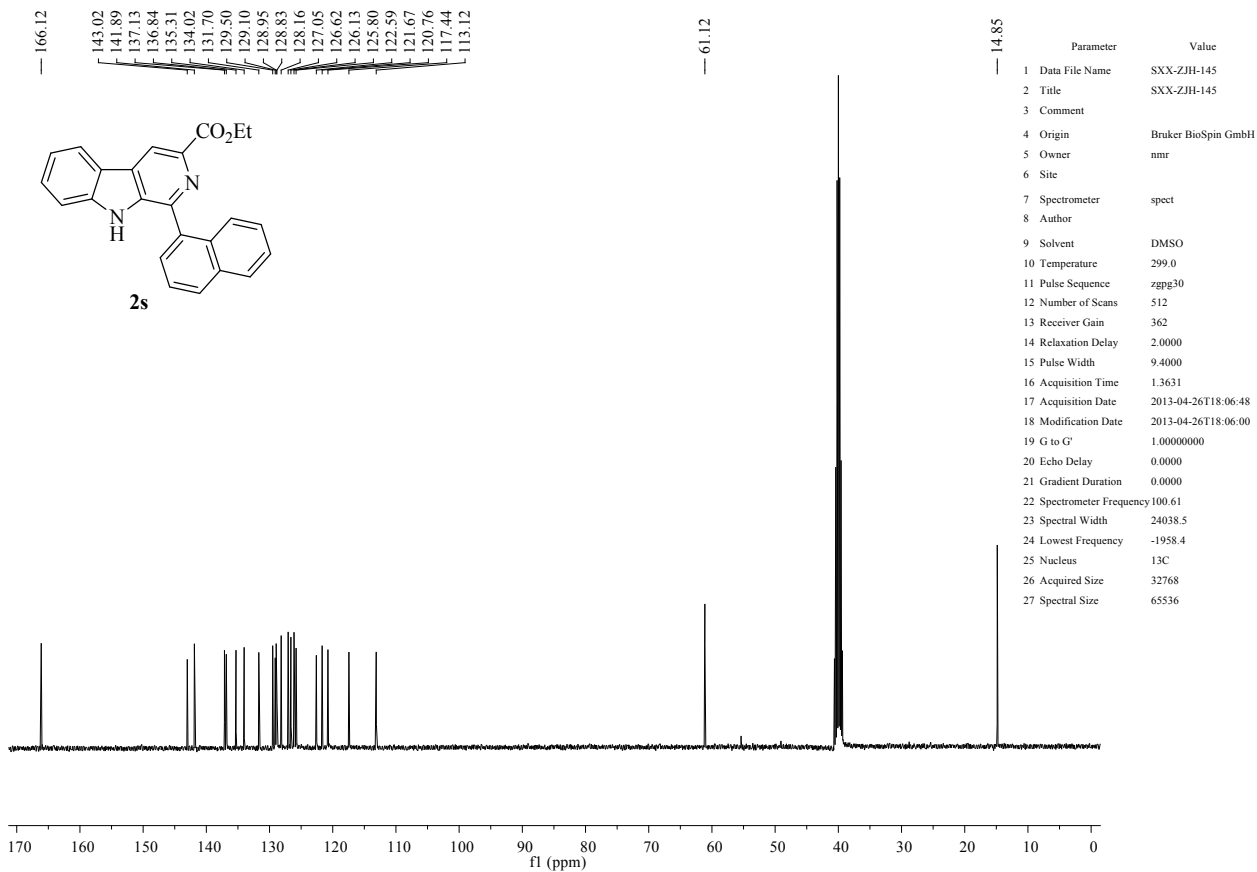
¹³C NMR (DMSO-*d*₆, 100 MHz) spectrum of compound **2r**:



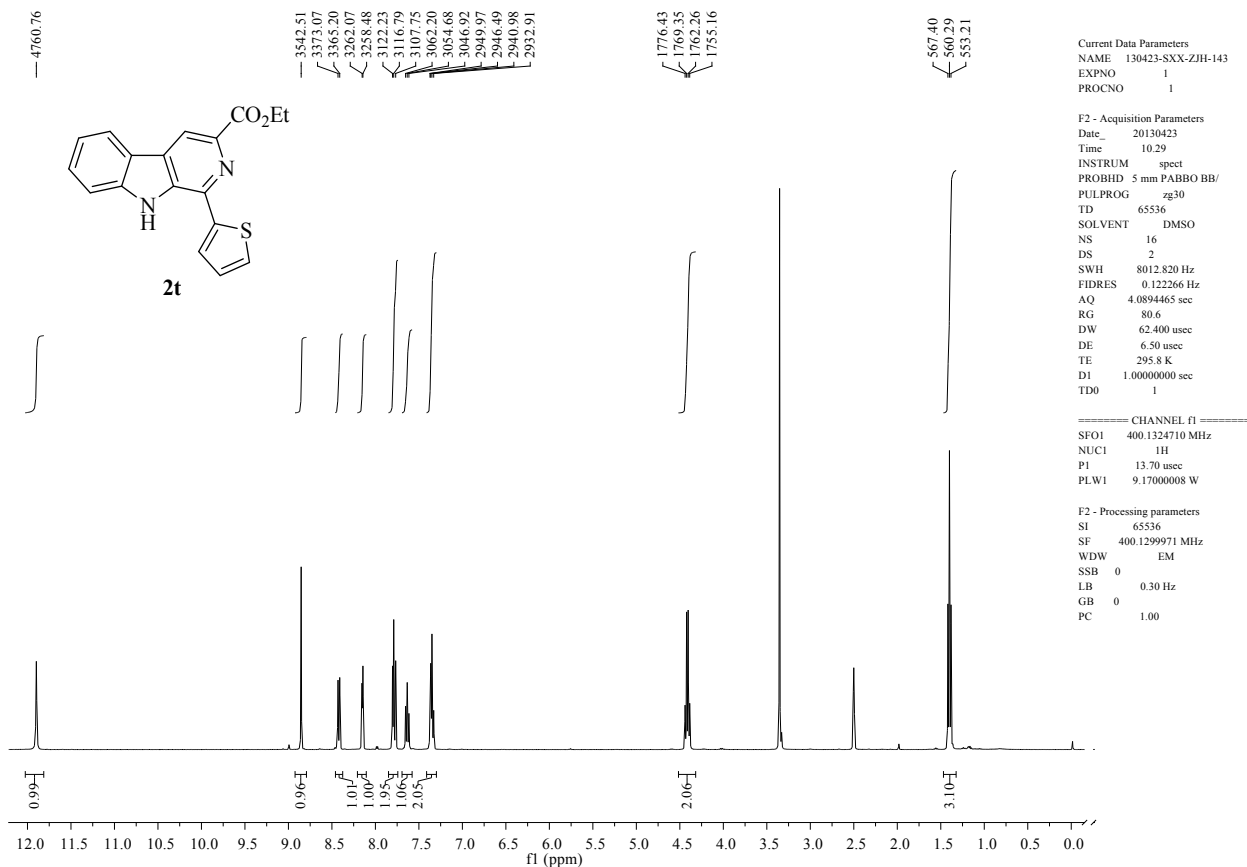
¹H NMR (DMSO-*d*₆, 400 MHz) spectrum of compound **2s**:



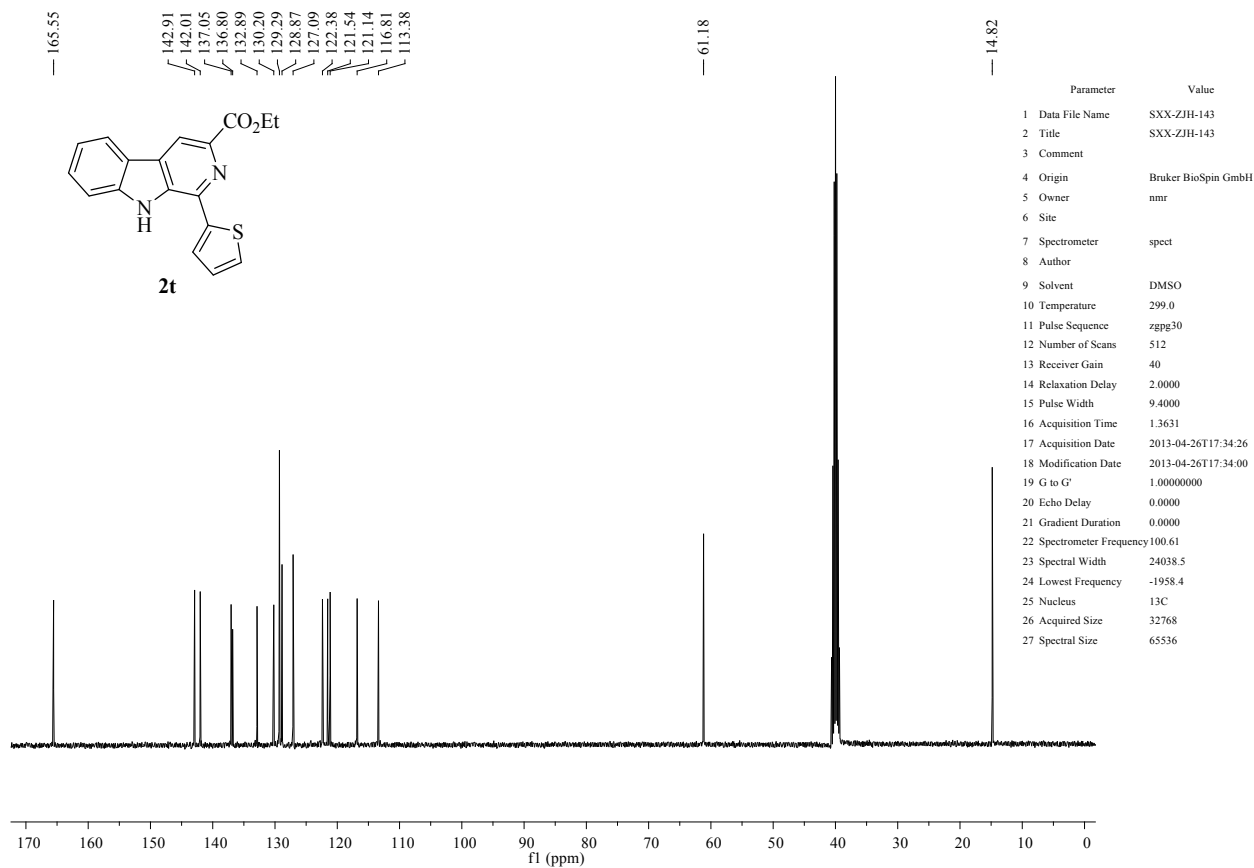
¹³C NMR (DMSO-*d*₆, 100 MHz) spectrum of compound **2s**:



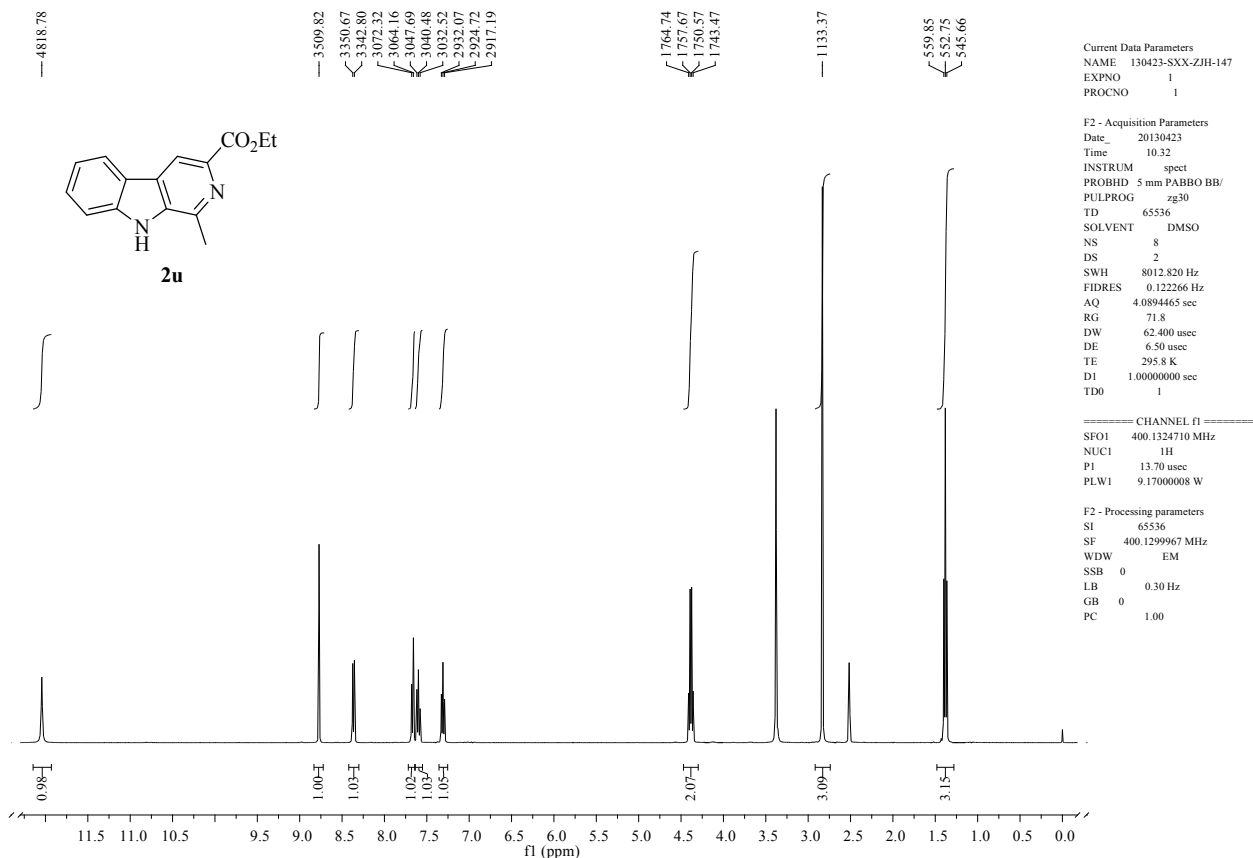
¹H NMR (DMSO-*d*₆, 400 MHz) spectrum of compound **2t**:



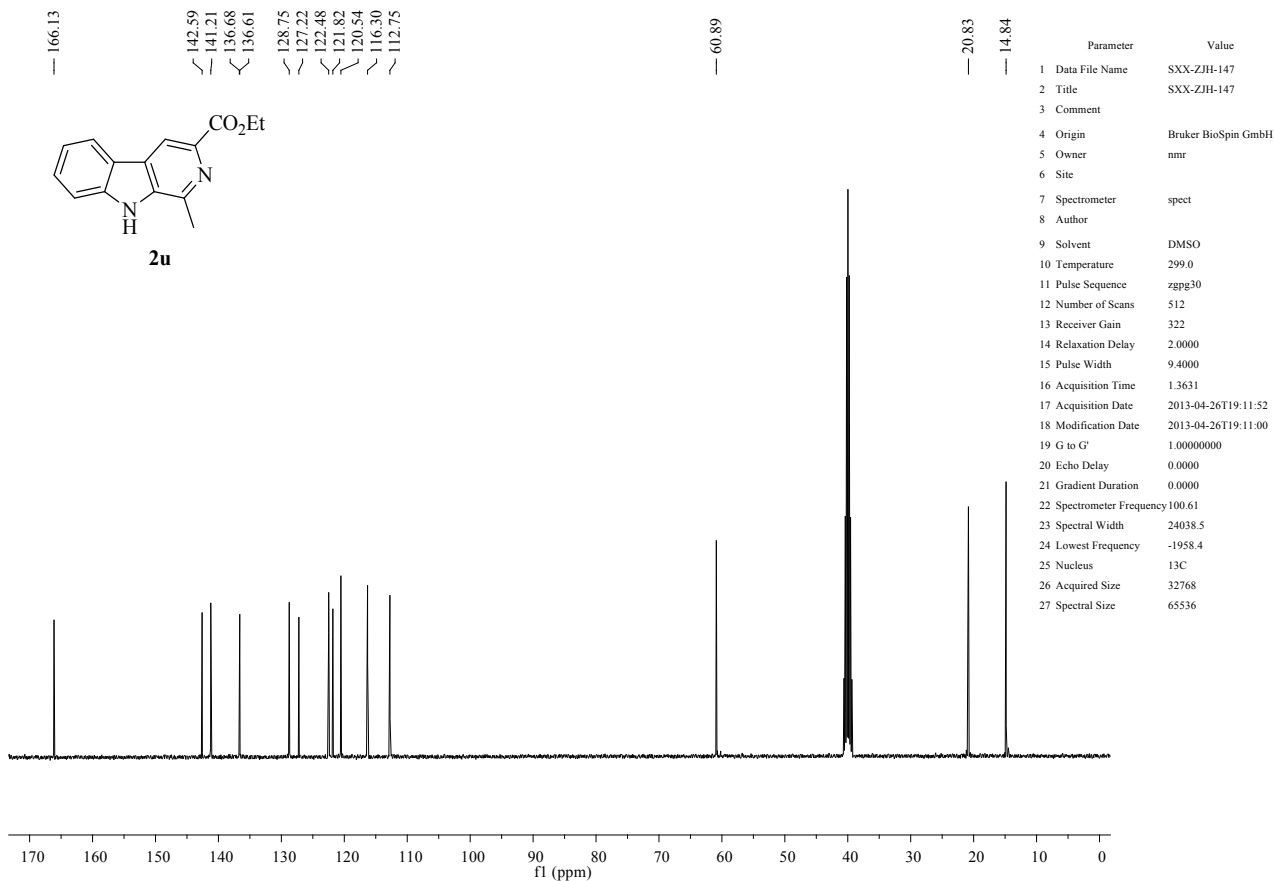
¹³C NMR (DMSO-*d*₆, 100 MHz) spectrum of compound **2t**:



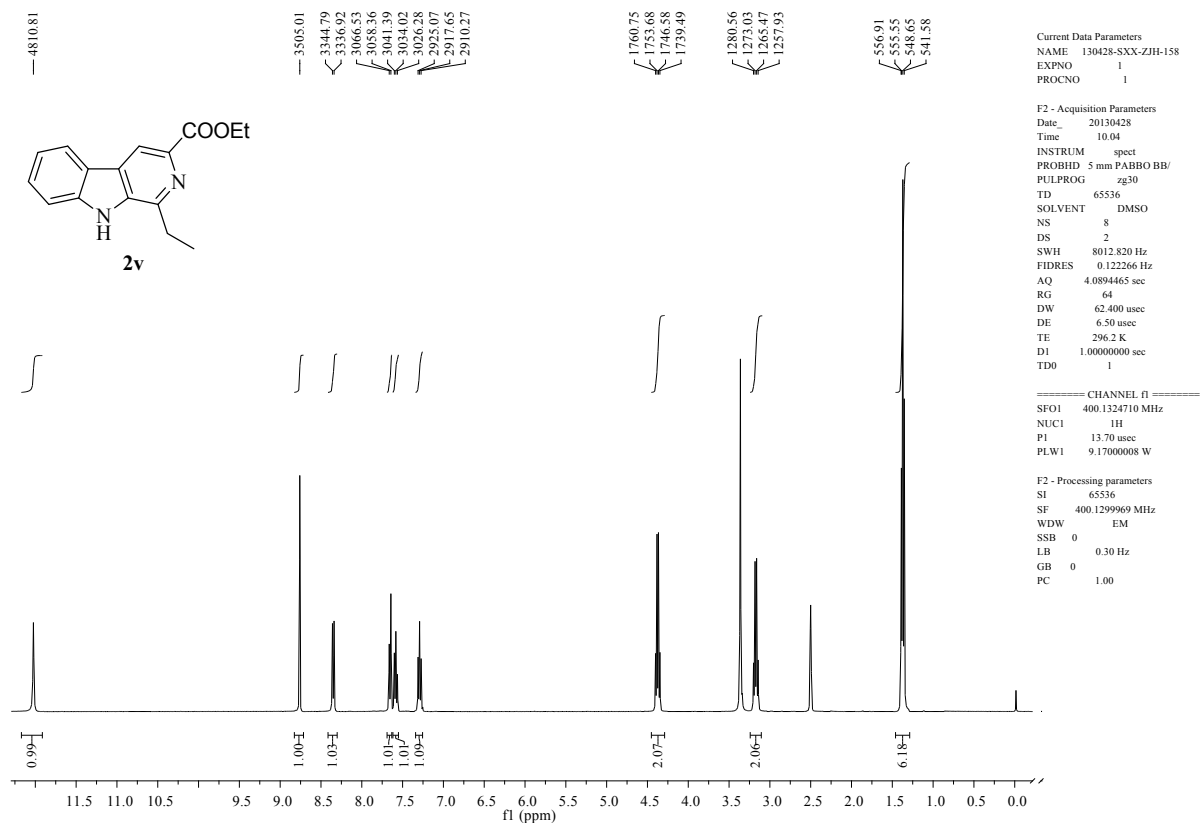
¹H NMR (DMSO-*d*₆, 400 MHz) spectrum of compound **2u**:



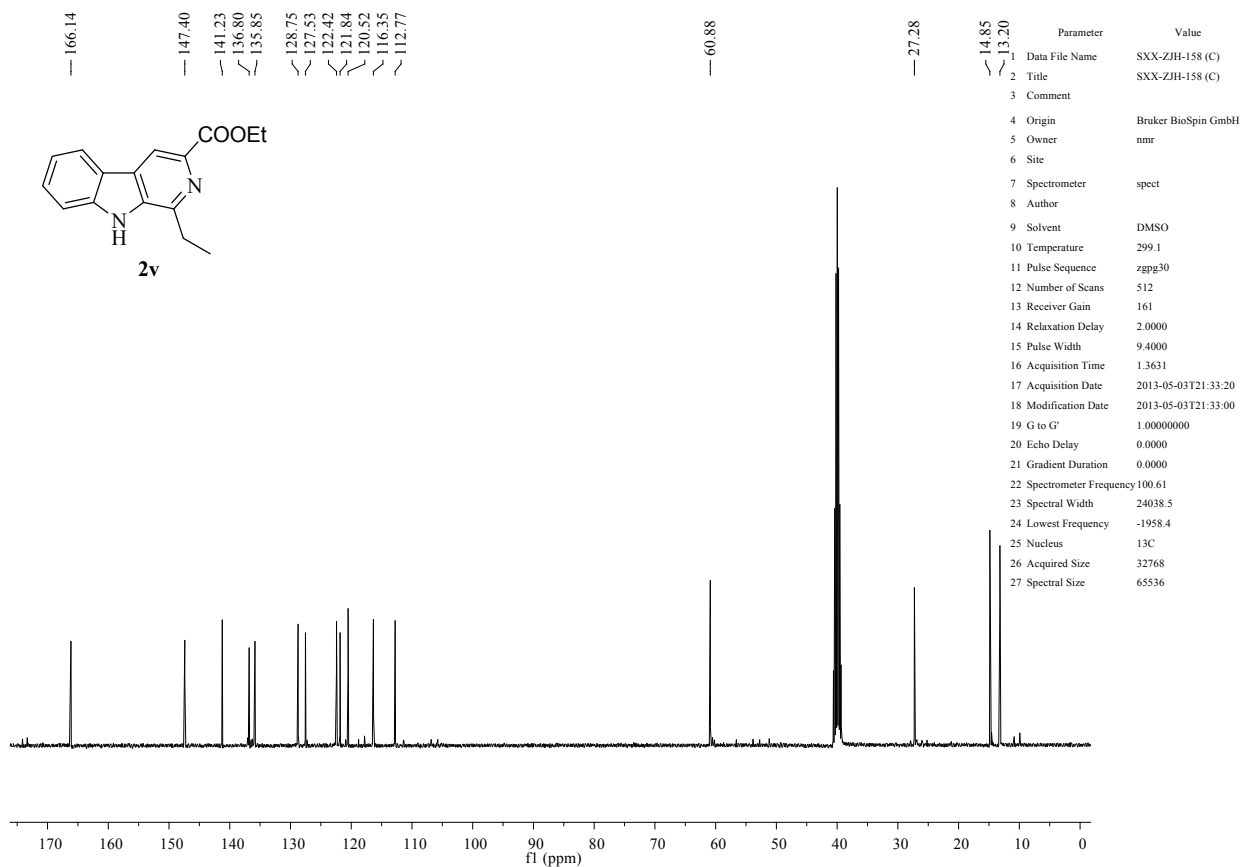
¹³C NMR (DMSO-*d*₆, 100 MHz) spectrum of compound **2u**:



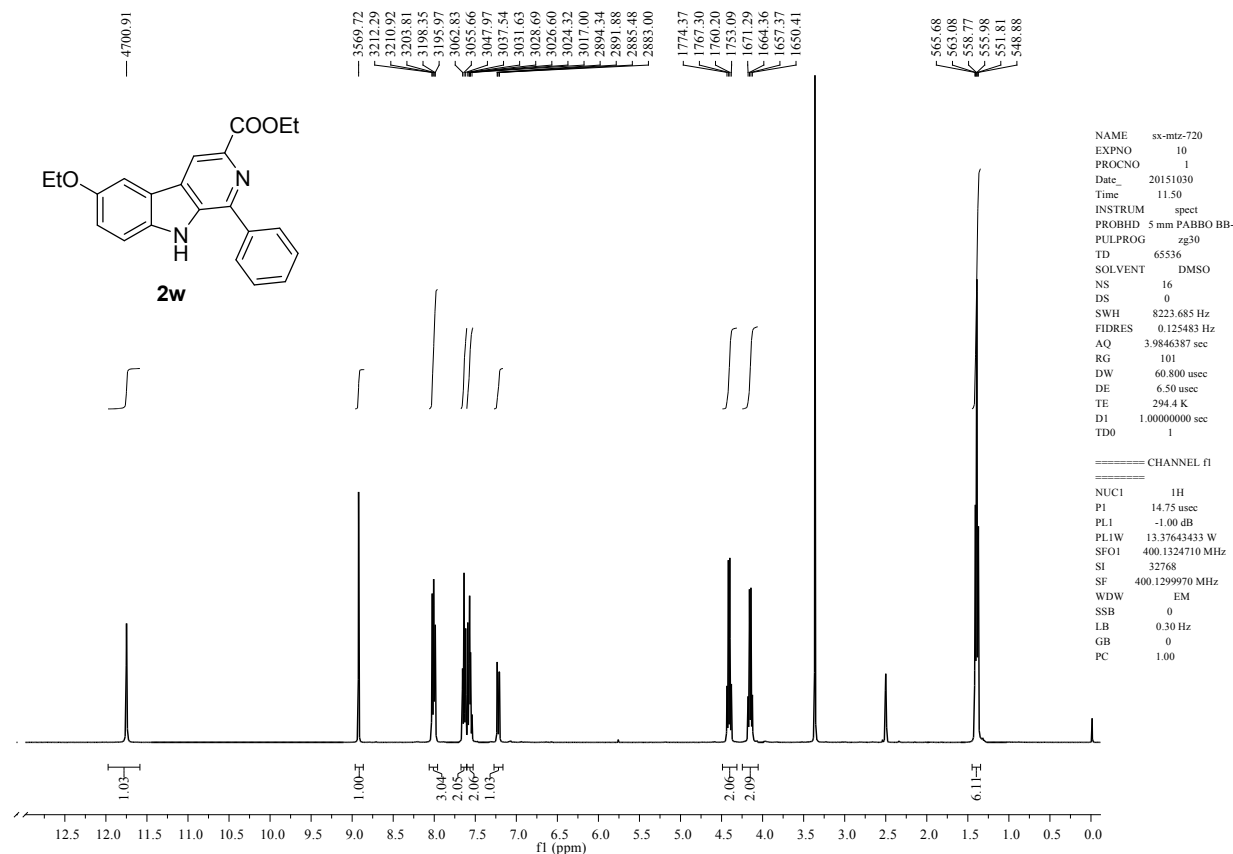
¹H NMR (DMSO-*d*₆, 400 MHz) spectrum of compound **2v**:



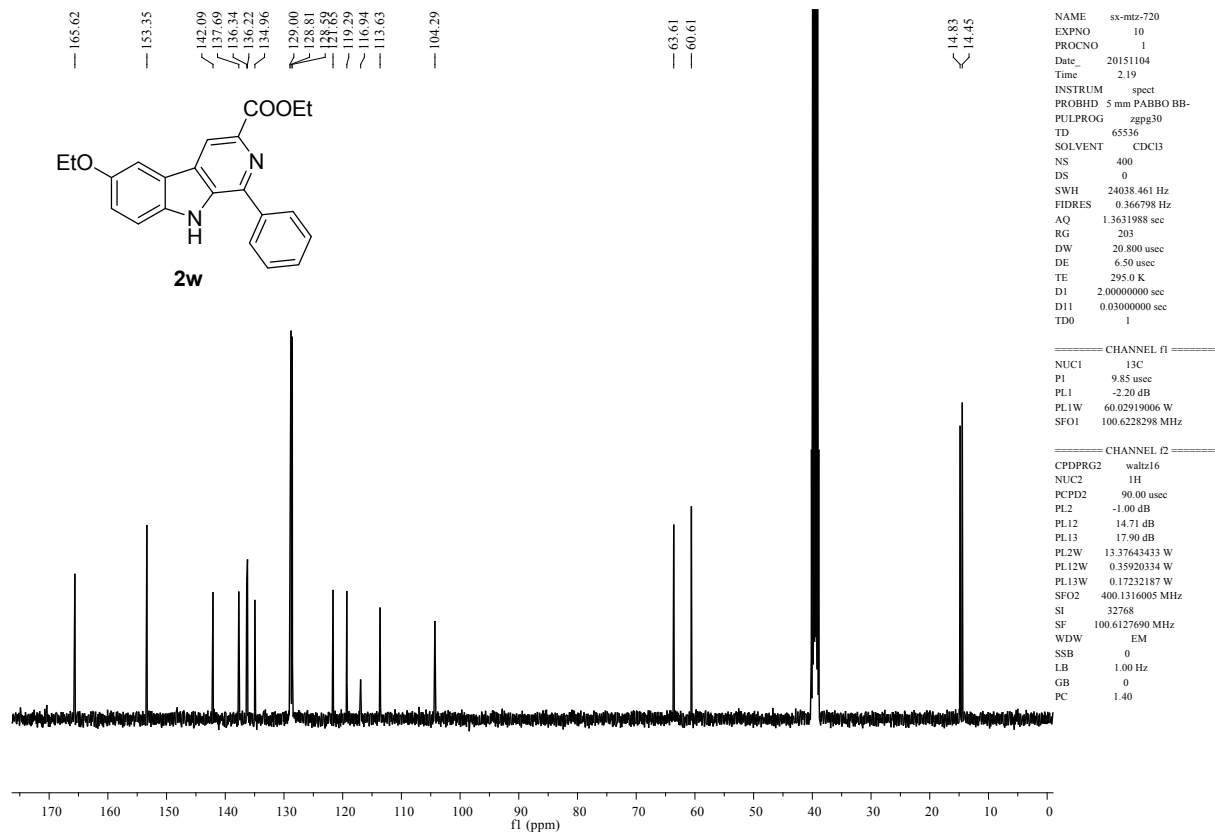
¹³C NMR (DMSO-*d*₆, 100 MHz) spectrum of compound **2v**:



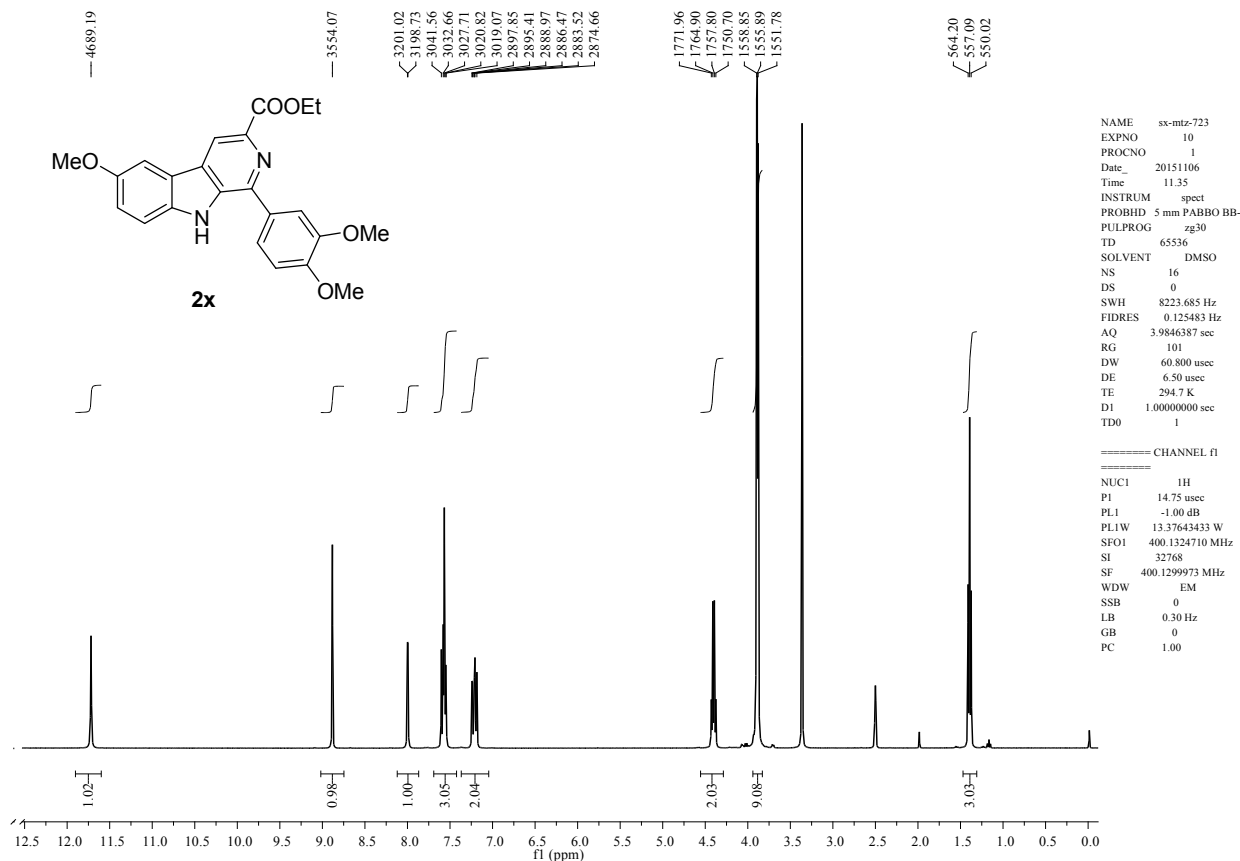
¹H NMR (DMSO-*d*₆, 400 MHz) spectrum of compound **2w**:



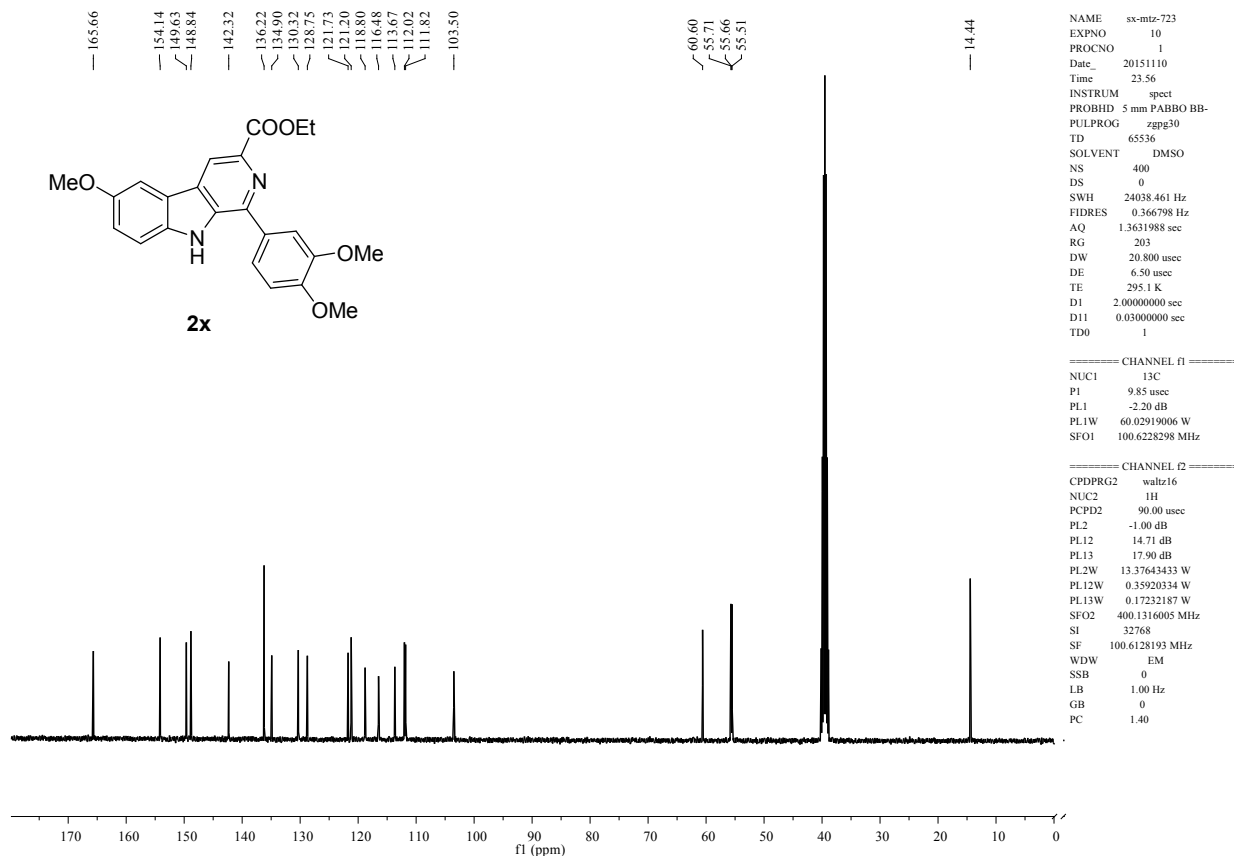
¹³C NMR (DMSO-*d*₆, 100 MHz) spectrum of compound **2w**:



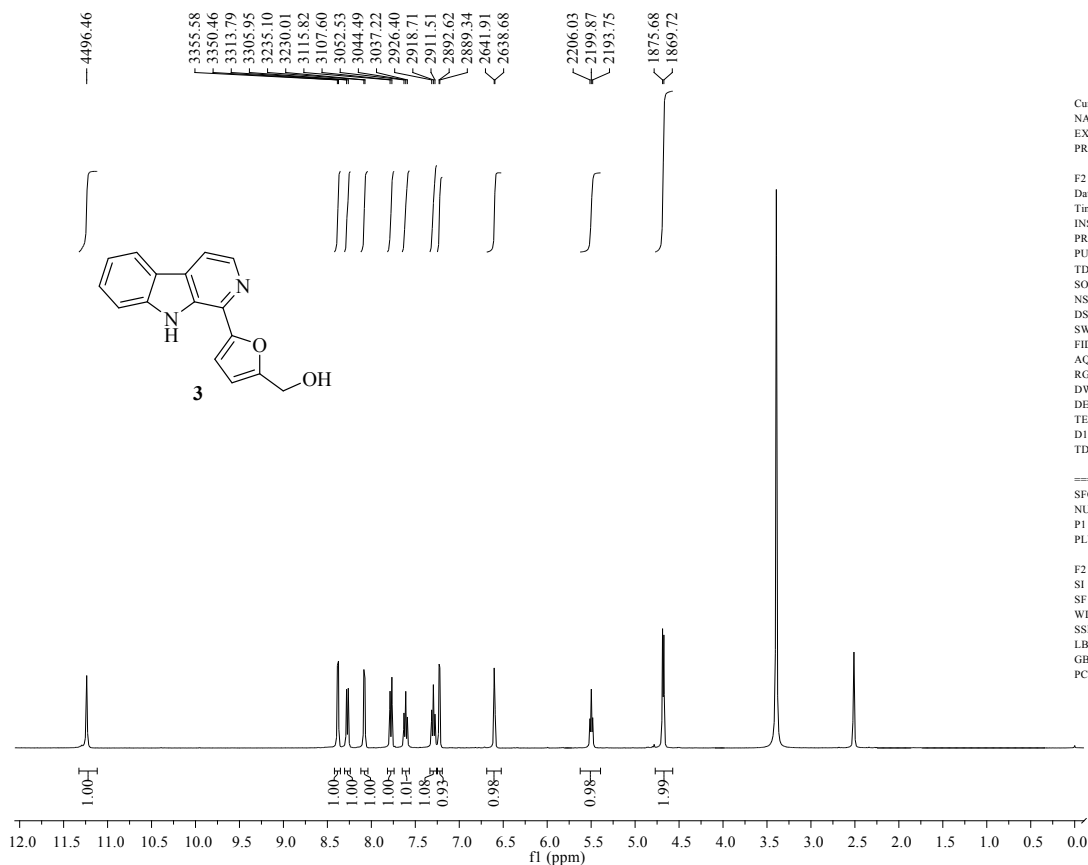
¹H NMR (DMSO-d₆, 400 MHz) spectrum of compound **2x**:



¹³C NMR (DMSO-d₆, 100 MHz) spectrum of compound **2x**:



¹H NMR (DMSO-*d*₆, 400 MHz) spectrum of perlolyrine **3**:



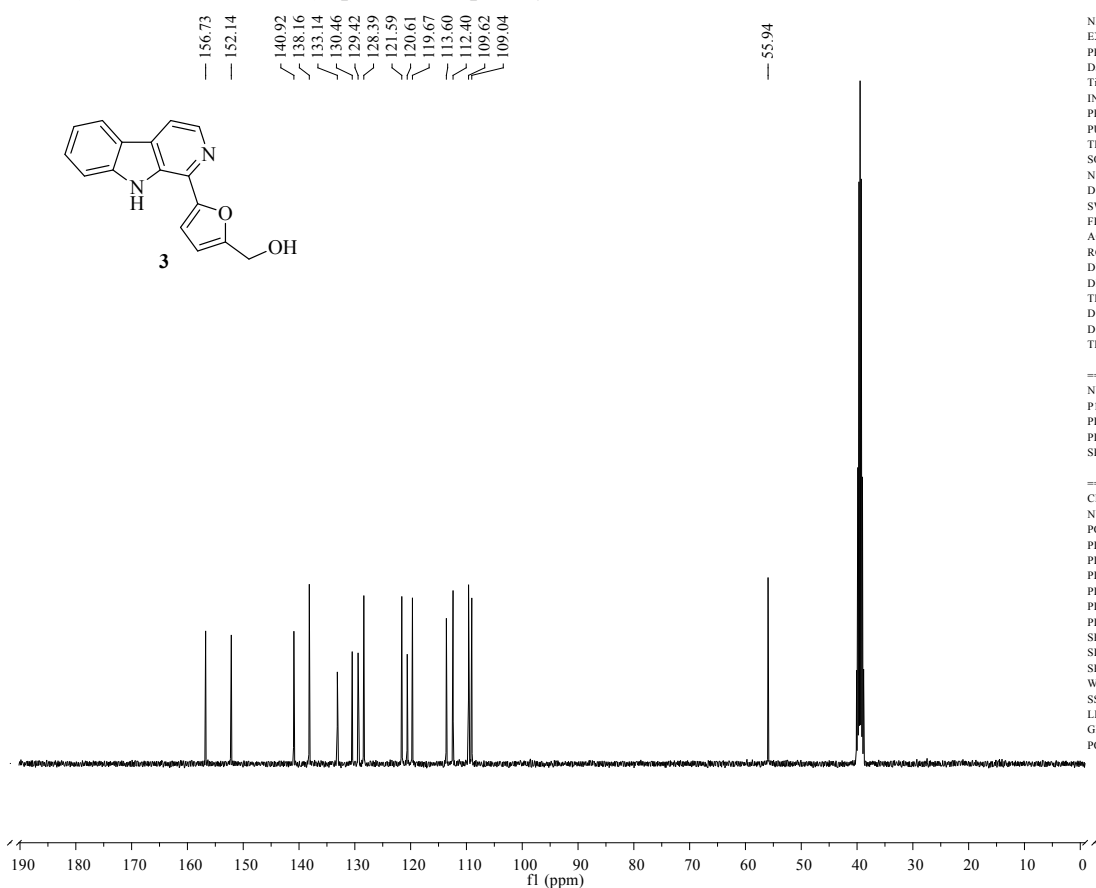
Current Data Parameters
 NAME SXX-ZB-302
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20130909
 Time 16.33
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 8
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 80.6
 DW 62.400 usec
 DE 6.50 usec
 TE 294.0 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 13.70 usec
 PLW1 9.17000008 W

F2 - Processing parameters
 SI 65536
 SF 400.1299987 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

¹³C NMR (DMSO-*d*₆, 100 MHz) spectrum of perlolyrine **3**:

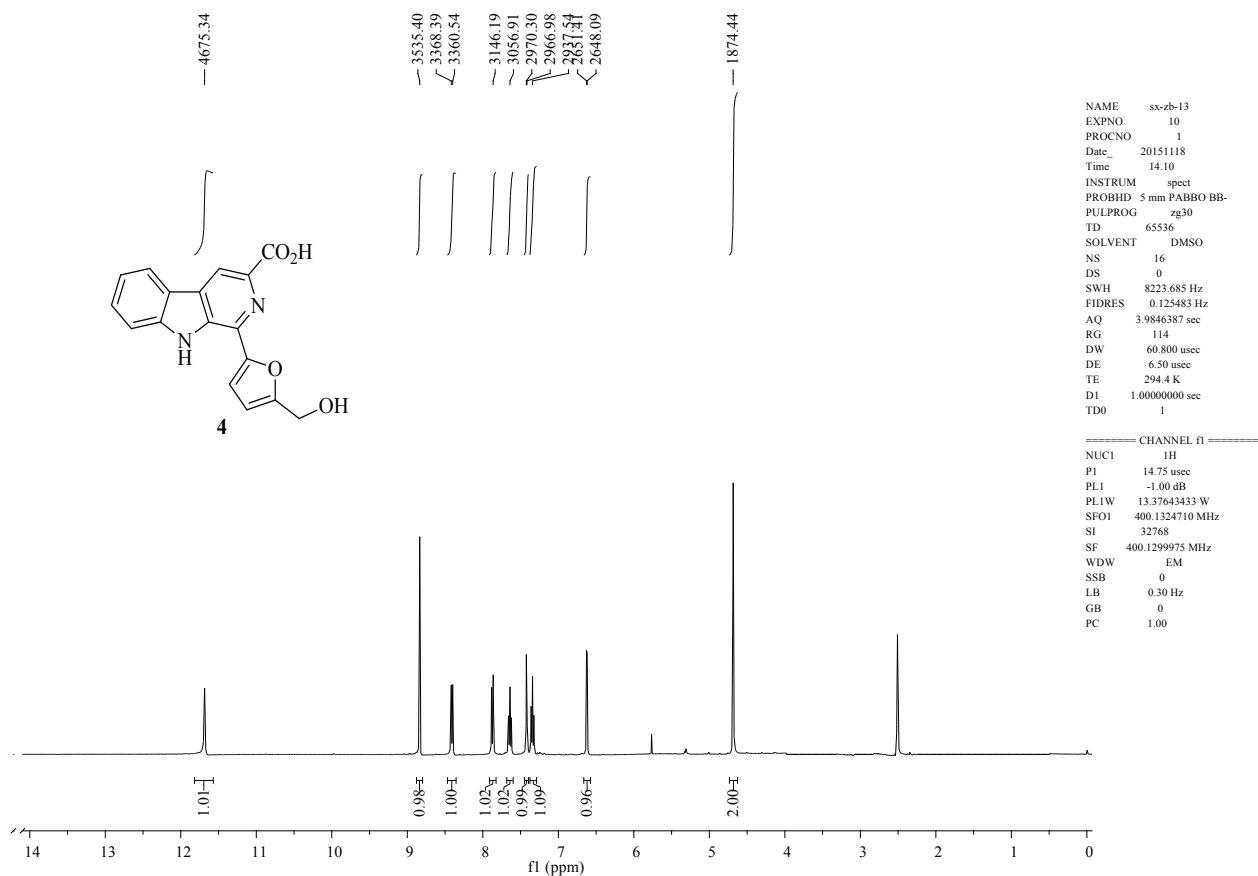


NAME sx-zb-302
 EXPNO 10
 PROCNO 1
 Date_ 20130916
 Time 21.10
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 400
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 203
 DW 20.800 usec
 DE 6.50 usec
 TE 295.9 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

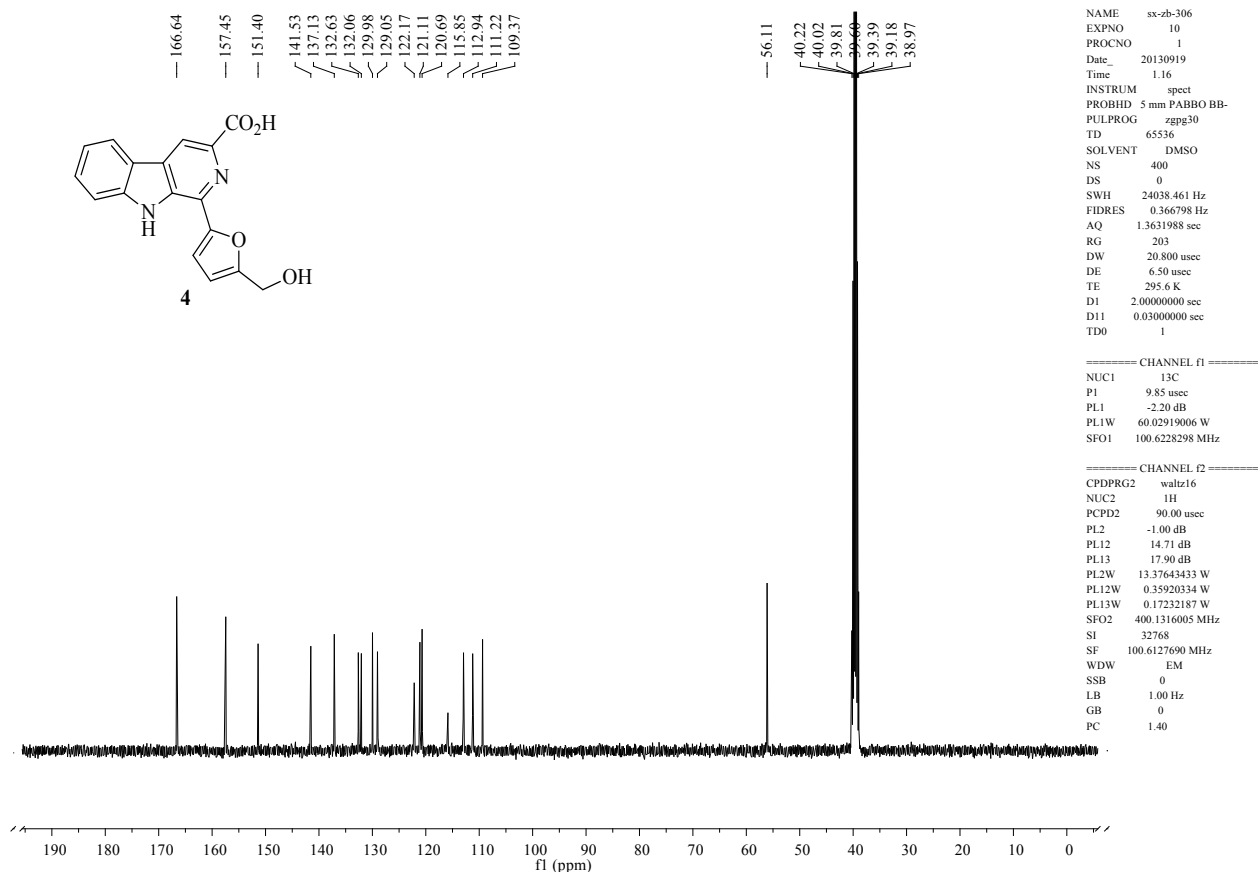
==== CHANNEL f1 =====
 NUC1 13C
 P1 9.85 usec
 PL1 -2.20 dB
 PL1W 60.02919006 W
 SFO1 100.6228298 MHz

==== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 -1.00 dB
 PL12 14.71 dB
 PL13 17.90 dB
 PL2W 13.37643433 W
 PL12W 0.35920334 W
 PL13W 0.17232187 W
 SFO2 400.1316005 MHz
 SI 32768
 SF 100.6128193 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

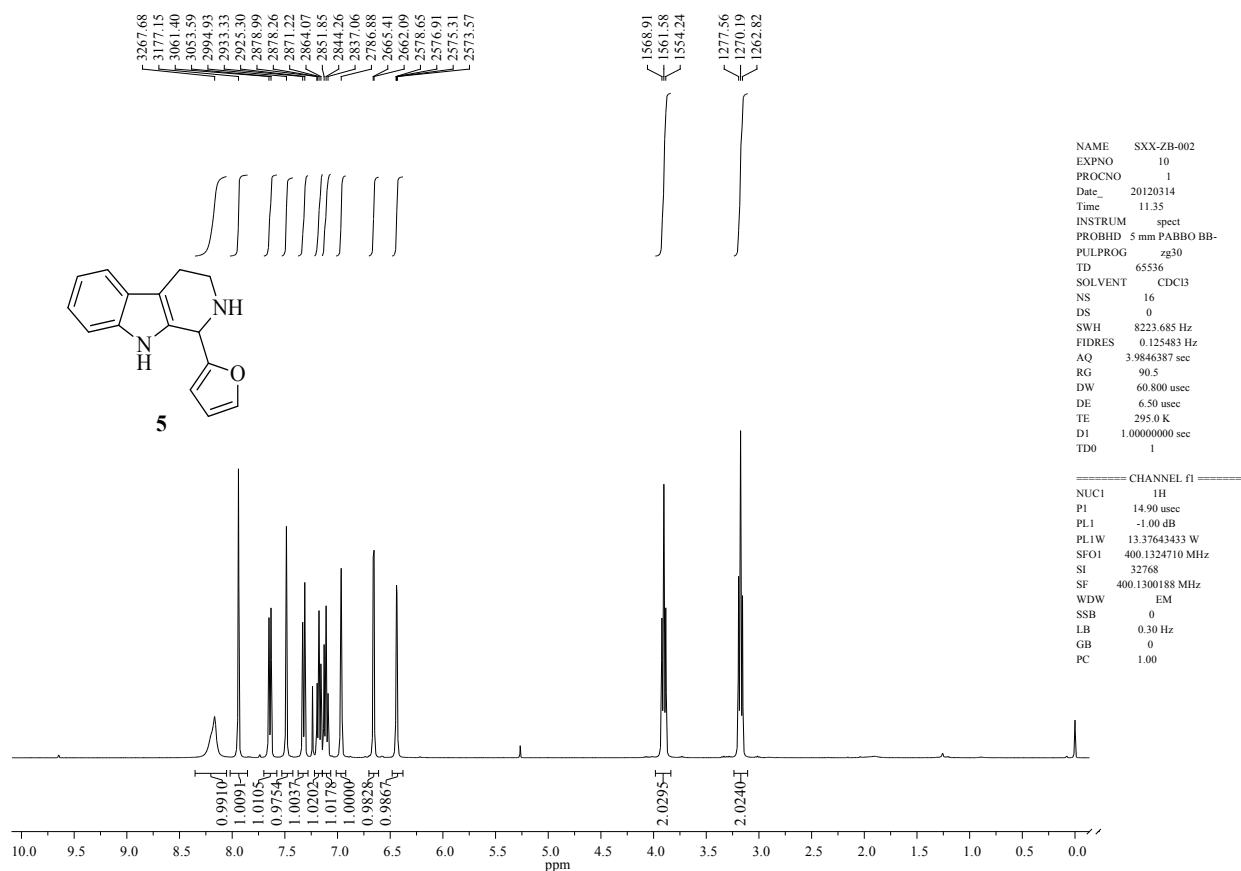
¹H NMR (DMSO-*d*₆, 400 MHz) spectrum of flazin 4:



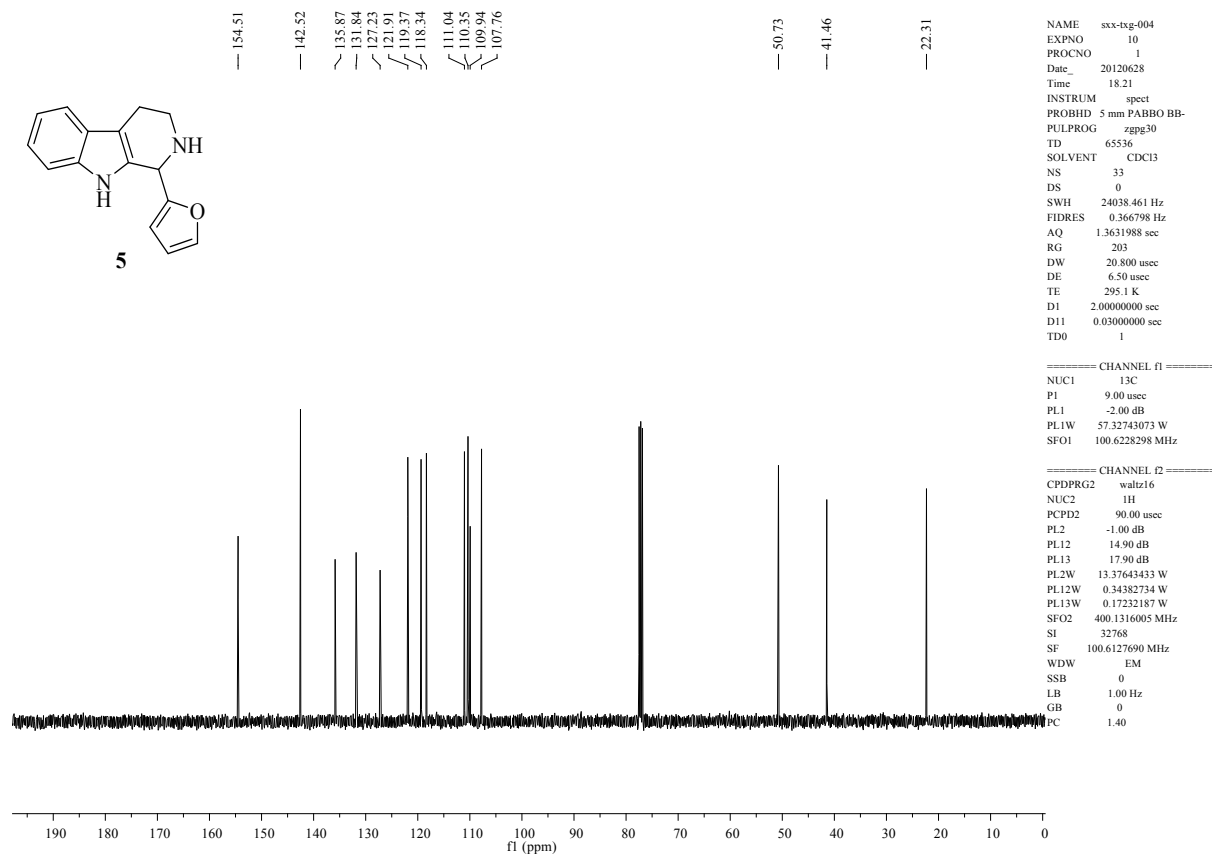
¹³C NMR (DMSO-*d*₆, 100 MHz) spectrum of flazin 4:



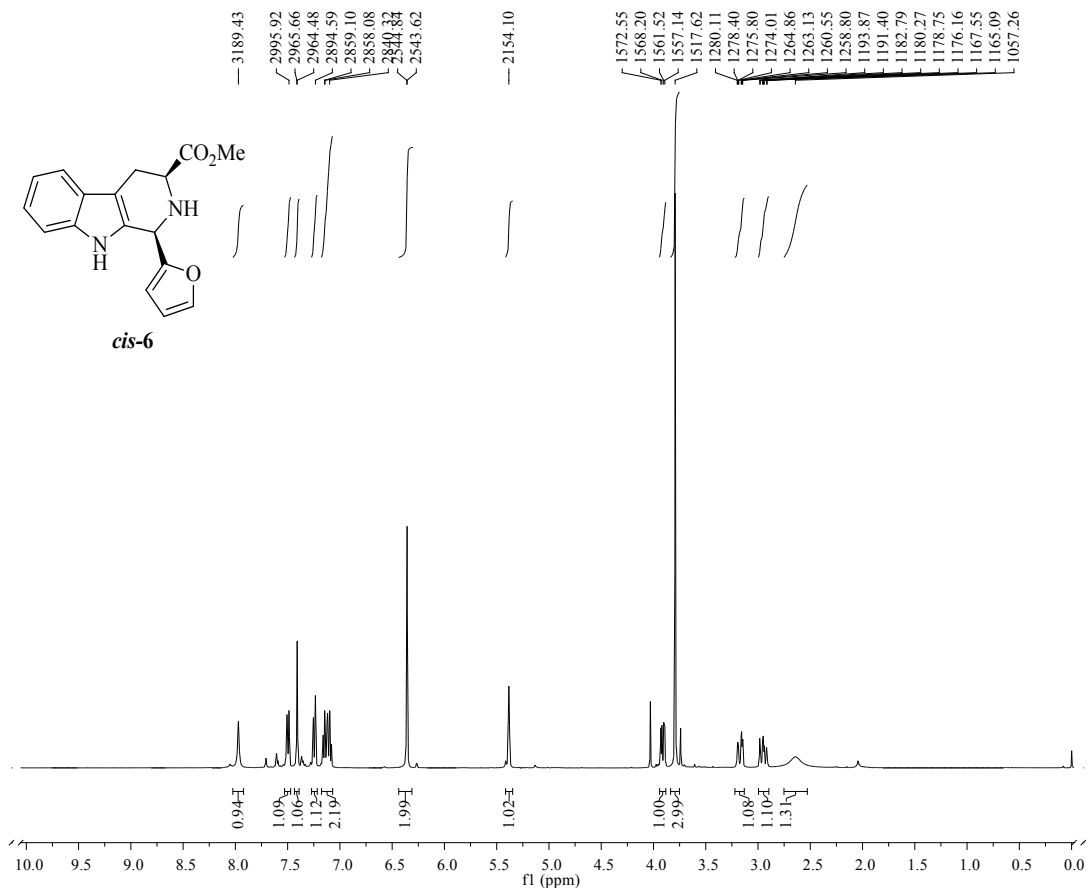
¹H NMR (CDCl₃, 400 MHz) spectrum of compound **5**:



¹³C NMR (CDCl₃, 100 MHz) spectrum of compound **9a**:



¹H NMR (CDCl₃, 400 MHz) spectrum of compound *cis-6*:



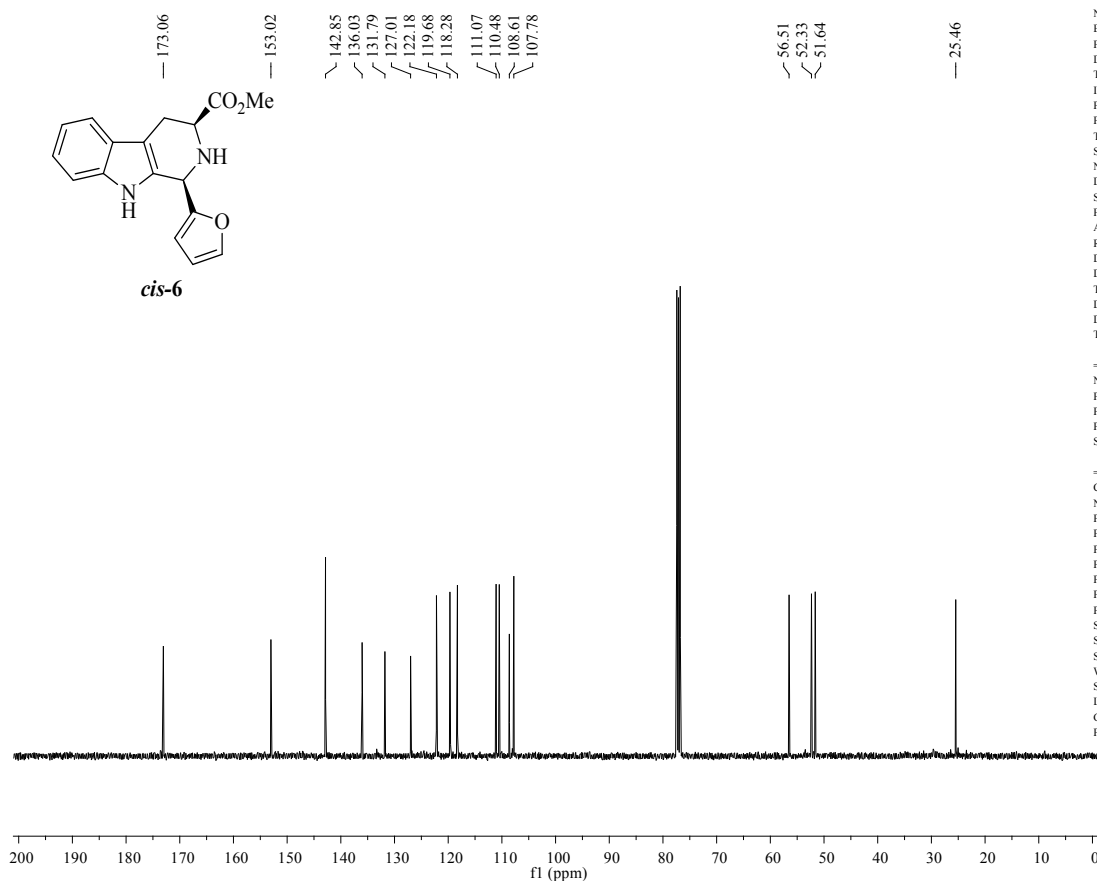
```

NAME      sx-zb-041
EXPNO    10
PROCNO   1
Date_    20151120
Time     7.05
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  zg30
TD        65536
SOLVENT  CDCl3
NS        16
DS        0
SWH       8223.685 Hz
FIDRES   0.125483 Hz
AQ        3.9846387 sec
RG        71.8
DW        60.800 usec
DE        6.50 usec
TE        294.7 K
D1        1.00000000 sec
TD0       1
    
```

```

===== CHANNEL f1 =====
NUC1      1H
P1        14.75 usec
PL1       -1.00 dB
PL1W     13.37643433 W
SFO1     400.1324710 MHz
SI        32768
SF        400.1300178 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
    
```

¹³C NMR (CDCl₃, 100 MHz) spectrum of compound *cis-6*:



```

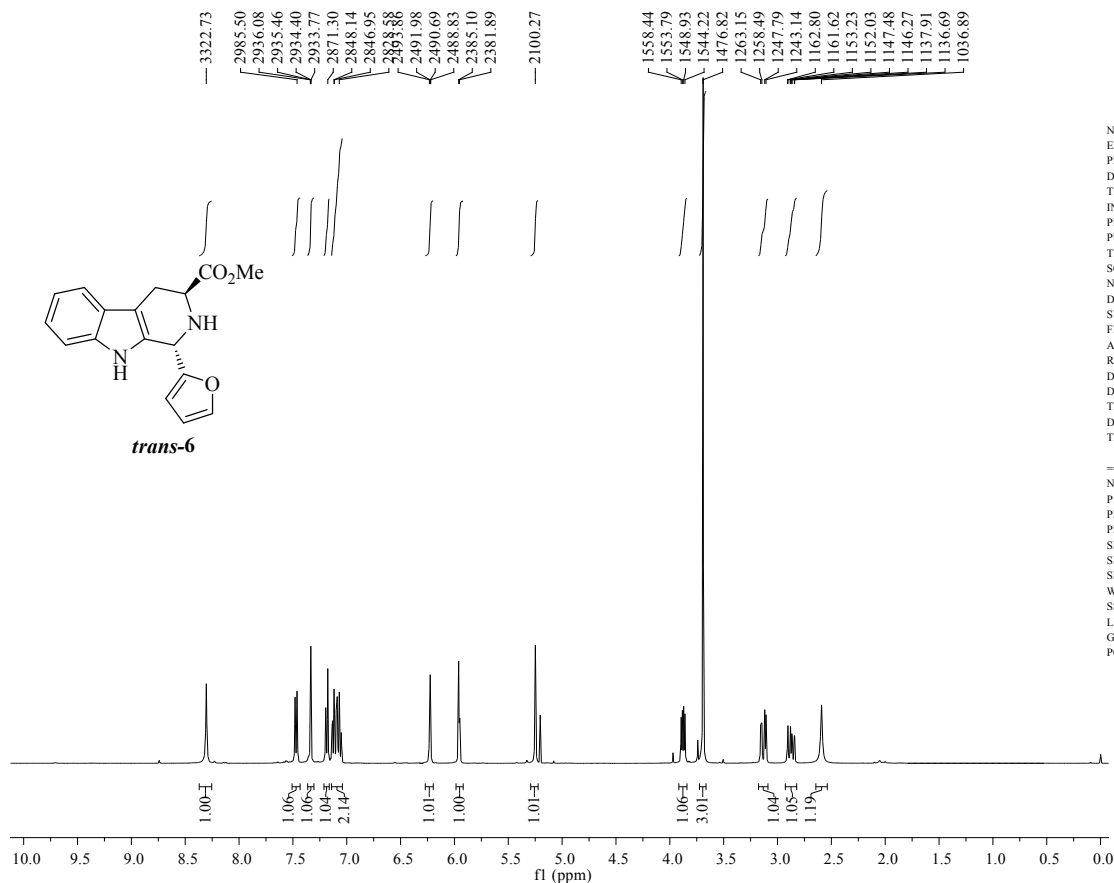
NAME      sx-zb-041
EXPNO    11
PROCNO   1
Date_    20151120
Time     7.30
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        400
DS        0
SWH       24038.461 Hz
FIDRES   0.366798 Hz
AQ        1.3631988 sec
RG        203
DW        20.800 usec
DE        6.50 usec
TE        295.5 K
D1        2.00000000 sec
D11      0.03000000 sec
TD0       1
    
```

```

===== CHANNEL f1 =====
NUC1      13C
P1        9.85 usec
PL1       -2.20 dB
PL1W     60.02919006 W
SFO1     100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2      1H
PCPD2    90.00 usec
PL2       -1.00 dB
PL12     14.71 dB
PL13     17.90 dB
PL2W     13.37643433 W
PL12W    0.35920334 W
PL13W    0.17232187 W
SFO2     400.1316005 MHz
SI        32768
SF        100.6127690 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
    
```

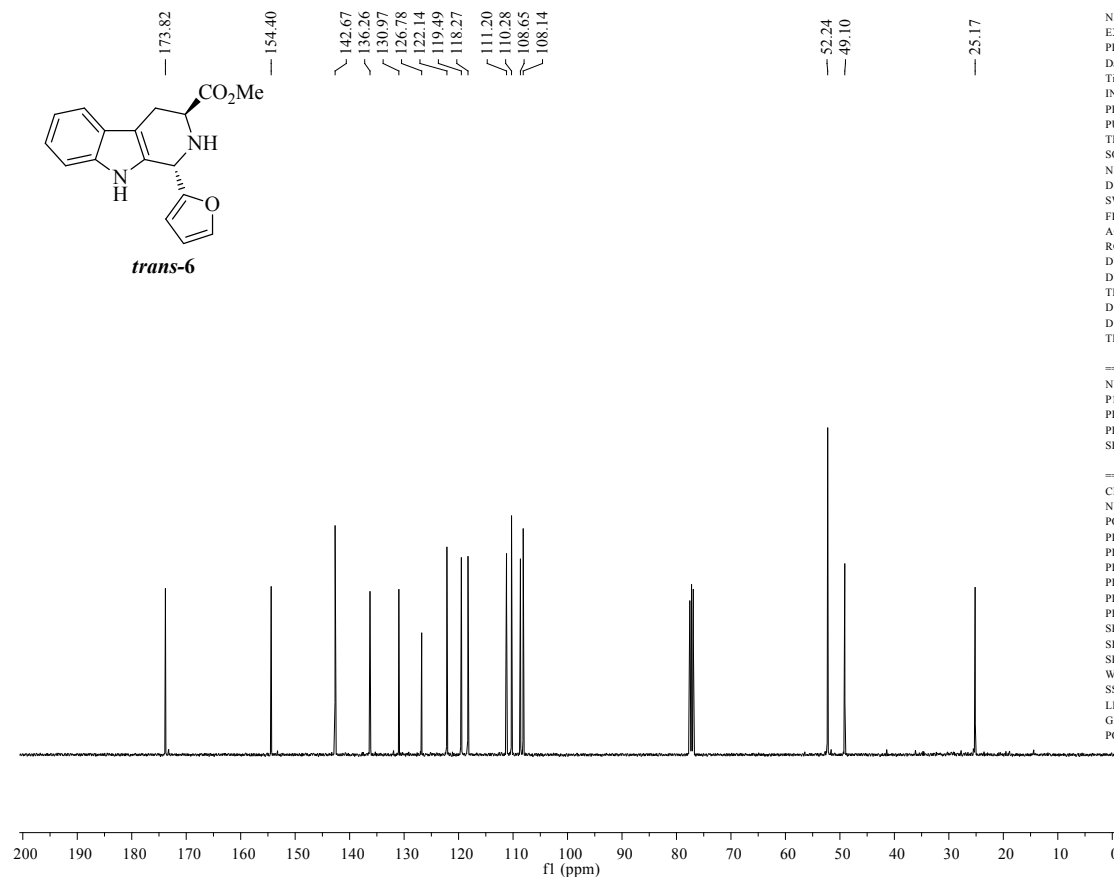
¹H NMR (CDCl₃, 400 MHz) spectrum of compound *trans*-6:



NAME sx-zh-042
 EXPNO 10
 PROCNO 1
 Date_ 20151120
 Time 7.37
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 32
 DW 60.800 usec
 DE 6.50 usec
 TE 294.8 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 14.75 usec
 PL1 -1.00 dB
 PL1W 13.37643433 W
 SFO1 400.1324710 MHz
 SI 32768
 SF 400.1300376 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

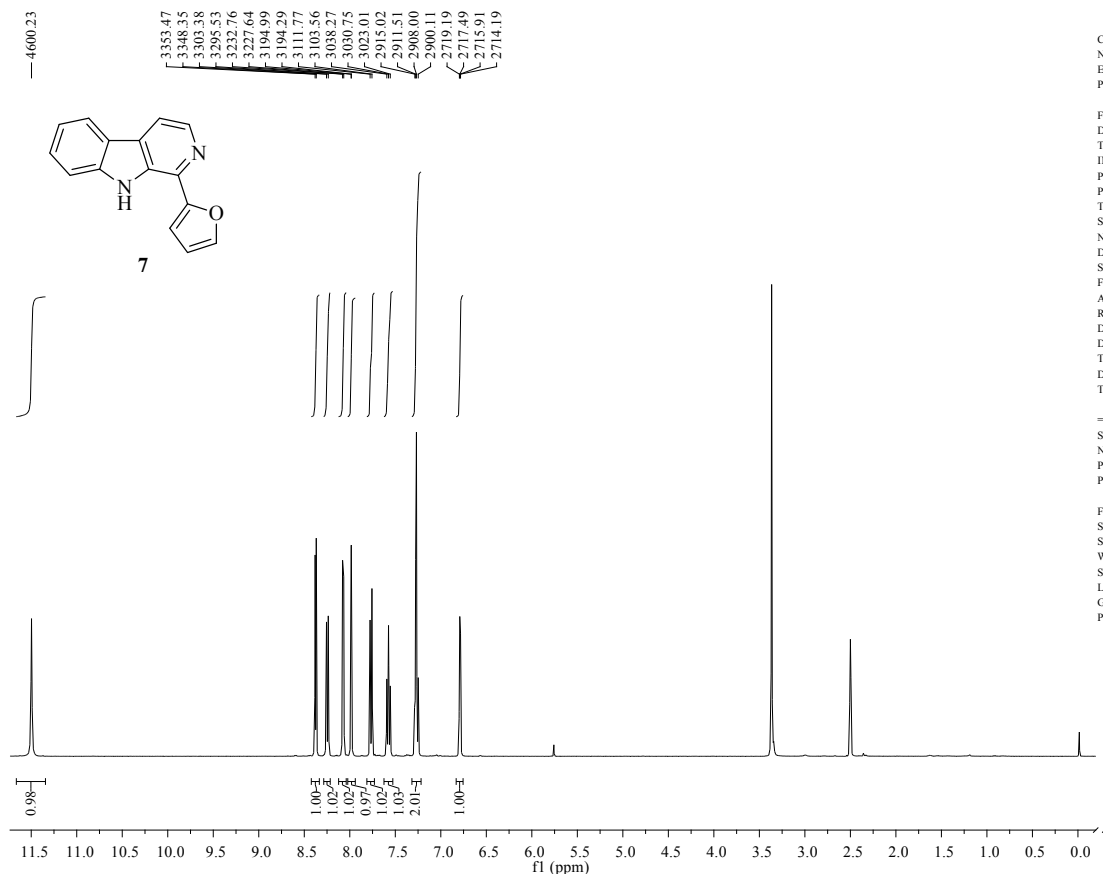
¹³C NMR (CDCl₃, 100 MHz) spectrum of compound *trans*-6:



NAME sx-zh-042
 EXPNO 11
 PROCNO 1
 Date_ 20151120
 Time 8.01
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 400
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 203
 DW 20.800 usec
 DE 6.50 usec
 TE 295.5 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.85 usec
 PL1 -2.20 dB
 PL1W 60.02919006 W
 SFO1 100.6228298 MHz
 ===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 -1.00 dB
 PL12 14.71 dB
 PL13 17.90 dB
 PL2W 13.37643433 W
 PL12W 0.35920334 W
 PL13W 0.17232187 W
 SFO2 400.1316005 MHz
 SI 32768
 SF 100.6127690 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

¹H NMR (DMSO-d₆, 400 MHz) spectrum of compound 7:



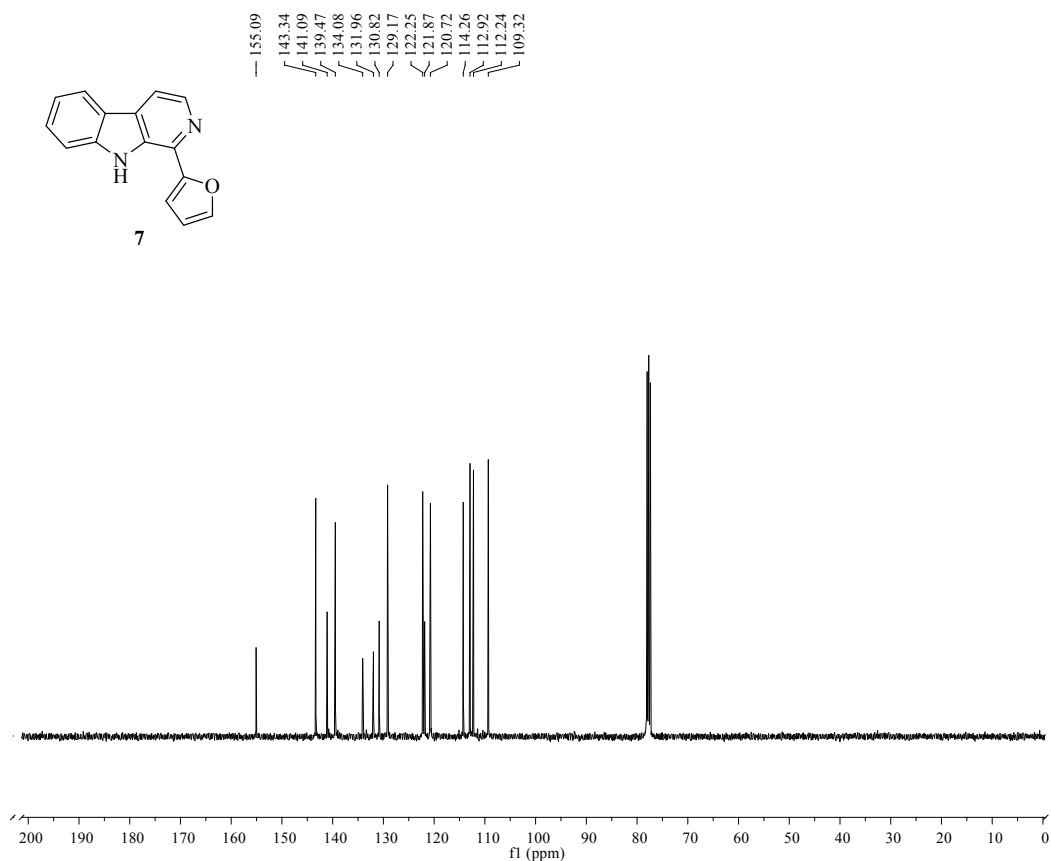
Current Data Parameters
 NAME 130424-SXX-ZJH-150
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20130424
 Time 14.07
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 8
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 80.6
 DW 62.400 usec
 DE 6.50 usec
 TE 296.0 K
 D1 1.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 13.70 usec
 PLW1 9.17000008 W

F2 - Processing parameters
 SI 65536
 SF 400.1299973 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

¹³C NMR (CDCl₃, 100 MHz) spectrum of compound 7:



Current Data Parameters
 NAME 0629-SXX-TXG-005
 EXPNO 2
 PROCNO 1

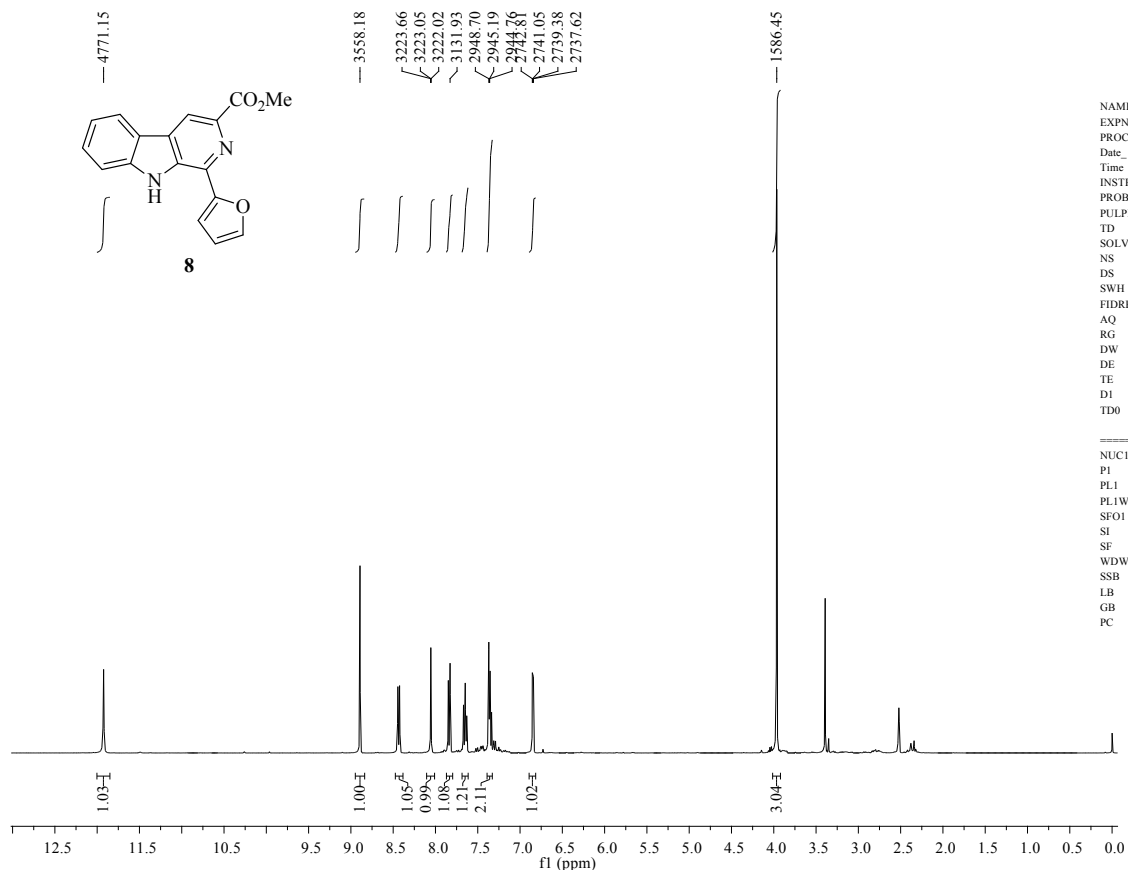
F2 - Acquisition Parameters
 Date_ 20120629
 Time 14.43
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 512
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 203
 DW 20.800 usec
 DE 6.50 usec
 TE 296.5 K
 D1 2.0000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 100.6228293 MHz
 NUC1 13C
 P1 9.00 usec
 PLW1 56.42200089 W

===== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 9.17000008 W
 PLW12 0.21247999 W
 PLW13 0.17211001 W

F2 - Processing parameters
 SI 32768
 SF 100.6127690 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

¹H NMR (DMSO-*d*₆, 400 MHz) spectrum of compound **8**:



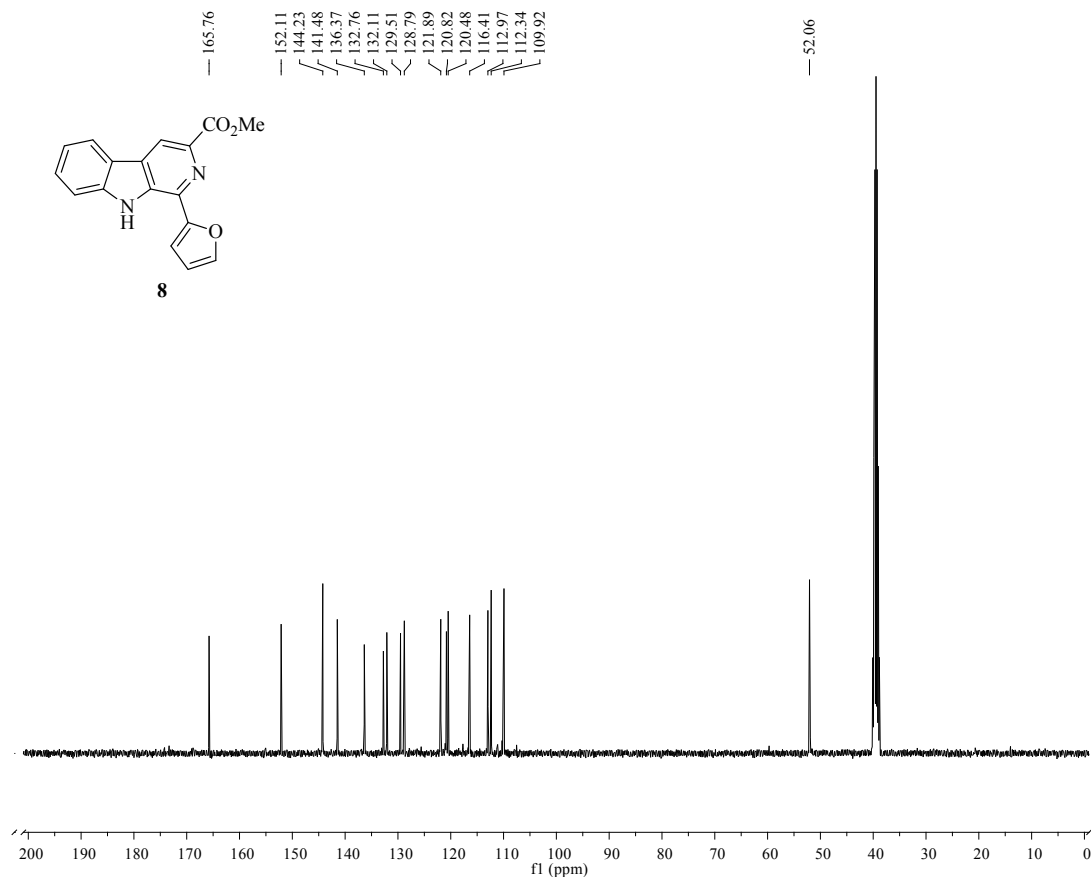
```

NAME      sxx-zb-015
EXPNO     10
PROCNO    1
Date_     20120424
Time      13.06
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   DMSO
NS         16
DS         0
SWH        8223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         114
DW         60.800 usec
DE         6.50 usec
TE         294.3 K
D1         1.00000000 sec
TD0        1
    
```

```

===== CHANNEL f1 =====
NUC1       1H
P1         14.90 usec
PL1        -1.00 dB
PL1W       13.37643433 W
SFO1       400.1324710 MHz
SI         32768
SF         400.1299947 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
    
```

¹³C NMR (DMSO-*d*₆, 100 MHz) spectrum of compound **8**:



```

NAME      sx-zb-305
EXPNO     10
PROCNO    1
Date_     20130916
Time      22.09
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   DMSO
NS         400
DS         0
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631988 sec
RG         203
DW         20.800 usec
DE         6.50 usec
TE         295.7 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
    
```

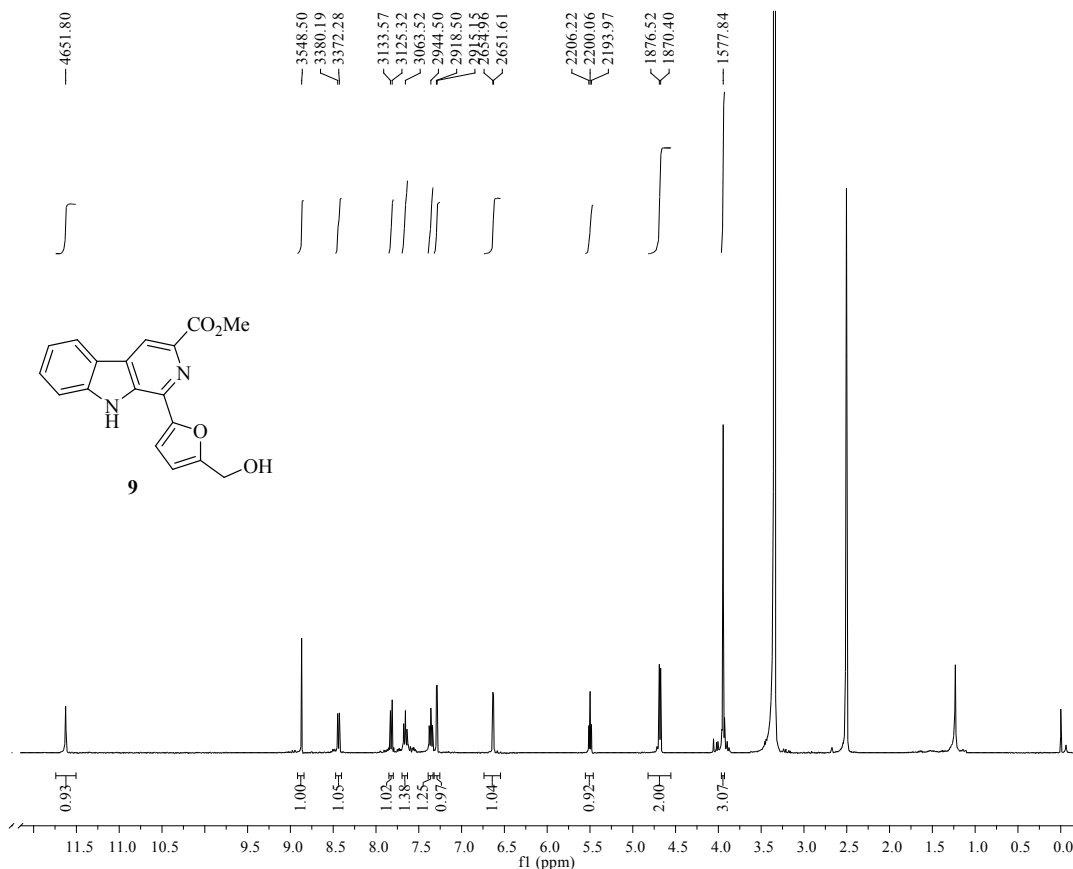
```

===== CHANNEL f1 =====
NUC1       13C
P1         9.85 usec
PL1        -2.20 dB
PL1W       60.02919006 W
SFO1       100.6228298 MHz
    
```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2     90.00 usec
PL2        -1.00 dB
PL12      14.71 dB
PL13      17.90 dB
PL2W      13.37643433 W
PL12W     0.35920334 W
PL13W     0.17232187 W
SFO2      400.1316005 MHz
SI         32768
SF         100.6128193 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
    
```

¹H NMR (DMSO-*d*₆, 400 MHz) spectrum of compound **9**:



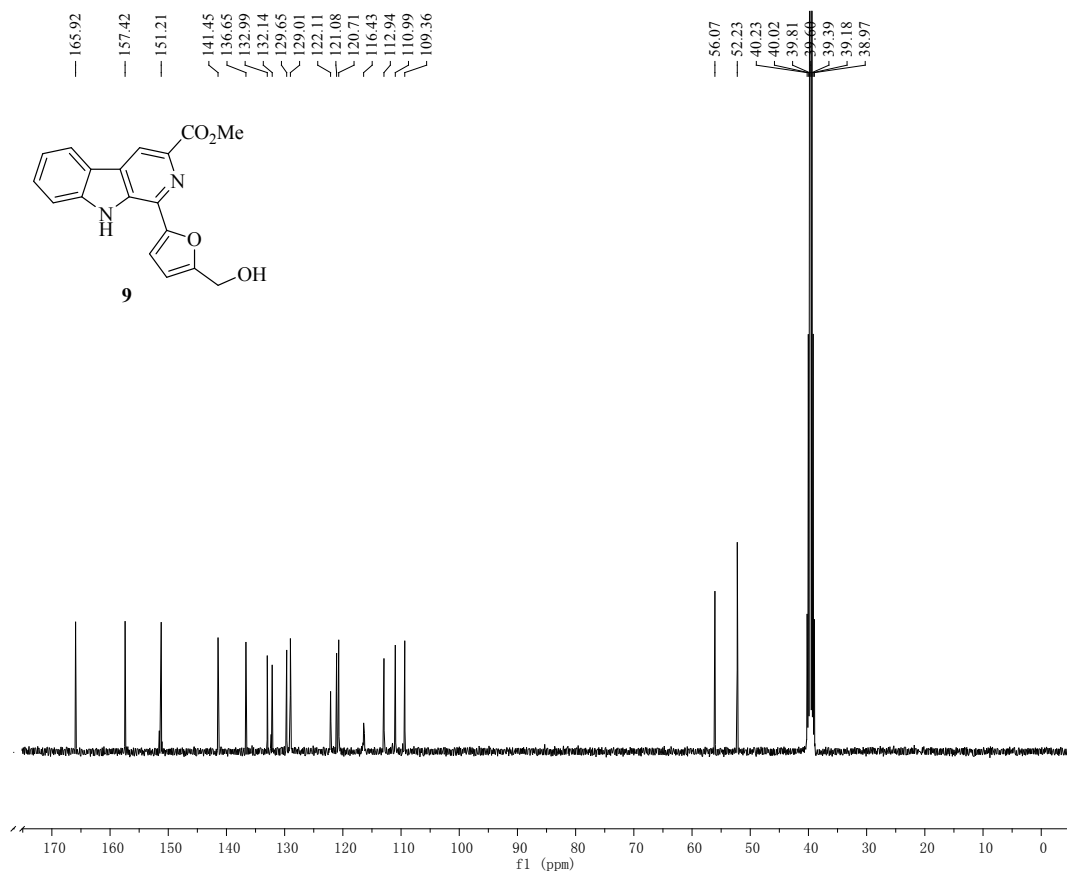
```

NAME      sxx-zh-017
EXPNO    10
PROCNO   1
Date_    20120424
Time     14.47
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  zg30
TD       65536
SOLVENT  DMSO
NS       16
DS       0
SWH      8223.685 Hz
FIDRES   0.125483 Hz
AQ       3.9846387 sec
RG       203
DW       60.800 usec
DE       6.50 usec
TE       294.9 K
D1       1.00000000 sec
TD0      1
    
```

```

===== CHANNEL f1 =====
NUC1     1H
P1       14.90 usec
PL1      -1.00 dB
PL1W     13.37643433 W
SFO1     400.1324710 MHz
SI       32768
SF       400.1300017 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
    
```

¹³C NMR (DMSO-*d*₆, 100 MHz) spectrum of compound **9**:



```

NAME      sxx-zh-017C
EXPNO    10
PROCNO   1
Date_    20130919
Time     1.44
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD       65536
SOLVENT  DMSO
NS       400
DS       0
SWH      24038.461 Hz
FIDRES   0.366798 Hz
AQ       1.3631988 sec
RG       203
DW       20.800 usec
DE       6.50 usec
TE       295.4 K
D1       2.00000000 sec
D11      0.03000000 sec
TD0      1
    
```

```

===== CHANNEL f1 =====
NUC1     13C
P1       9.85 usec
PL1      -2.20 dB
PL1W     60.02919006 W
SFO1     100.6228298 MHz
    
```

```

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2    90.00 usec
PL2      -1.00 dB
PL12     14.71 dB
PL13     17.90 dB
PL2W     13.37643433 W
PL12W    0.35920334 W
PL13W    0.17232187 W
SFO2     400.1316005 MHz
SI       32768
SF       100.6127690 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
    
```