Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

Copper(II)-mediated regioselective *N*-arylation of pyrroles, indoles, pyrazoles and carbazole *via* dehydrogenative coupling

Pradeep Sadhu and Tharmalingam Punniyamurthy*

Department of Chemistry, Indian Institute of Technology Guwahati, Guwahati 781039, India

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General Information: $Cu(OAc)_2$ (98%), Cs_2CO_3, 8-aminoquinoline (98%), pyrazole (98%), 3-methylpyrazole (97%) and 2-phenyl imidazole (98%) were purchased from Aldrich and were used as received. Pyrrole (99%) was purchased from Otto and distilled under nitrogen according to the standard procedure.[1] Indole (99%) was purchased from Otto and used as received. Substituted indoles and pyrroles were purchased from Avra synthesis. The solvents were purchased and dried according to standard procedure prior to use.[2] Substituted 8-aminoquinoline amides were prepared according to reported procedure.[3] 1-(2-Methyl-4-phenyl-1H-pyrrol-3-yl)ethan-1-one[4] and 2-(2-(1H-indol-3-yl)ethyl)isoindoline-1,3-dione[5] were prepared according to the literature. Purification of the reaction products was carried out by column chromatography using Merck aluminium oxide active, neutral Activity I-II . Analytical TLC was performed on Merck silica gel G/GF 254 plate. NMR spectra were recorded on Bruker Avance III 600 using $CDCl_3$ as solvent and Me_4Si as internal standard. Chemical shifts (δ) were reported in ppm and spin-spin coupling constants (J) were given in Hz. Melting points were determined using Büchi B-540 melting point apparatus and are uncorrected. FT-IR spectra were recorded using Thermo Fisher Scientific spectrometer. Mass spectra were recorded on a Q-Tof ESI-MS Instrument (model HAB 273). X-Ray data were collected on a Bruker SMART APEX equipped with a CCD area detector using Mo/Kα radiation. The structure was solved by direct method using *SHELXL-97* (Göttingen, Germany).

General Procedure for $Cu(OAc)_2$-Mediated *N*-Arylation of Azoles. Azoles (0.6 mmol, 3 equiv) were added to a stirred solution of the substrates **1** (0.2 mmol, 1 equiv), $Cu(OAc)_2$ (0.3 mmol, 1.5 equiv, 54.5 mg), Cs_2CO_3 (0.4 mmol, 2 equiv, 130 mg) and solvent (1 mL) at 70 °C under air. The mixture was stirred and the progress of the reaction was monitored by TLC using ethyl acetate and hexane as eluent. After the appropriate time, the resulting solution was diluted with ethyl acetate (3 x 15 mL) and then washed with $NH_3 \cdot H_2O$ (1 x 5 mL) and brine (2 x 5 mL). Drying over Na_2SO_4 and evaporation of the solvent gave a residue that was purified on neutral alumina column chromatography using n-hexane and ethyl acetate as eluent to afford analytically pure substituted *N*-arylated azoles.

Procedure for the Removal of Directing Group. To a stirred solution of NaOH (1.4 mmol, 7.0 equiv, 56 mg) in EtOH (1.5 mL) was added 2-(1*H*-pyrrol-1-yl)-*N*-(quinolin-8-yl)benzamide **2a** (0.2 mmol, 1

equiv, 62.6 mg). The resultant solution was stirred at room temperature for 2 minutes and heated to 110 °C for 48 h. After completion, the reaction was cooled to room temperature, the resulting solution was diluted with ethyl acetate (4 x 15 mL) and then washed with 0.5 N HCl (4 x 5 mL) and brine (2 x 5 mL). Drying over Na_2SO_4 and evaporation of the solvent gave a pure product as a pale brown solid.

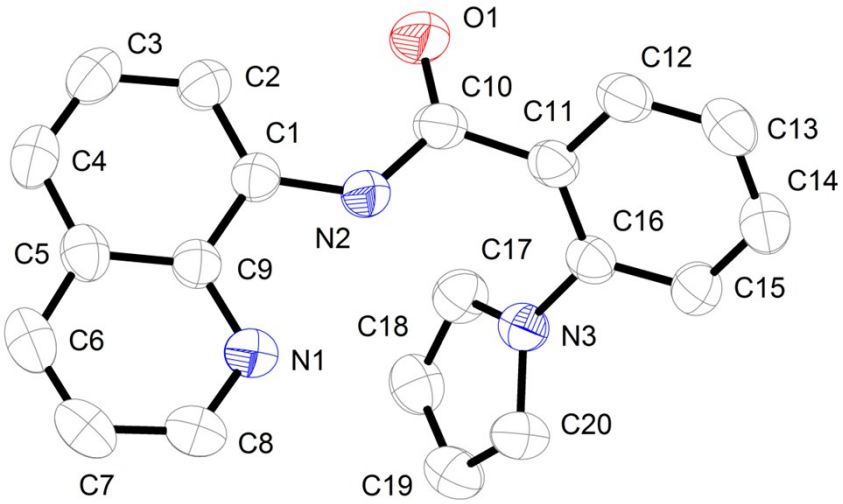
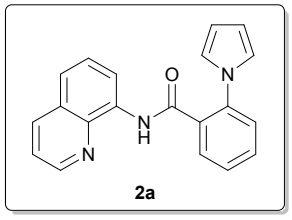


Figure 1: ORTEP diagram of 2-(1*H*-Pyrrol-1-yl)-*N*-(quinolin-8-yl)benzamide **2a** with 50% ellipsoid [CCDC 1427013].

Crystal Data and Structure Refinement for 2a at 296(2) K

Identification code	**2a**
Empirical formula	$C_{20}\ H_{15}\ N_3\ O$
Formula weight	313.35
Temperature	296(2)
Wavelength	0.71073
Crystal system	monoclinic
Space group	'$P2_1/n$'
Unit cell dimensions	a = 9.7059(3) Å b = 15.7541(4) Å c = 10.5250(3) Å

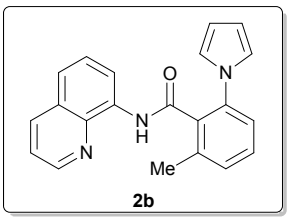
	$\alpha = \gamma = 90°$ $\beta = 95.513(2)°$
Volume	1601.91(8) Å^3
Z	4
Density (calculated)	1.299 Mg/m3
Absorption coefficient	0.083
F(000)	656
Crystal size	0.44 x 0.36 x 0.28
Theta range for data collection	2.33° to 24.99°
Index ranges	-10<=*h*<=10, -18<=*k*<=18, -12<=*l*<=12
Reflections collected	21958
Independent reflections	2701
Completeness to theta = 24.99°	95.60 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2701 / 0 / 217
Goodness-of-fit on F^2	1.081
Final R indices [I>2sigma (I)]	*R1* = 0.0405, *wR2* = 0.0961
R indices (all data)	*R1* = 0.0466, *wR2* = 0.1011



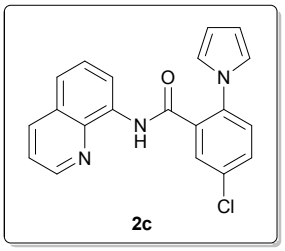
2a

2-(1*H*-Pyrrol-1-yl)-*N*-(quinolin-8-yl)benzamide 2a. Analytical TLC on silica gel, 1:9 ethyl acetate/hexane R_f = 0.49; white solid; 49 mg, yield 79%; mp 141-142 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ

9.90 (br s, 1H), 8.84 (d, J = 7.8 Hz, 1H), 8.66-8.65 (m, 1H), 8.12 (d, J = 7.8 Hz, 1H), 7.90 (d, J = 7.8 Hz, 1H), 7.59-7.44 (m, 5H), 7.41-7.39 (m, 1H), 6.99 (s, 2H), 6.18 (s, 2H); $^{13}C\{H\}$ NMR (150 MHz, $CDCl_3$) δ 165.8, 148.1, 138.9, 138.6, 136.3, 134.7, 132.6, 131.6, 130.1, 128.0, 127.5, 126.5, 122.13, 122.1, 121.8, 121.5, 116.8, 110.6; FT-IR (KBr) 1670, 1601, 1522, 1498, 1385, 1329, 1262, 1070, 1014, 924, 898 cm^{-1}. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{20}H_{15}N_3O$ 314.1293, found 314.1286.

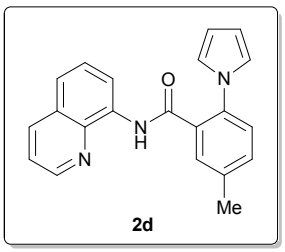


2-Methyl-6-(1*H*-pyrrol-1-yl)-*N*-(quinolin-8-yl)benzamide 2b. Analytical TLC on silica gel, 1:9 ethyl acetate/hexane R_f = 0.48; white solid; 55 mg, yield 84%; mp 146-147 °C; ^1H NMR (600 MHz, $CDCl_3$) δ 9.72 (br s, 1H), 8.82 (dd, J = 7.2 Hz, 1.2 Hz, 1H), 8.68 (dd, J = 4.8 Hz, 1.8 Hz, 1H), 8.13 (dd, J = 9.0 Hz, 1.8 Hz, 1H), 7.55-7.50 (m, 2H), 7.43-7.39 (m, 2H), 7.29-7.27 (m, 2H), 7.00-6.99 (m, 2H), 6.10-6.09 (m, 2H), 2.52 (s, 3H); $^{13}C\{H\}$ NMR (150 MHz, $CDCl_3$) δ 166.4, 148.3, 138.63, 138.6, 137.4, 136.4, 134.4, 133.6, 130.1, 129.3, 128.1, 127.5, 123.4, 122.2, 121.8, 116.9, 110.0, 19.9; FT-IR (KBr) 3471, 1676, 1510, 1483, 1326, 1265, 1123, 1088, 951, 897 cm^{-1}. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{21}H_{17}N_3O$ 328.1450, found 328.1448.

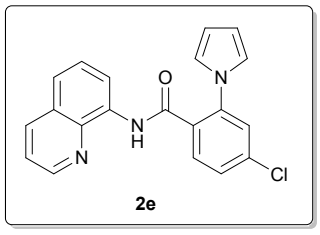


5-Chloro-2-(1*H*-pyrrol-1-yl)-*N*-(quinolin-8-yl)benzamide 2c. Analytical TLC on silica gel, 1:9 ethyl acetate/hexane R_f = 0.47; white solid; 48 mg, yield 69%; mp 173-174 °C; ^1H NMR (600 MHz, $CDCl_3$) δ 9.90 (br s, 1H), 8.80 (d, J = 7.2 Hz, 1H), 8.65 (d, J = 3.6 Hz, 1H), 8.13 (d, J = 8.4 Hz, 1H), 7.87 (s, 1H), 7.55-7.51 (m, 3H), 7.42-7.40 (m, 1H), 7.38 (d, J = 8.4 Hz, 1H), 6.95 (s, 2H), 6.19 (s, 2H); $^{13}C\{H\}$ NMR

(150 MHz, $CDCl_3$) δ 164.2, 148.2, 138.5, 137.3, 136.3, 134.3, 133.7, 133.3, 131.6, 130.1, 128.0, 127.8, 127.4, 122.4, 122.1, 121.9, 116.9, 111.0; FT-IR (KBr) 3330, 1670, 1523, 1494, 1327, 1260, 1108, 1073, 925, 825 cm^{-1}. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{20}H_{14}ClN_3O$ 348.0904, found 348.0901.

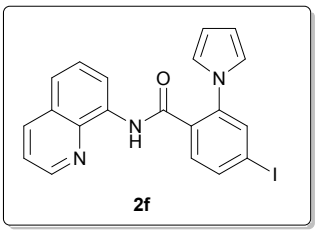


5-Methyl-2-(1*H*-pyrrol-1-yl)-*N*-(quinolin-8-yl)benzamide 2d. Analytical TLC on silica gel, 1:9 ethyl acetate/hexane R_f = 0.41; white solid; 51 mg, yield 78%; mp 145-146 °C; ^1H NMR (600 MHz, $CDCl_3$) δ 9.87 (br s, 1H), 8.83 (d, *J* = 7.2 Hz, 1H), 8.65 (d, *J* = 3.6 Hz, 1H), 8.12 (d, *J* = 7.8 Hz, 1H), 7.69 (s, 1H), 7.55-7.49 (m, 2H), 7.41-7.36 (m, 2H), 7.33 (d, *J* = 7.8 Hz, 1H), 6.96 (s, 2H), 6.16 (s, 2H), 2.46 (s, 3H); $^{13}C\{H\}$ NMR (150 MHz, $CDCl_3$) δ 166.0, 148.1, 138.6, 137.6, 136.4, 136.3, 134.7, 132.4, 132.2, 130.5, 128.0, 127.5, 126.4, 122.2, 122.0, 121.8, 116.8, 110.4, 21.2; FT-IR (KBr) 1665, 1648, 1524, 1485, 1384, 1327, 1262, 1070, 1015, 824 cm^{-1}. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{21}H_{17}N_3O$ 328.1450, found 328.1458.



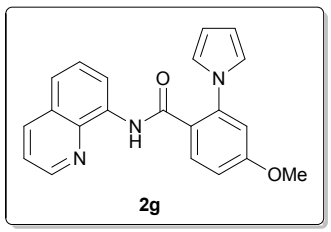
4-Chloro-2-(1*H*-pyrrol-1-yl)-*N*-(quinolin-8-yl)benzamide 2e. Analytical TLC on silica gel, 1:9 ethyl acetate/hexane R_f = 0.49; thick liquid; 44 mg, yield 63%; ^1H NMR (600 MHz, $CDCl_3$) δ 9.90 (br s, 1H), 8.81 (d, *J* = 7.2 Hz, 1H), 8.64 (d, *J* = 2.4 Hz, 1H), 8.12 (d, *J* = 8.4 Hz, 1H), 7.85 (d, *J* = 8.4 Hz, 1H), 7.55-7.50 (m, 2H), 7.45-7.44 (m, 2H), 7.41-7.39 (m, 1H), 6.97 (s, 2H), 6.19 (s, 2H); $^{13}C\{H\}$ NMR (150 MHz, $CDCl_3$) δ 164.8, 148.1, 139.8, 138.5, 137.3, 136.3, 134.4, 131.5, 130.7, 128.0, 127.6, 127.4, 126.5, 122.3,

122.0, 121.9, 116.8, 111.2; FT-IR (neat) 1673, 1595, 1527, 1482, 1385, 1326, 1263, 1108, 1021, 920, 825 cm^{-1}. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{20}H_{14}ClN_3O$ 348.0904, found 348.0904.



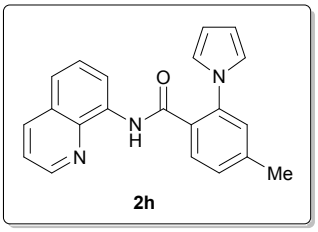
2f

4-Iodo-2-(1*H*-pyrrol-1-yl)-*N*-(quinolin-8-yl)benzamide 2f. Analytical TLC on silica gel, 1:9 ethyl acetate/hexane R_f = 0.49; white solid; 54 mg, yield 61%; mp 125-126 °C; ^1H NMR (600 MHz, $CDCl_3$) δ 9.90 (br s, 1H), 8.80 (d, *J* = 7.2 Hz, 1H), 8.64-8.63 (m, 1H), 8.12 (d, *J* = 8.4 Hz, 1H), 7.82-7.81 (m, 2H), 7.62 (d, *J* = 8.4 Hz, 1H), 7.55-7.50 (m, 2H), 7.41-7.39 (m, 1H), 6.96 (s, 2H), 6.18 (s, 2H); $^{13}C\{H\}$ NMR (150 MHz, $CDCl_3$) δ 164.9, 148.2, 139.6, 138.5, 136.5, 136.3, 135.3, 134.4, 131.8, 131.5, 128.0, 127.4, 122.3, 122.0, 121.9, 116.8, 111.2, 97.1; FT-IR (KBr) 1669, 1583, 1523, 1488, 1384, 1263, 1067, 1017, 929, 896 cm^{-1}. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{20}H_{14}IN_3O$ 440.0260, found 440.0266.



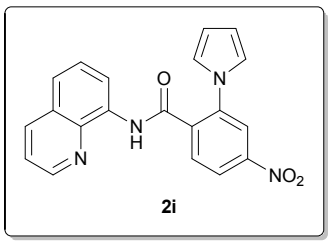
2g

4-Methoxy-2-(1*H*-pyrrol-1-yl)-*N*-(quinolin-8-yl)benzamide 2g. Analytical TLC on silica gel, 1:9 ethyl acetate/hexane R_f = 0.32; white solid; 48 mg, yield 70%; mp 127-128 °C; ^1H NMR (600 MHz, $CDCl_3$) δ 9.86 (br s, 1H), 8.83 (d, *J* = 7.2 Hz, 1H), 8.63 (dd, *J* = 4.2 Hz, 1.2 Hz, 1H), 8.11 (dd, *J* = 8.4 Hz, 1.2 Hz, 1H), 7.90 (d, *J* = 9.0 Hz, 1H), 7.54 (t, *J* = 8.4 Hz, 1H), 7.49 (dd, *J* = 8.4 Hz, 1.2 Hz, 1H), 7.39-7.37 (m, 1H), 7.01-6.98 (m, 3H), 6.92 (d, *J* = 2.4 Hz, 1H), 6.192-6.19 (m, 2H), 3.90 (s, 3H); $^{13}C\{H\}$ NMR (150 MHz, $CDCl_3$) δ 165.4, 162.1, 148.0, 140.5, 138.6, 136.2, 134.8, 132.0, 128.0, 127.5, 124.8, 122.1, 121.8, 121.7, 116.6, 113.2, 111.9, 110.7, 55.9; FT-IR (KBr) 3439, 1664, 1609, 1522, 1325, 1246, 1232, 1048, 898 cm^{-1}. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{21}H_{17}N_3O_2$ 344.1399, found 344.1402.



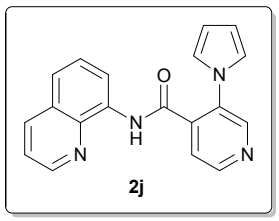
2h

4-Methyl-2-(1*H*-pyrrol-1-yl)-*N*-(quinolin-8-yl)benzamide 2h. Analytical TLC on silica gel, 1:9 ethyl acetate/hexane R_f = 0.49; white solid; 44 mg, yield 68%; mp 140-141 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.89 (br s, 1H), 8.84 (d, *J* = 7.2 Hz, 1H), 8.64-8.63 (m, 1H), 8.11 (d, *J* = 7.8 Hz, 1H), 7.81 (d, *J* = 7.8 Hz, 1H), 7.54 (t, *J* = 7.8 Hz, 1H), 7.49 (d, *J* = 8.4 Hz, 1H), 7.39-7.37 (m, 1H), 7.28 (d, *J* = 7.8 Hz, 1H), 7.24 (s, 1H), 6.98 (s, 2H), 6.18 (s, 2H), 2.46 (s, 3H); ^{13}C{H} NMR (150 MHz, $CDCl_3$) δ 165.8, 148.0, 142.3, 138.8, 138.6, 136.2, 134.7, 130.2, 129.7, 128.2, 128.0, 127.5, 127.1, 122.1, 121.9, 121.7, 116.7, 110.5, 21.5; FT-IR (KBr) 1668, 1614, 1524, 1487, 1385, 1326, 1262, 1099, 1072, 897 cm^{-1}. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{21}H_{17}N_3O$ 328.1450, found 328.1451.

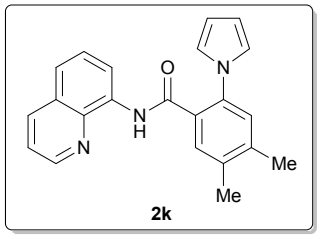


2i

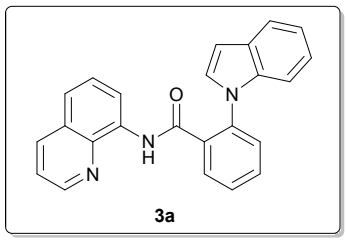
4-Nitro-2-(1*H*-pyrrol-1-yl)-*N*-(quinolin-8-yl)benzamide 2i. Analytical TLC on silica gel, 1:9 ethyl acetate/hexane R_f = 0.10; thick yellow liquid; 44 mg, yield 62%; ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.99 (br s, 1H), 8.80 (d, *J* = 3.6 Hz, 1H), 8.65 (d, *J* = 3.6 Hz, 1H), 8.32-8.29 (m, 2H), 8.15 (d, *J* = 8.4 Hz, 1H), 8.06 (d, *J* = 8.4 Hz, 1H), 7.56 (d, *J* = 3.6 Hz, 2H), 7.44-7.42 (m, 1H), 7.03 (s, 2H), 6.24 (s, 2H); ^{13}C{H} NMR (150 MHz, $CDCl_3$) δ 163.8, 149.4, 148.3, 139.7, 138.5, 137.4, 136.4, 134.0, 131.5, 128.0, 127.4, 122.8, 122.0, 121.9, 121.8, 121.3, 117.1, 111.9; FT-IR (neat) 1678, 1525, 1486, 1348, 1262, 1074, 1023, 946, 898 cm^{-1}. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{20}H_{14}N_4O_3$ 359.1144, found 359.1147.



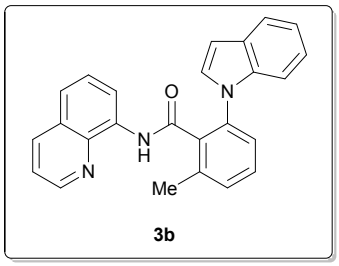
3-(1*H*-Pyrrol-1-yl)-*N*-(quinolin-8-yl)isonicotinamide 2j. Analytical TLC on silica gel, 1:9 ethyl acetate/hexane R_f = 0.20; white solid; 32 mg, yield 51%; mp 179-180 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 10.03 (br s, 1H), 8.81 (t, *J* = 4.2 Hz, 1H), 8.78 (s, 1H), 8.75 (d, *J* = 4.8 Hz, 1H), 8.66-8.65 (m, 1H), 8.15-8.14 (m, 1H), 7.79 (d, *J* = 4.8 Hz, 1H), 7.56 (d, *J* = 4.2 Hz, 2H), 7.44-7.42 (m, 1H), 7.015-7.01 (m, 2H), 6.273-6.27 (m, 2H); ^{13}C{H} NMR (150 MHz, $CDCl_3$) δ 163.3, 148.9, 148.3, 147.9, 138.7, 138.5, 136.4, 134.2, 134.0, 128.0, 127.4, 123.2, 122.8, 122.2, 122.0, 117.2, 111.6; FT-IR (KBr) 1677, 1527, 1486, 1425, 1326, 1265, 1114, 1076, 1013, 922, 826 cm^{-1}. HRMS (APCI) m/z: $[M+H]^+$ calcd for $C_{19}H_{14}N_4O$ 315.1246, found 315.1243.



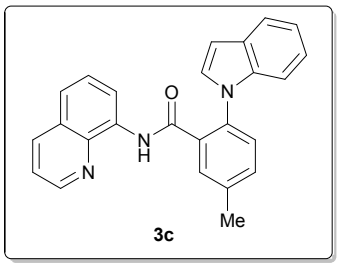
4,5-Dimethyl-2-(1*H*-pyrrol-1-yl)-*N*-(quinolin-8-yl)benzamide 2k. Analytical TLC on silica gel, 1:9 ethyl acetate/hexane R_f = 0.50; white solid; 48 mg, yield 71%; mp 195-196 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.87 (br s, 1H), 8.83 (d, *J* = 7.8 Hz, 1H), 8.63 (d, *J* = 3.0 Hz, 1H), 8.11 (d, *J* = 8.4 Hz, 1H), 7.68 (s, 1H), 7.54 (t, *J* = 7.8 Hz, 1H), 7.49 (d, *J* = 8.4 Hz, 1H), 7.39-7.37 (m, 1H), 7.20 (s, 1H), 6.95 (s, 2H), 6.16 (s, 2H), 2.37 (s, 3H), 2.36 (s, 3H); ^{13}C{H} NMR (150 MHz, $CDCl_3$) δ 165.9, 148.0, 140.8, 138.6, 136.6, 136.3, 136.2, 134.8, 131.1, 129.8, 128.0, 127.7, 127.5, 122.2, 121.9, 121.7, 116.7, 110.3, 19.9, 19.5; FT-IR (KBr) 1662, 1529, 1488, 1385, 1329, 1263, 1090, 1023, 862 cm^{-1}. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{22}H_{19}N_3O$ 342.1606, found 342.1608.



2-(1*H*-Indol-1-yl)-*N*-(quinolin-8-yl)benzamide 3a. Analytical TLC on silica gel, 1:9 ethyl acetate/hexane R_f = 0.46; white solid; 54 mg, yield 74%; mp 133-134°C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.96 (br s, 1H), 8.72 (d, *J* = 7.2 Hz, 1H), 8.14-8.13 (m, 1H), 8.02 (dd, *J* = 4.2 Hz, 1.2 Hz, 1H), 7.98 (d, *J* = 8.4 Hz, 1H), 7.66 (td, *J* = 7.8 Hz, 1.2 Hz, 1H), 7.59-7.51 (m, 3H), 7.49 (d, *J* = 8.4 Hz, 1H), 7.46 (t, *J* = 7.8 Hz, 1H), 7.40 (d, *J* = 8.4 Hz, 1H), 7.29 (d, *J* = 3.0 Hz, 1H), 7.26-7.24 (m, 1H), 7.23-7.20 (m, 1H), 7.13 (t, *J* = 7.2 Hz, 1H), 6.52 (d, *J* = 3.0 Hz, 1H); ^{13}C{H} NMR (150 MHz, $CDCl_3$) δ 164.9, 147.8, 138.2, 137.5, 137.1, 135.9, 134.4, 133.6, 132.0, 131.3, 129.6, 129.1, 128.3, 128.2, 127.7, 127.3, 122.8, 121.9, 121.5, 121.2, 120.6, 116.5, 110.7, 104.8; FT-IR (KBr) 1672, 1596, 1482, 1385, 1326, 1264, 1212,1143, 1012, 897 cm^{-1}. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{24}H_{17}N_3O$ 364.1450, found 364.1451.

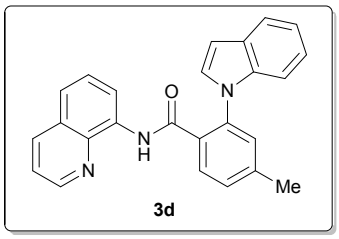


2-(1*H*-Indol-1-yl)-6-methyl-*N*-(quinolin-8-yl)benzamide 3b. Analytical TLC on silica gel, 1:9 ethyl acetate/hexane R_f = 0.45; white solid; 61 mg, yield 81%; mp 175-176 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.58 (br s, 1H), 8.64 (d, *J* = 7.2 Hz, 1H), 8.29 (d, *J* = 3.6 Hz, 1H), 7.99 (d, *J* = 7.8 Hz, 1H), 7.50-7.39 (m, 5H), 7.37 (d, *J* = 7.2 Hz, 2H), 7.32 (s, 1H), 7.25-7.23 (m, 1H), 7.22 (t, *J* = 7.8 Hz, 1H), 7.03 (t, *J* = 7.8 Hz, 1H), 6.36 (s, 1H), 2.57 (s, 3H); ^{13}C{H} NMR (150 MHz, $CDCl_3$) δ 165.9, 147.9, 138.2, 138.1, 137.5, 136.7, 136.1, 135.5, 134.1, 130.2, 130.1, 129.1, 128.9, 127.7, 127.2, 125.4, 122.3, 122.1, 121.5, 120.9, 120.3, 116.6, 110.8, 103.7, 20.0; FT-IR (KBr) 1675, 1596, 1524, 1482, 1385, 1326, 1264, 1212, 1116, 1012, 897 cm^{-1}. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{25}H_{19}N_3O$ 378.1606, found 378.1598.



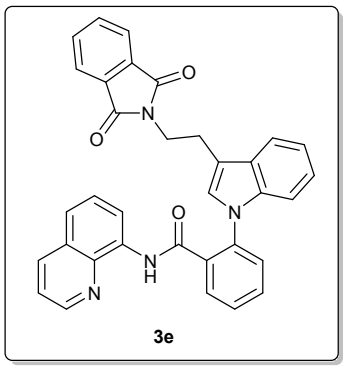
3c

2-(1*H*-Indol-1-yl)-5-methyl-*N*-(quinolin-8-yl)benzamide 3c . Analytical TLC on silica gel, 1:9 ethyl acetate/hexane R_f = 0.40; white solid; 58 mg, yield 77%; mp 147-148 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.94 (br s, 1H), 8.72 (d, *J* = 7.2 Hz, 1H), 8.00-7.97 (m, 2H), 7.94 (s, 1H), 7.53 (d, *J* = 7.8 Hz, 1H), 7.47-7.39 (m, 6H), 7.25-7.20 (m, 2H), 7.13 (t, *J* = 7.2 Hz, 1H), 6.50 (d, *J* = 3.0 Hz, 1H), 2.53 (s, 3H); ^{13}C{H} NMR (150 MHz, $CDCl_3$) δ 165.1, 147.7, 138.4, 138.3, 137.7, 135.9, 134.5, 133.3, 132.7, 131.6, 129.5, 129.2, 128.3, 127.7, 127.3, 122.7, 121.8, 121.5, 121.1, 120.5, 116.5, 110.7, 104.6, 21.3; FT-IR (KBr) 1729, 1667, 1524, 1485, 1384, 1327,1260, 1133, 928, 825 cm^{-1}. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{25}H_{19}N_3O$ 378.1606, found 378.1604.

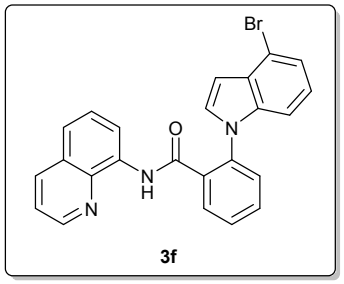


3d

2-(1*H*-Indol-1-yl)-4-methyl-*N*-(quinolin-8-yl)benzamide 3d. Analytical TLC on silica gel, 1:9 ethyl acetate/hexane R_f = 0.46; colourless thick liquid; 52 mg, yield 69%; ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.98 (br s, 1H), 8.72 (d, *J* = 7.8 Hz, 1H), 8.07 (d, *J* = 7.8 Hz, 1H), 7.98-7.96 (m, 2H), 7.54 (d, *J* = 8.4 Hz, 1H), 7.48-7.43 (m, 2H), 7.39 (d, *J* = 7.8 Hz, 2H), 7.33 (s, 1H), 7.27-7.23 (m, 2H), 7.22-7.19 (m, 1H), 7.14 (t, *J* = 7.2 Hz, 1H), 6.53 (d, *J* = 2.4 Hz, 1H), 2.48 (s, 3H); ^{13}C{H} NMR (150 MHz, $CDCl_3$) δ 164.9, 147.7, 142.8, 138.2, 137.6, 137.0, 135.9, 134.5, 131.3, 130.5, 129.6, 129.2, 129.1, 128.8, 127.7, 127.3, 122.7, 121.7, 121.5, 121.2, 120.6, 116.4, 110.7, 104.8, 21.5; FT-IR (neat) 1666, 1527, 1461, 1327, 1276, 1212, 1135, 1012, 900 cm^{-1}. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{25}H_{19}N_3O$ 378.1606, found 378.1597.

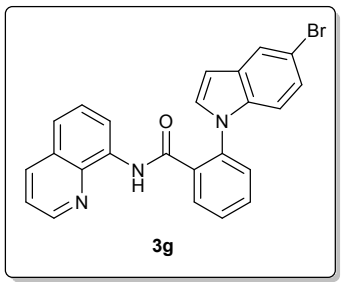


2-(3-(2-(1,3-Dioxoisoindolin-2-yl)ethyl)-1*H*-indol-1-yl)-*N*-(quinolin-8-yl)benzamide 3e. Analytical TLC on silica gel, 1:9 ethyl acetate/hexane R_f = 0.24; white solid; 61 mg, yield 57%; mp 179-180 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.90 (br s, 1H), 8.71 (d, *J* = 7.2 Hz, 1H), 8.18 (dd, *J* = 7.8 Hz, 1.2 Hz, 1H), 7.99 (dd, *J* = 4.2 Hz, 1.2 Hz, 1H), 7.95 (dd, *J* = 9.2 Hz, 1.2 Hz, 1H), 7.80-7.94 (m, 2H), 7.69-7.63 (m, 2H), 7.59-7.57 (m, 2H), 7.56-7.52 (m, 2H), 7.47-7.42 (m, 2H), 7.37 (d, *J* = 7.8 Hz, 1H), 7.27-7.25 (m, 1H), 7.23-7.21 (m, 2H), 7.17 (t, *J* = 7.2 Hz, 1H), 3.42-3.39 (m, 2H), 2.90-2.87 (m, 2H); ^{13}C{H} NMR (150 MHz, $CDCl_3$) δ 168.3, 165.0, 147.8, 138.1, 137.9, 137.0, 135.9, 134.4, 134.1, 133.4, 132.3, 132.0, 131.4,129.1, 128.1, 127.6, 127.3, 126.8, 123.3, 123.1, 121.8, 121.5, 120.5, 120.5, 119.3, 116.4, 114.7, 110.7, 37.9, 24.5; FT-IR (KBr) 2921, 1770, 1711, 1668, 1527, 1486, 1397, 1328, 1258, 1224,1128, 1052, 825 cm^{-1}. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{34}H_{24}N_4O_3$ 537.1927, found 537.1294.



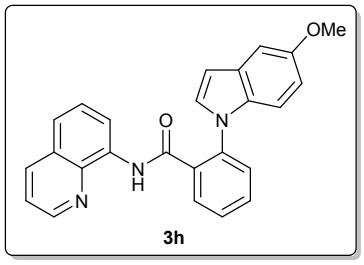
2-(4-Bromo-1*H*-indol-1-yl)-*N*-(quinolin-8-yl) 3f. Analytical TLC on silica gel, 1:9 ethyl acetate/hexane R_f = 0.43; white solid; 69 mg, yield 78%; mp 148-149 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.83 (br s, 1H), 8.63 (d, *J* = 7.2 Hz, 1H), 8.08 (d, *J* = 7.6 Hz, 1H), 8.00 (d, *J* = 4.4 Hz, 1H), 7.94 (d, *J* = 8.4 Hz, 1H), 7.60-7.51 (m, 2H), 7.43-7.30 (m, 4H), 7.26 (d, *J* = 3.2 Hz, 1H), 7.22-7.17 (m, 2H), 7.02 (t, *J* = 8.0 Hz, 1H), 6.50 (d, *J* = 3.2 Hz, 1H); ^{13}C{H} NMR (100 MHz, $CDCl_3$) δ 164.6, 147.9, 138.2, 137.8, 136.6, 136.1,

134.2, 133.5, 132.1, 131.4, 130.3, 129.7, 128.7, 128.4, 127.7, 127.2, 123.7, 123.6, 122.1, 121.7, 116.6, 115.1, 109.9, 105.1; FT-IR (KBr) 1668, 1596, 1486, 1385, 1359, 1297, 1203, 1179, 1086, 888 cm^{-1}. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{24}H_{16}BrN_3O$ 444.0535, found 444.0532.



3g

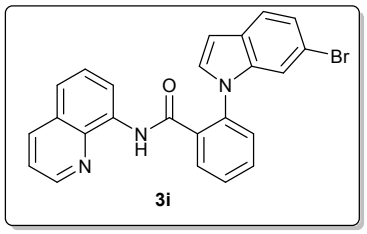
2-(5-Bromo-1*H*-indol-1-yl)-*N*-(quinolin-8-yl)benzamide 3g. Analytical TLC on silica gel, 1:9 ethyl acetate/hexane R_f = 0.45; white solid; 66 mg, yield 75%; mp 141-142 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.84 (br s, 1H), 8.71 (dd, *J* = 7.6 Hz, 2.0 Hz, 1H), 8.11-8.09 (m, 2H), 8.02 (dd, *J* = 8.4 Hz, 1.6 Hz, 1H), 7.67-7.65 (m, 1H), 7.62-7.58 (m, 2H), 7.53 (d, *J* = 7.6 Hz, 1H), 7.48-7.41 (m, 2H), 7.34 (d, *J* = 2.4 Hz, 2H), 7.30-7.25 (m, 2H), 6.43 (d, J = 3.2 Hz, 1H); ^{13}C{H} NMR (150 MHz, $CDCl_3$) δ 164.8, 147.8, 138.1, 136.5, 136.2, 136.1, 134.2, 133.9, 132.1, 131.2, 131.1, 130.3,128.7, 128.2, 127.7, 127.3, 125.6, 123.7, 122.1, 121.7, 116.4, 113.9, 112.2, 104.1; FT-IR (KBr) 1669, 1596, 1486, 1386, 1327, 1289, 1227,1198, 1053, 896 cm^{-1}. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{24}H_{16}BrN_3O$ 442.0555, found 442.0554.



3h

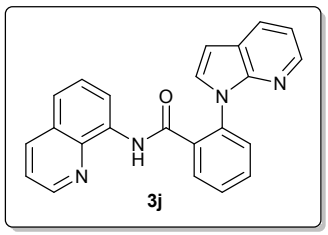
2-(5-Methoxy-1*H*-indol-1-yl)-*N*-(quinolin-8-yl)benzamide 3h. Analytical TLC on silica gel, 1:9 ethyl acetate/hexane R_f = 0.32; white solid; 64 mg, yield 81%; mp 150-151 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.96 (br s,1H), 8.73 (dd, J = 7.6 Hz, 1.6 Hz, 1H), 8.13-8.09 (m, 2H), 7.99 (dd, J = 8.4 Hz, 1.6 Hz, 1H), 7.66 (td, J = 7.6 Hz, 1.6 Hz, 1H), 7.58-7.52 (m, 2H), 7.47 (t, *J* = 8.0 Hz, 1H), 7.41-7.36 (m, 2H), 7.27-7.22 (m, 2H), 6.97 (d, J = 2.4 Hz, 1H), 6.91-6.88 (m, 1H), 6.44 (d, J = 3.2 Hz, 1H), 3.81 (s, 3H); ^{13}C{H}

NMR (100 MHz, $CDCl_3$) δ 165.0, 154.8, 147.7, 138.2, 137.1, 136.0, 134.4, 133.5, 132.8, 131.9, 131.1, 130.1, 129.7, 128.1, 127.7, 127.2, 121.9, 121.5, 116.4, 112.8,111.4, 104.5, 102.9, 56.0; FT-IR (KBr) 1668, 1598, 1487, 1386, 1327, 1256, 1218, 1193, 1032, 899 cm^{-1}. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{25}H_{19}N_3O_2$ 394.1556, found 394.1556.



3i

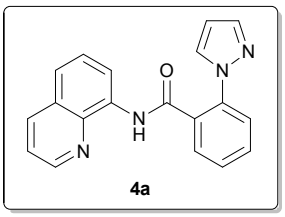
2-(6-Bromo-1*H*-indol-1-yl)-*N*-(quinolin-8-yl)benzamide 3a. Analytical TLC on silica gel, 1:9 ethyl acetate/hexane R_f = 0.46; white solid; 68 mg, yield 77%; mp 110-111 °C; ^1H NMR (400 MHz, $CDCl_3$) δ 9.78 (br s, 1H), 8.63 (dd, J = 7.6 Hz, 1.6 Hz,1H), 8.03-7.99 (m, 2H), 7.91 (dd, J = 8.0 Hz, 1.6 Hz, 1H), 7.60-7.57 (m, 2H), 7.54-7.50 (m, 1H), 7.46-7.37 (m, 1H), 7.35-7.31 (m, 2H), 7.28 (d, *J* = 8.4 Hz, 1H), 7.17-7.14 (m, 3H), 6.36 (d, J = 3.2 Hz, 1H); $^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$) δ 164.8, 147.9, 138.2, 138.1, 136.3, 136.0, 134.2, 134.1, 132.0, 131.1, 129.8, 128.7, 128.24, 128.2, 127.7, 127.2, 123.9, 122.4, 122.0, 121.6, 116.4, 116.3, 113.7, 104.7; FT-IR (KBr) 1671, 1597, 1485, 1385, 1327, 1267, 1230, 1137, 1052, 891 cm^{-1}. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{24}H_{16}BrN_3O$ 442.0555, found 442.0553.



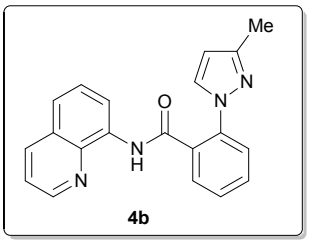
3j

2-(1*H*-Pyrrolo[2,3-b]pyridin-1-yl)-*N*-(quinolin-8-yl)benzamide 3a. Analytical TLC on silica gel, 1:9 ethyl acetate/hexane R_f = 0.28; colourless thick liquid; 46 mg, yield 63%; ^1H NMR (600 MHz, $CDCl_3$) δ 9.91 (br s, 1H), 8.60 (d, *J* = 7.2 Hz, 1H), 8.24 (d, *J* = 4.8 Hz, 1H), 8.12-8.11 (m, 1H), 7.97 (d, *J* = 7.8 Hz, 1H), 7.93 (d, *J* = 8.4 Hz, 1H), 7.73 (dd, *J* = 7.8 Hz, 1.2 Hz, 1H), 7.60-7.56 (m, 2H), 7.51 (t, *J* = 7.2 Hz, 1H), 7.38-7.32 (m, 3H), 7.19-7.17 (m, 1H), 6.97-6.95 (m, 1H), 6.40 (d, *J* = 3.6 Hz, 1H); $^{13}C\{H\}$ NMR

(150 MHz, $CDCl_3$) δ 165.5, 148.7, 147.9, 143.9, 138.3, 136.0, 135.6, 134.5, 134.2, 131.8, 130.4, 129.5, 129.2, 128.7, 128.4, 127.8, 127.3,121.8, 121.51, 121.5, 116.8, 116.5, 102.4; FT-IR (KBr) 1670, 1586, 1485, 1384, 1326, 1281, 1202, 1130, 1052, 896 cm^{-1}. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{23}H_{16}N_4O$ 365.1402, found 365.1408.

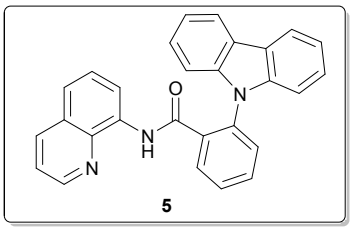


2-(1*H*-Pyrazol-1-yl)-*N*-(quinolin-8-yl)benzamide 4a. Analytical TLC on silica gel, 1:5 ethyl acetate/hexane R_f = 0.48; white solid; 34 mg, yield 54%; mp 122-123 °C; ^1H NMR (600 MHz, $CDCl_3$) δ 9.99 (br s, 1H), 8.83 (dd, *J* = 7.2 Hz, 1.2 Hz, 1H), 8.70 (dd, *J* = 4.8 Hz, 1.8 Hz, 1H), 8.13 (dd, *J* = 7.8 Hz, 1.2 Hz, 1H), 7.87 (d, *J* = 7.8 Hz, 1H), 7.81 (d, *J* = 2.4 Hz, 1H), 7.63-7.60 (m, 3H), 7.55-7.50 (m, 3H), 7.42-7.40 (m, 1H), 6.30-6.29 (m, 1H); $^{13}C\{H\}$ NMR (150 MHz, $CDCl_3$) δ 165.8, 148.3, 141.6, 138.6, 138.2, 136.3, 134.7, 132.5, 131.4, 130.7, 129.7, 128.4, 128.0, 127.5, 125.8, 122.1, 121.8, 116.9, 107.7; FT-IR (KBr) 1671, 1603, 1520, 1485, 1385, 1326, 1265, 1099, 1043, 937, 826 cm^{-1}. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{19}H_{14}N_4O$ 315.1246, found 315.1247.

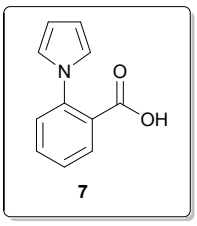


2-(3-Methyl-1*H*-pyrazol-1-yl)-*N*-(quinolin-8-yl)benzamide 4b. Analytical TLC on silica gel, 1:5 ethyl acetate/hexane R_f = 0.51; colourless thick liquid; 38 mg, yield 58%; ^1H NMR (600 MHz, $CDCl_3$) δ 10.01 (br s, 1H), 8.83 (d, *J* = 7.8 Hz, 1H), 8.70 (dd, *J* = 4.2 Hz, 1.8 Hz, 1H), 8.13 (dd, *J* = 7.8 Hz, 1.2 Hz, 1H), 7.85-7.84 (m, 1H), 7.66 (d, *J* = 1.8 Hz, 1H), 7.62-7.57 (m, 2H), 7.55-7.47 (m, 3H), 7.41-7.39 (m, 1H), 6.042-6.04 (m, 1H), 2.18 (s, 3H), $^{13}C\{H\}$ NMR (150 MHz, $CDCl_3$) δ 166.0, 150.9, 148.3, 138.7, 138.2, 136.3, 134.8, 132.2, 131.6, 131.5, 129.8, 128.1, 128.0, 127.4, 125.9, 122.1, 121.8, 116.9, 107.7, 13.8; FT-

IR (neat) 1676, 1524, 1485, 1326, 1265, 1129, 948, 826 cm^{-1}. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{20}H_{16}N_4O$ 329.1402, found 329.1403.



2-(9*H*-Carbazol-9-yl)-*N*-(quinolin-8-yl)benzamide 5. Analytical TLC on silica gel, 1:9 ethyl acetate/hexane R_f = 0.40; white solid; 34 mg, yield 41%; mp 184-185 °C; ^1H NMR (600 MHz, $CDCl_3$) δ 10.46 (br s, 1H), 8.62-8.61 (m, 1H), 8.41 (dd, *J* = 7.8 Hz, 1.8 Hz, 1H), 8.02 (d, *J* = 7.2 Hz, 2H), 7.87 (dd, *J* = 8.4 Hz, 1.2 Hz, 1H), 7.71-7.68 (m, 3H), 7.49 (dd, *J* = 7.8 Hz, 1.2 Hz, 1H), 7.40-7.33 (m, 5H), 7.29 (d, *J* = 8.4 Hz, 1H), 7.24 (td, *J* = 7.8 Hz, 1.2 Hz, 2H), 7.11-7.09 (m, 1H); $^{13}C\{H\}$ NMR (150 MHz, $CDCl_3$) δ 164.2, 147.5, 142.1, 138.2, 135.7, 135.6, 134.4, 132.9, 132.1, 129.7, 129.0, 127.5, 127.1, 126.5, 124.7, 121.6, 121.3, 120.6, 120.3, 116.4, 110.5; FT-IR (KBr) 3320, 1669, 1527, 1451, 1259, 920, 822 cm^{-1}. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{28}H_{19}N_3O$ 414.1606, found 414.1614.



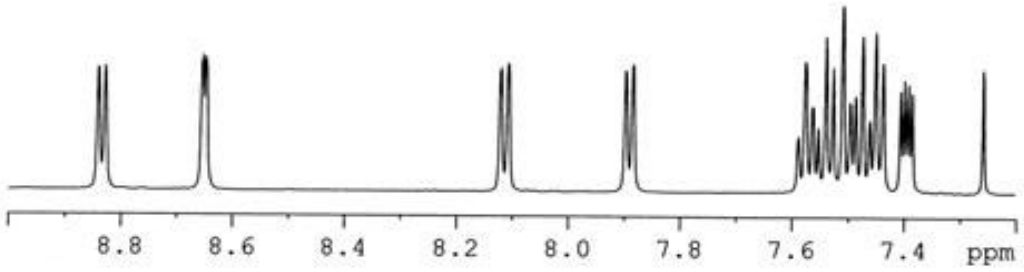
2-(1*H*-Pyrrol-1-yl)benzoic acid 7. Analytical TLC on silica gel, 9:1 ethyl acetate/hexane R_f = 0.25; pale brown solid; 31 mg, yield 84%; mp 96-97 °C; ^1H NMR (600 MHz, $CDCl_3$) δ 7.95-7.94 (m, 1H), 7.16 (td, *J* = 7.2 Hz, 1.2 Hz, 1H), 7.44 (t, *J* = 7.2 Hz, 1H), 7.40 (d, *J* = 7.8 Hz, 1H), 6.85-6.84 (m, 2H), 6.33-6.32 (m, 2H); $^{13}C\{H\}$ NMR (150 MHz, $CDCl_3$) δ 170.1, 141.1, 133.3, 131.6, 127.4, 127.3, 126.5, 122.3, 110.1; FT-IR (KBr) 1670, 1600, 1499, 1407, 1303, 1263, 1078, 942 cm^{-1}. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{11}H_{10}NO_2$ 188.0712, found 188.0712.

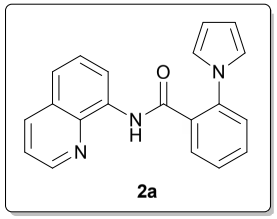
References

1. W. L. F. Armarego and C. L. L. Chai, In *Purification of Laboratory Chemicals*, Sixth Edition, Elsevier's Science and Technology, UK, 2009, 428-429.
2. B. S. Furniss, A. J. Hannaford, P. W. G. Smith and A. R. Tatchell, In *Vogel's Textbook of Practical Organic Chemistry*, Fifth Edition, Pearson Education Pte. Ltd., Indian Branch, Delhi, 2004, 928-929.
3. (a) Y. Aihara and N. Chatani, *J. Am. Chem. Soc*., 2013, **135**, 5308; (b) Y. Ano, M. Tobisu and N. Chatani, *Org. Lett*., 2012, **14**, 354.
4. S. Chiba, Y. Wang, G. Lapointe and K. Narasaka, *Org. Lett*., 2008, **10**, 313.
5. T. D. Montgomery, A. E. Nibbs, Y. Zhu and V. H. Rawal, *Org. Lett*. 2014, **16**, 3480.

NMR Spectra (^{1}H and ^{13}C)

SP-Ph-9feb_1H



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PROCNO                   1

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INSTRUM                spect
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PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       16
DS                        2
SWH               12019.230 Hz
FIDRES             0.366798 Hz
AQ                 1.3631488 sec
RG                      113
DW                   41.600 usec
DE                    6.50 usec
TE                   297.9 K
D1                1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
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NUC1                     1H
P1                    12.00 usec
PLW1           21.00000000 W

F2 - Processing parameters
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WDW                      EM
SSB                   0
LB                             0.30 Hz
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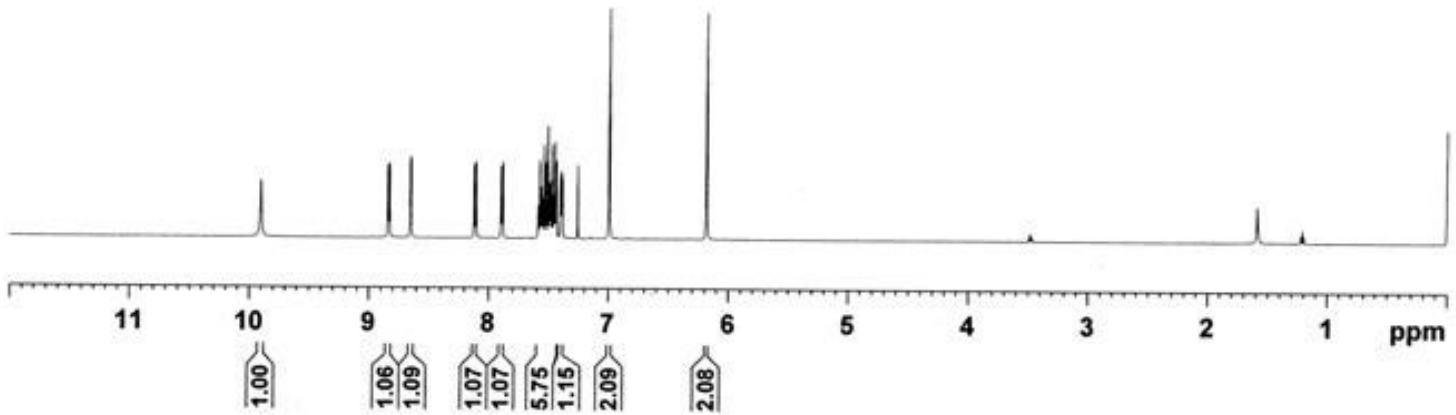
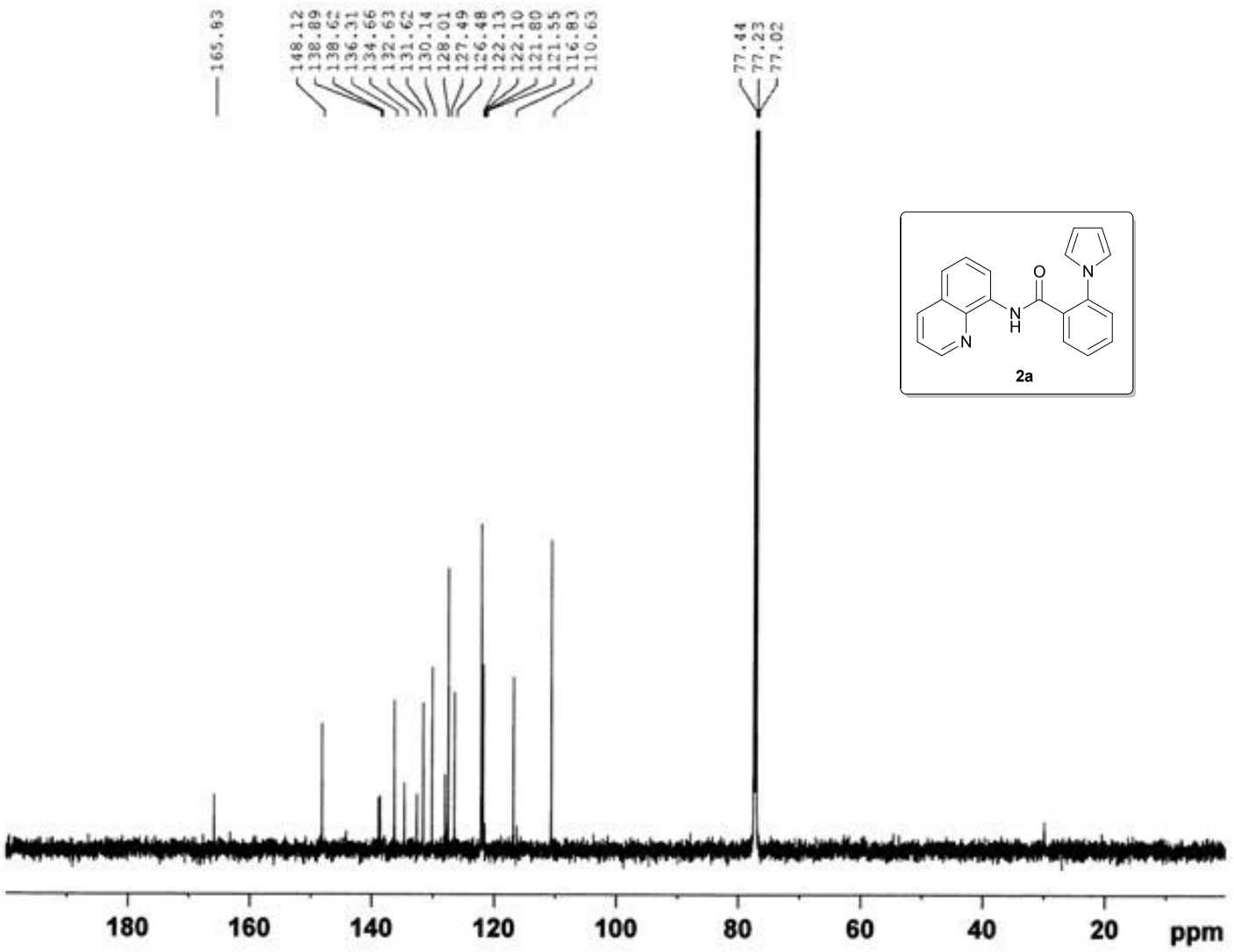


SP-PY-07-12-14_13C



2a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PROCNO                    1

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Time                 12.09
INSTRUM              spect
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PULPROG            zgpg30
TD                  32768
SOLVENT             CDCl3
NS                   1185
DS                      2
SWH            36057.691 Hz
FIDRES          1.100393 Hz
AQ             0.4543829 sec
RG                  65.24
DW                 13.867 usec
DE                   6.50 usec
TE                 298.9 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
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NUC1                 13C
P1                 10.50 usec
PLW1        95.00000000 W

======== CHANNEL f2 ========
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NUC2                 1H
CPDPRG[2         waltz16
PCPD2              70.00 usec
PLW2        21.00000000 W
PLW12        0.61714000 W
PLW13        0.30239999 W

F2 - Processing parameters
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WDW                    EM
SSB       0
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GB        0
PC                  1.40
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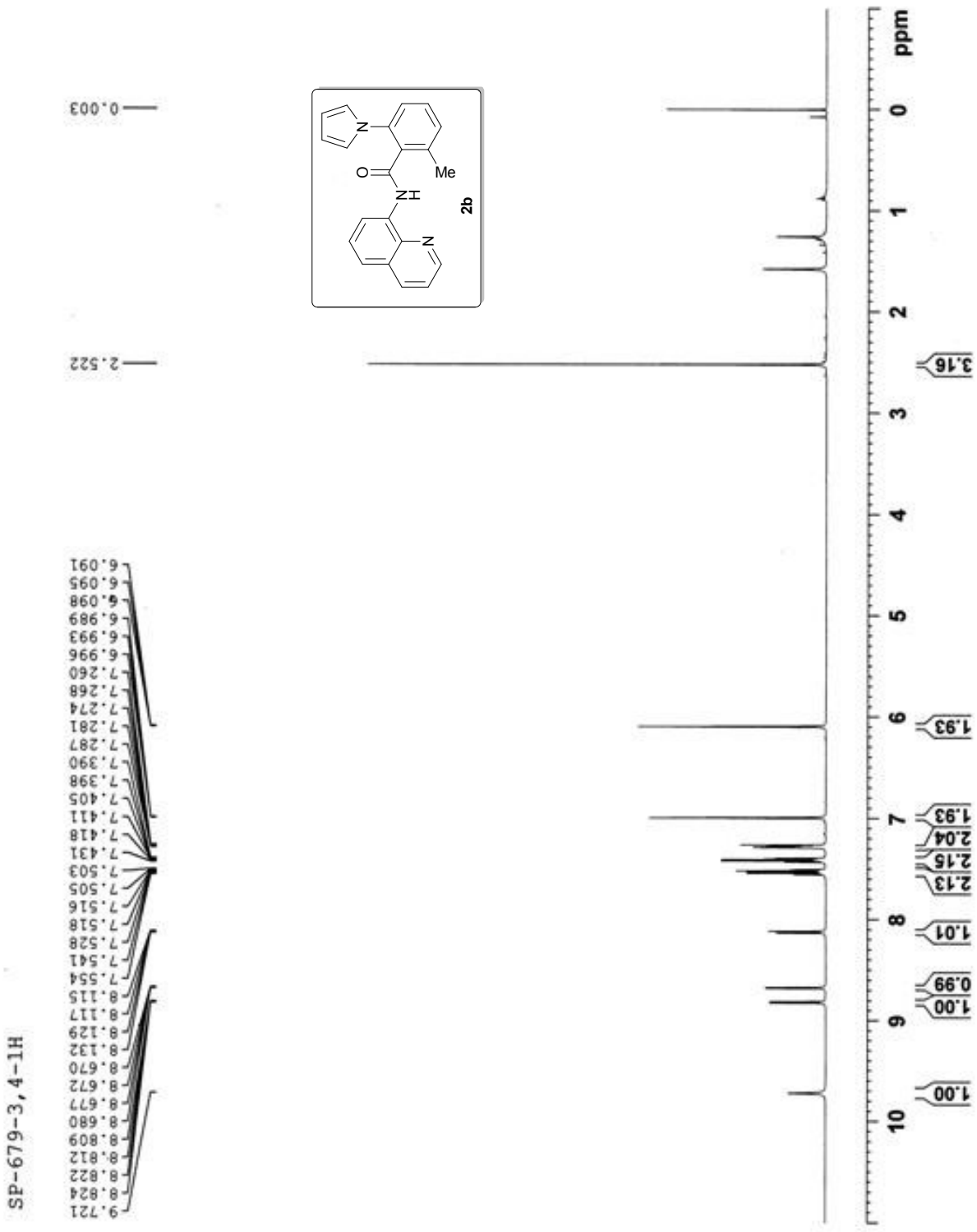
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2b
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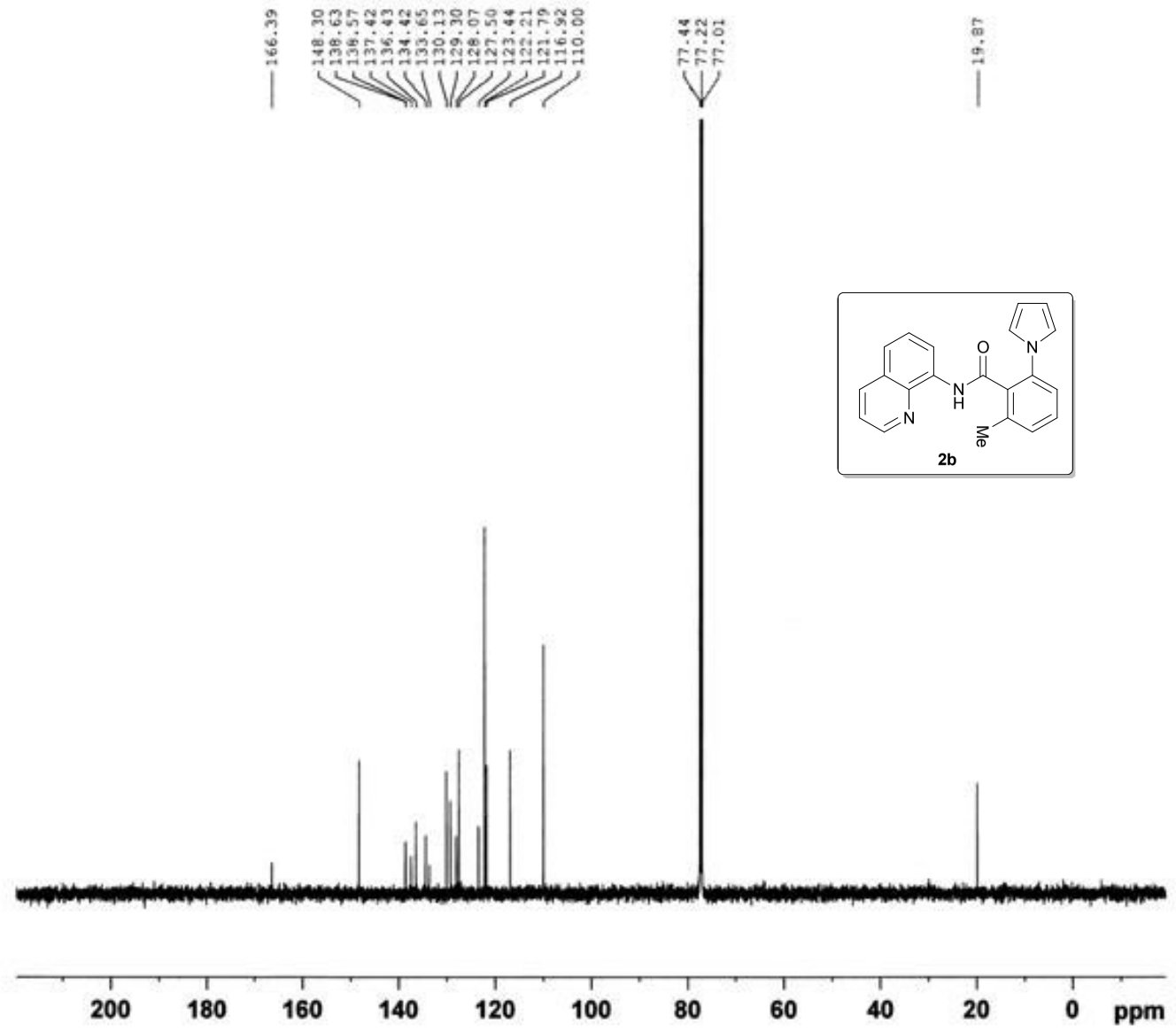
2.04
2.15
2.13
1.01
0.99
1.00
1.00

SP-679-3,4-13C



2b

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Current Data Parameters
NAME      SP-679-3,4-13C
EXPNO                  1
PROCNO                 1

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INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                32768
SOLVENT           CDCl3
NS                  218
DS                    2
SWH           36057.691 Hz
FIDRES         1.100393 Hz
AQ            0.4543829 sec
RG                65.24
DW               13.867 usec
DE                6.50 usec
TE                298.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        150.9279571 MHz
NUC1               13C
P1               10.50 usec
PLW1        95.00000000 W

======== CHANNEL f2 ========
SFO2        600.1724007 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            70.00 usec
PLW2        21.00000000 W
PLW12        0.61714000 W
PLW13        0.30239999 W

F2 - Processing parameters
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WDW                EM
SSB       0
LB               1.00 Hz
GB        0
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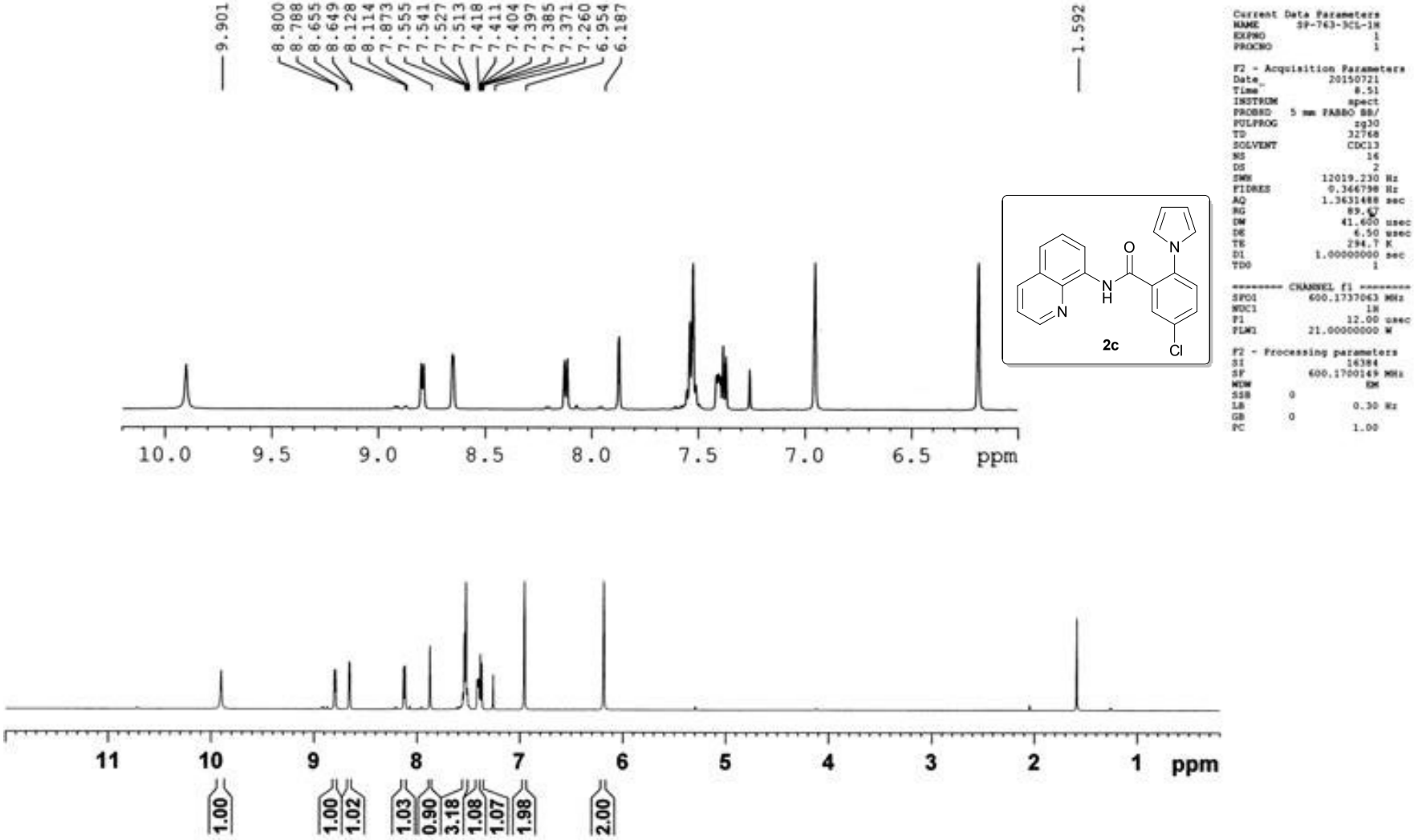
SP-763-3CL-1H



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EXPNO 1
PROCNO 1

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Time 8.51
INSTRUM spect
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PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 89.67
DW 41.600 usec
DE 6.50 usec
TE 294.7 K
D1 1.00000000 sec
TD0 1

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NDC1 18
P1 12.00 usec
PLW1 21.00000000 W

F2 - Processing parameters
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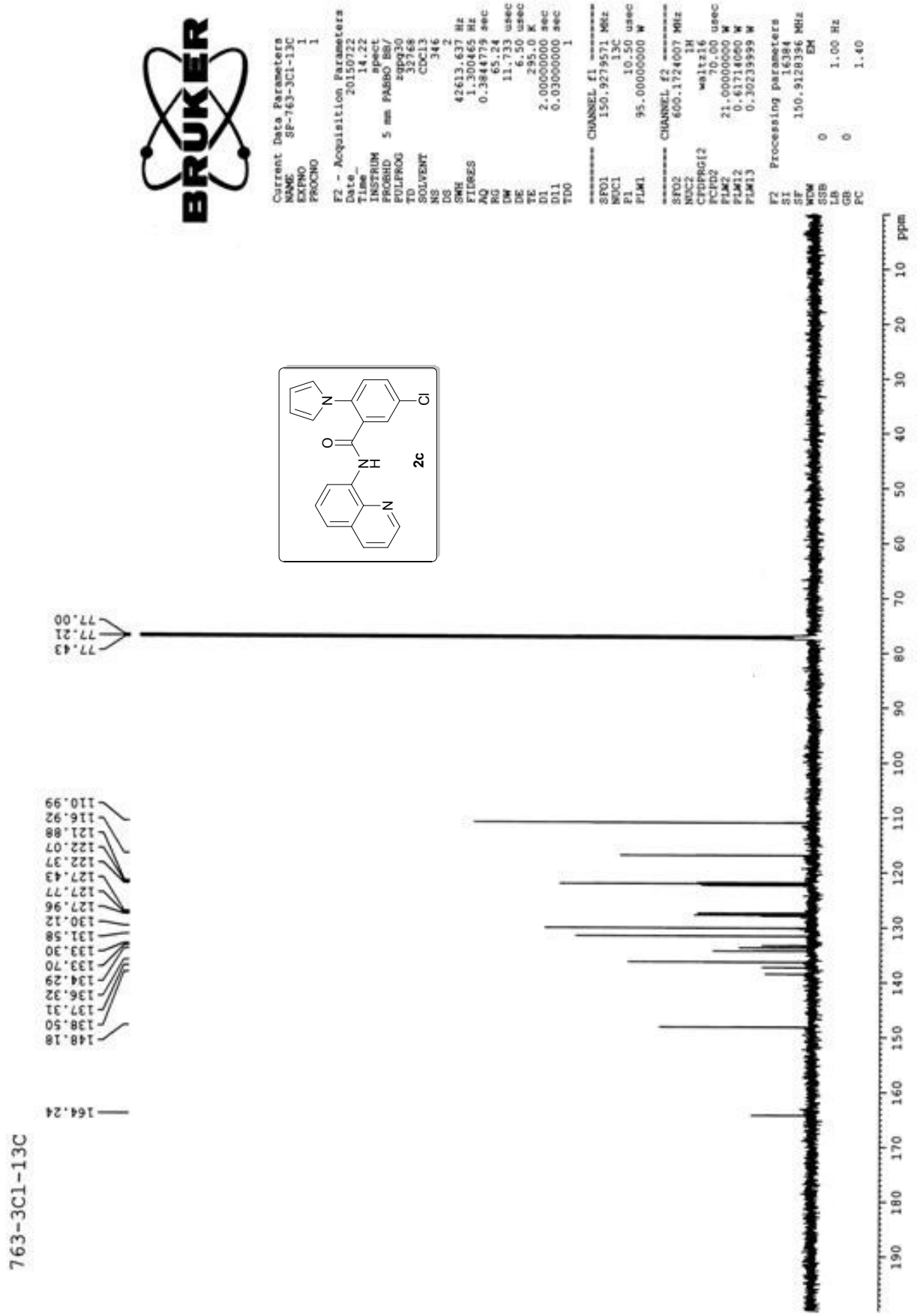
SF 600.1700149 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


763-3C1-13C
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137.31
136.32
134.29
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133.30
131.58
130.12
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127.77
127.43
122.37
122.07
121.88
116.92
110.99
77.43
77.21
77.00
2c
Cl
O
NH
N
Current Data Parameters
NAME 8P-763-3C1-13C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150722
Time_ 14.22
INSTRUM spect
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PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 346
DS 2
SWH 42613.637 Hz
FIDRES 1.300465 Hz
AQ 0.3844779 sec
RG 65.24
DW 11.733 usec
DE 6.50 usec
TE 295.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
SFO1 150.9279571 MHz
NUC1 13C
P1 10.50 usec
PLW1 95.00000000 W
CHANNEL f2
SFO2 600.1324007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 21.00000000 W
PLW12 0.61714060 W
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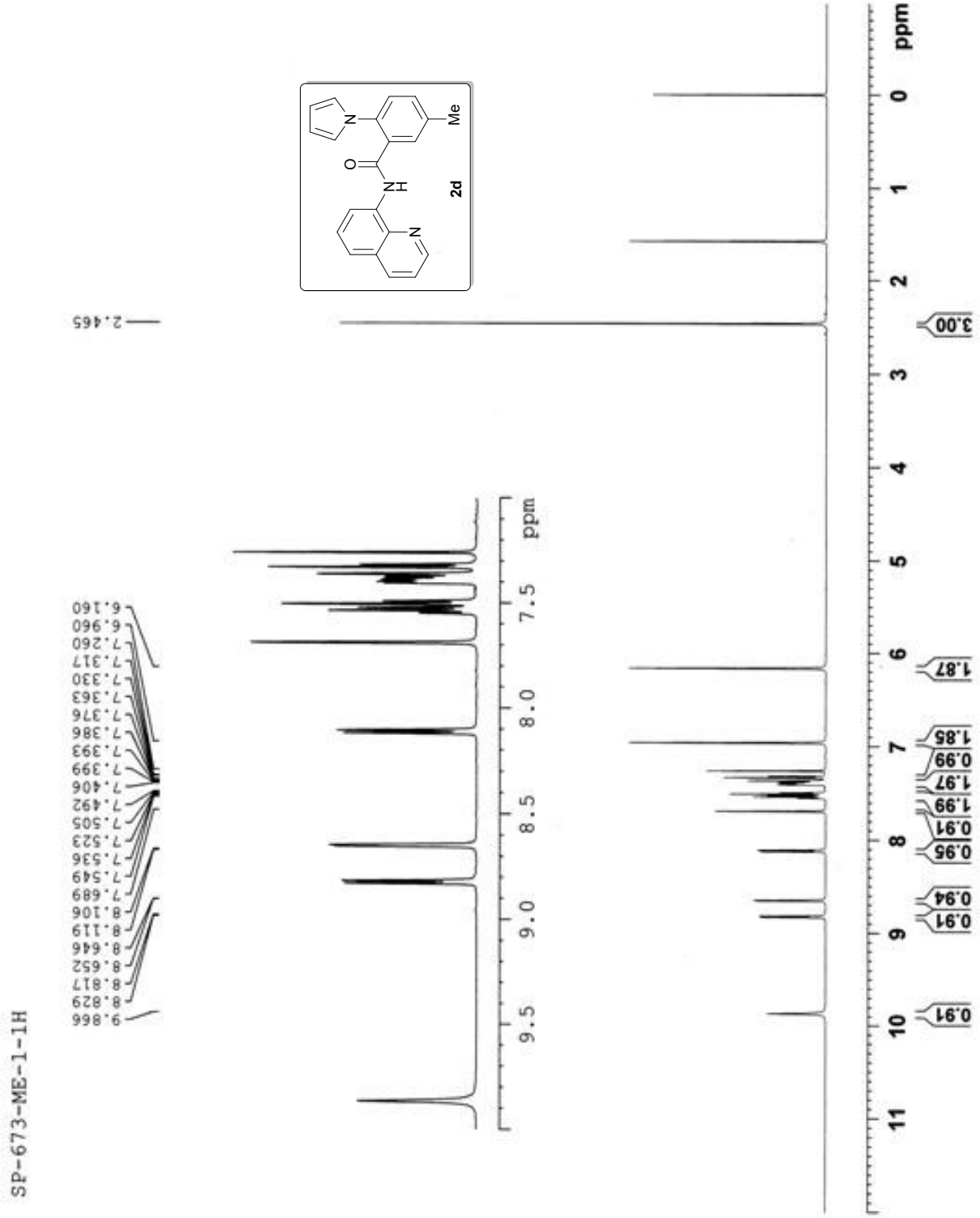
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
ppm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EXPNO 1
PROCNO 1
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INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.366788 Hz
AQ 1.3631488 sec
RG 143.33
DW 41.600 usec
DE 6.50 usec
TE 293.2 K
D1 1.00000000 sec
TD0 1
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NUC1 1H
P1 12.00 usec
PLW1 21.00000000 W
F2 - Processing parameters
SI 16384
SF 600.1700147 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
2d
Me
N
O
NH
N
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1.85
0.99
1.97
1.99
0.91
0.95
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0.94
0.91
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8.829
8.817
8.652
8.646
8.119
8.106
7.689
7.549
7.536
7.523
7.505
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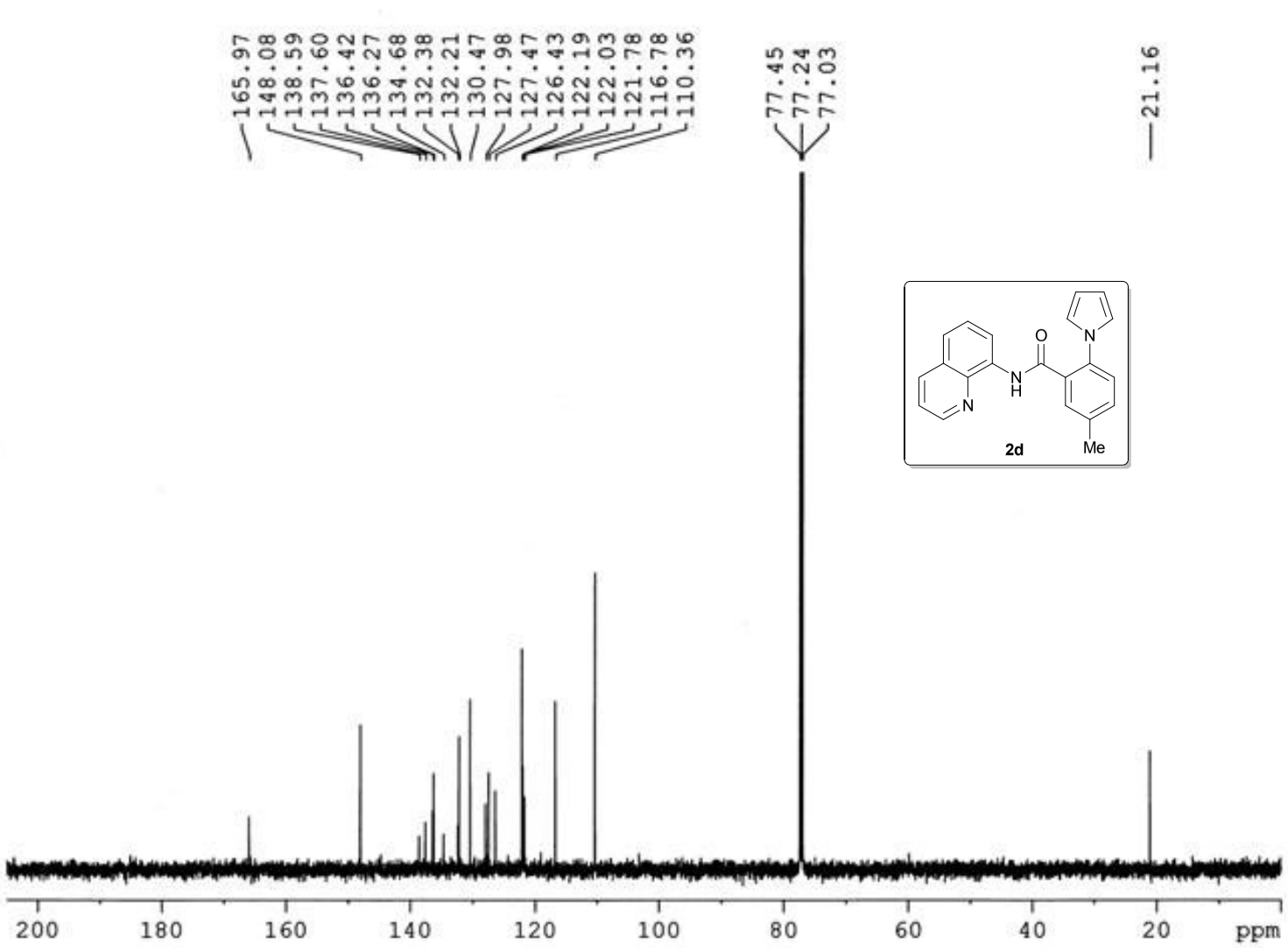
ppm
ppm

SP-673-13C



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Current Data Parameters
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EXPNO                          1
PROCNO                         1

F2 - Acquisition Parameters
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INSTRUM                    spect
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PULPROG                  zgpg30
TD                        32768
SOLVENT                   CDCl3
NS                           54
DS                            2
SWH                   36057.691 Hz
FIDRES                 1.100393 Hz
AQ                    0.4543829 sec
RG                       200.18
DW                       13.867 usec
DE                         6.50 usec
TE                        297.3 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                           1

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NUC1                        13C
P1                        10.50 usec
PLW1                95.00000000 W

======== CHANNEL f2 ========
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NUC2                        1H
CPDPRG[2                waltz16
PCPD2                     70.00 usec
PLW2                21.00000000 W
PLW12                0.61714000 W
PLW13                0.30239999 W

F2 - Processing parameters
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WDW                          EM
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GB                  0
PC                        1.40
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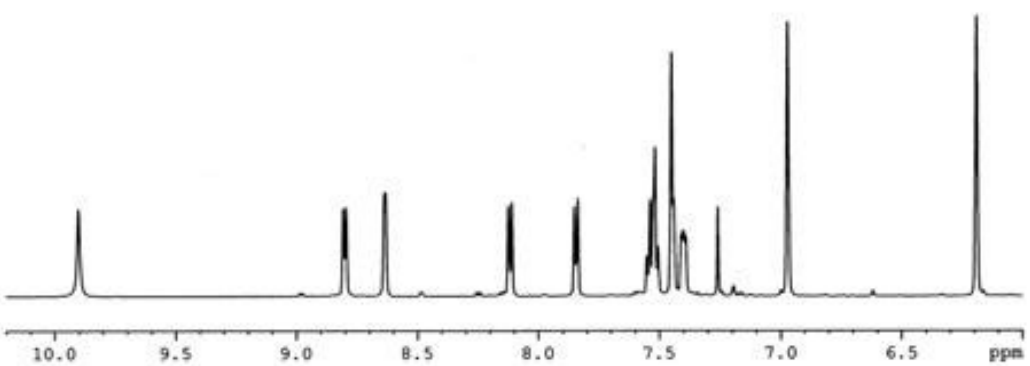
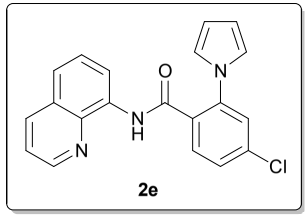
SP-4Cl-1H



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Current Data Parameters
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EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
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Time               9.30
INSTRUM                 spect
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PULPROG                 zg30
TD                     32768
SOLVENT                CDCl3
NS                        16
DS                         2
SWH               12019.230 Hz
FIDRES             0.366798 Hz
AQ                 1.3631488 sec
RG                       113
DW                  41.600 usec
DE                   6.50 usec
TE                  293.9 K
D1                 1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1              600.1737063 MHz
NDC1                        1H
P1                       12.00 usec
PLW1             21.00000000 W

F2 - Processing parameters
SI                        16384
SF                600.1700149 MHz
WDW                         EM
SSB          0
LB                        0.30 Hz
GB           0
PC                        1.00
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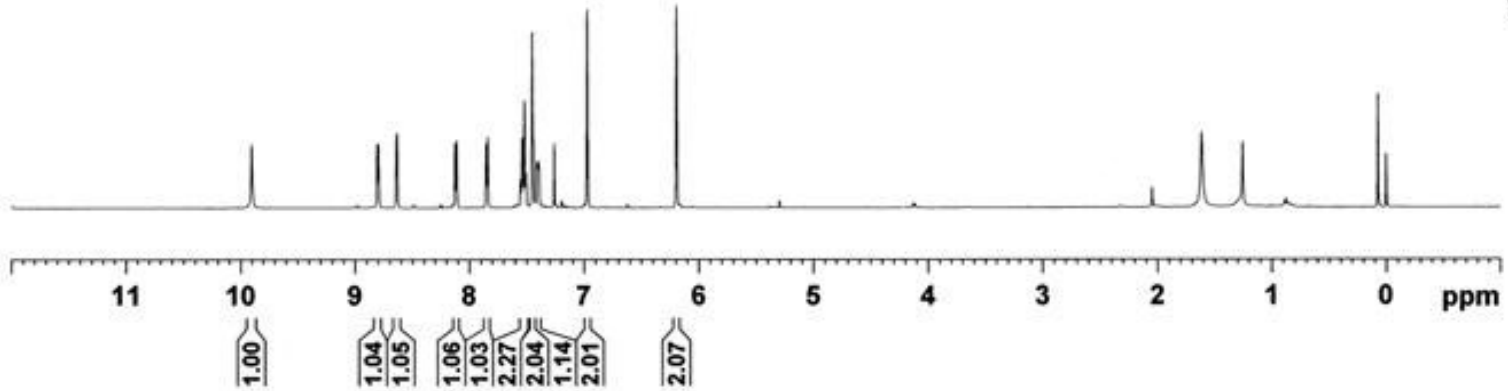

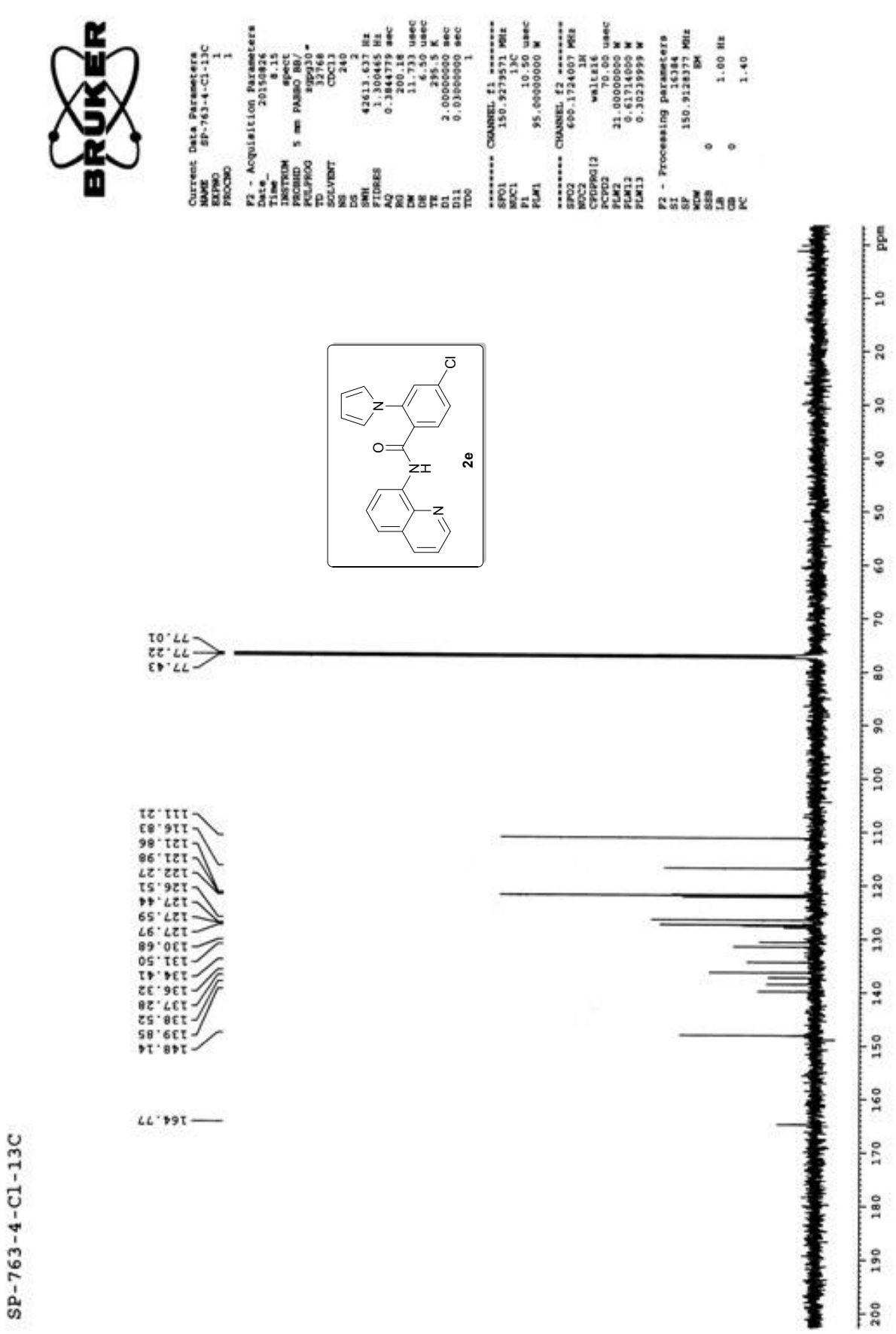
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EXPNO 1
PROCNO 1
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Time 0.15
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 240
DS 2
SWH 42613.637 Hz
FIDRES 1.300465 Hz
AQ 0.3844779 sec
RG 200.18
DW 11.733 usec
DE 6.50 usec
TE 295.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 150.9279571 MHz
NUC1 13C
P1 10.50 usec
PLW1 95.00000000 W
======== CHANNEL f2 ========
SFO2 600.1724007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 21.00000000 W
PLW12 0.61714000 W
PLW13 0.30239999 W
F2 - Processing parameters
SI 16384
SF 150.9128377 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
2e
77.01
77.22
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126.51
127.44
127.59
127.97
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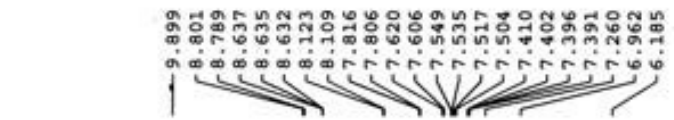
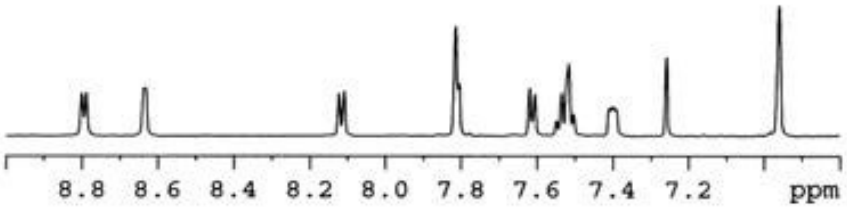
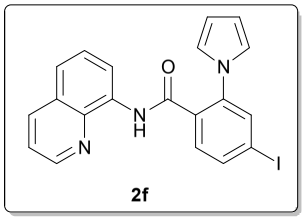
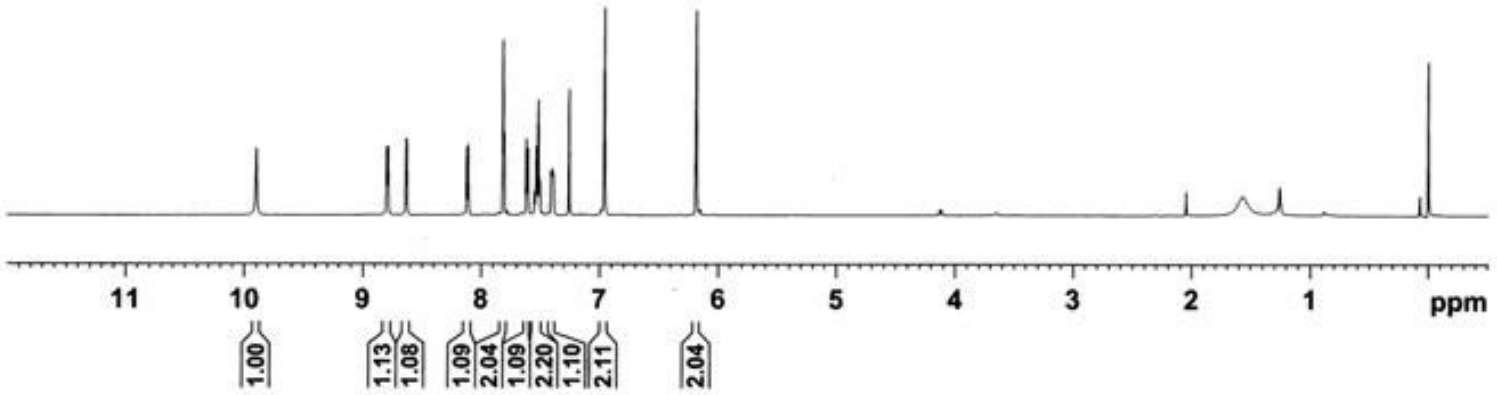
SP-797-4I-1H







2f

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EXPNO                    1
PROCNO                   1

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Time                   8.32
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PROBHD     5 mm PABBO BB/
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       16
DS                        2
SWH               12019.230 Hz
FIDRES             0.366798 Hz
AQ                 1.3631488 sec
RG                    143.33
DW                   41.600 usec
DE                    6.50 usec
TE                   295.3 K
D1                1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1            600.1737063 MHz
NUC1                    1H
P1                   12.00 usec
PLW1            21.00000000 W

F2 - Processing parameters
SI                       16384
SF              600.1700147 MHz
WDW                        EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
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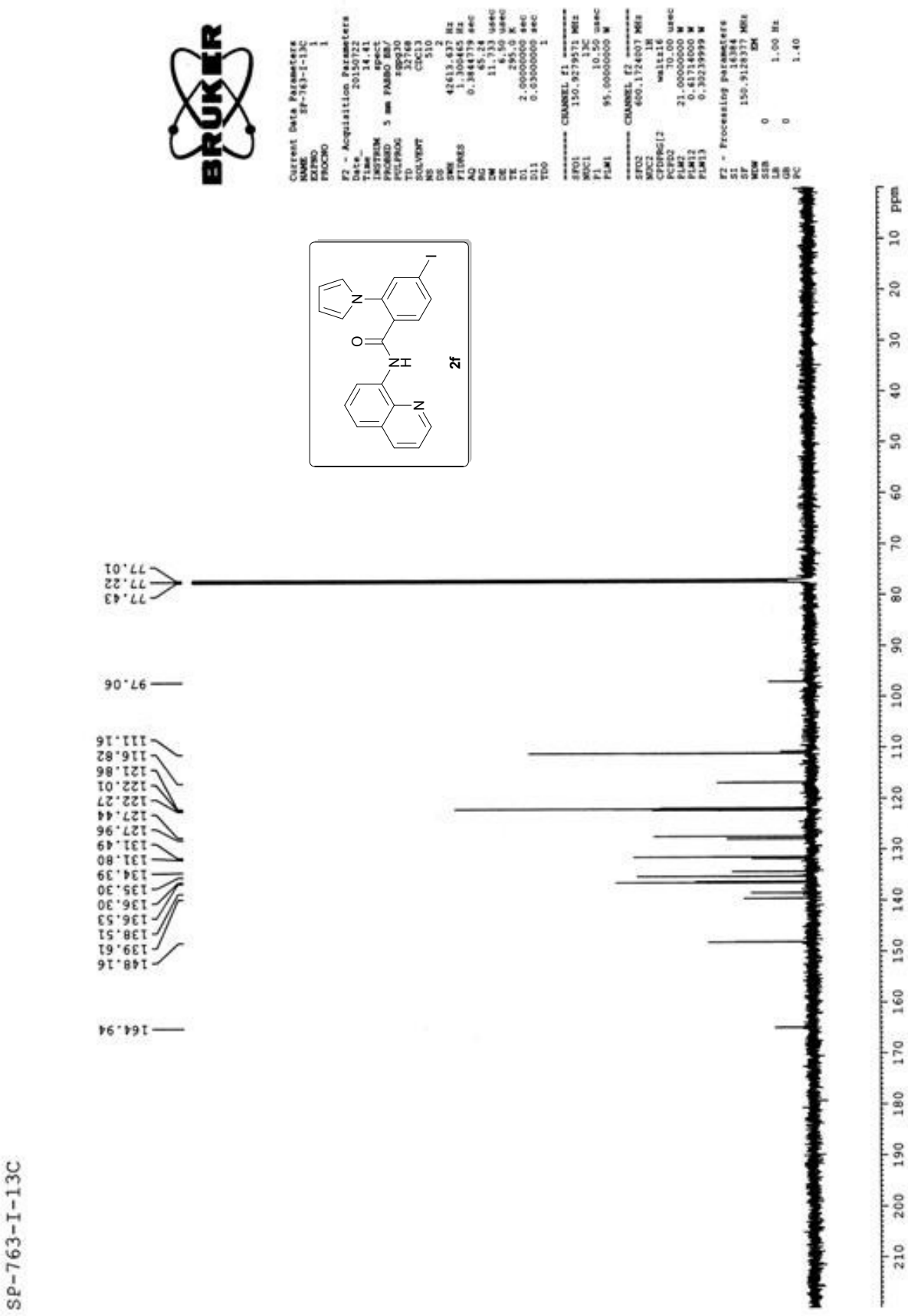
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121.86
122.01
122.27
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136.53
138.51
139.61
148.16
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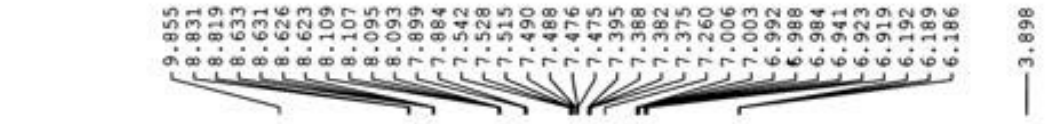
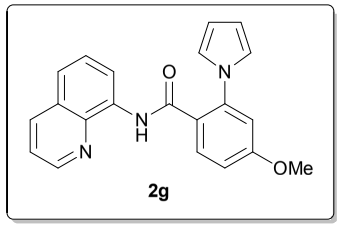
ppm

SP-676-1H

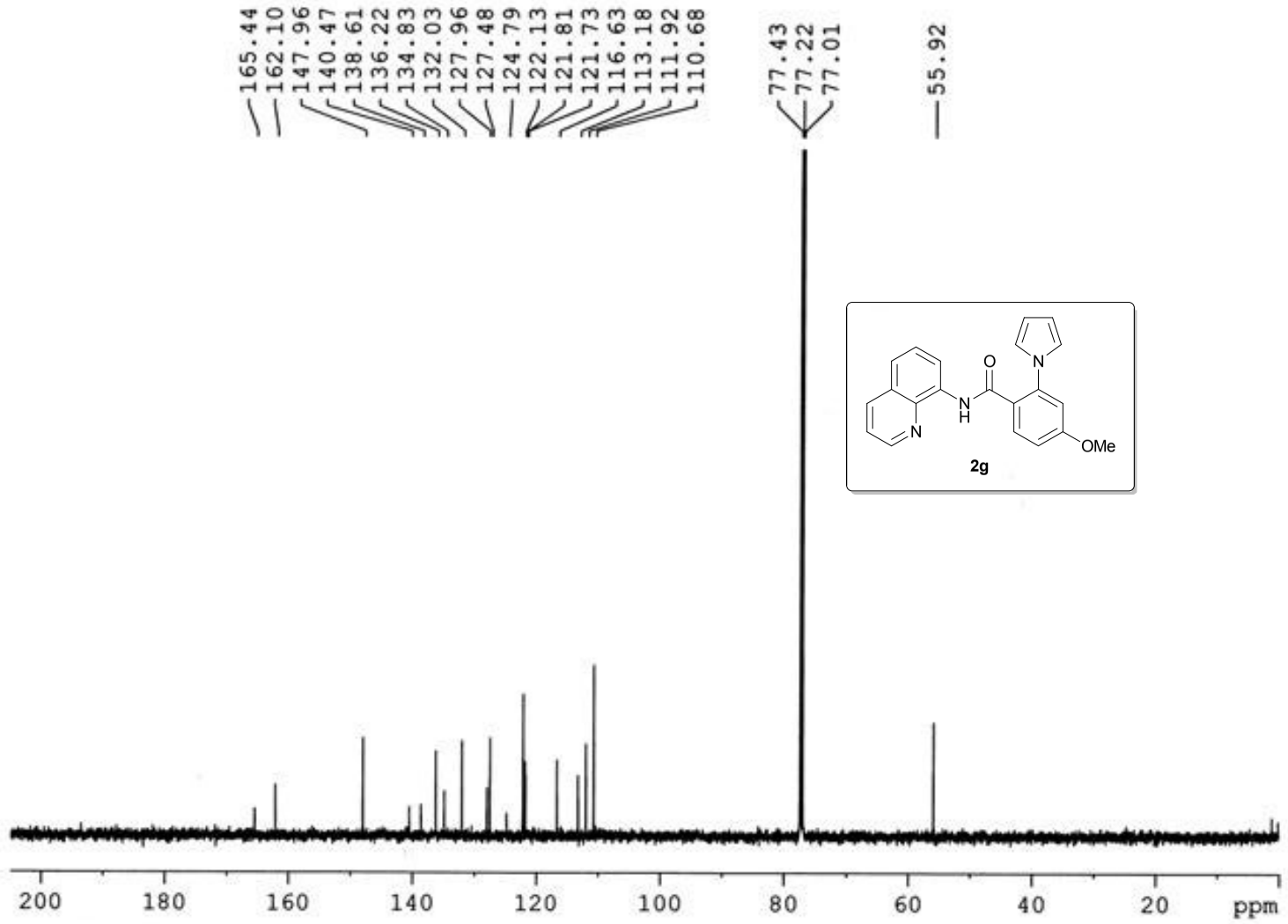




2g

```
Current Data Parameters
NAME                  SP-676-1H
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20150302
Time                      13.21
INSTRUM                   spect
PROBHD     5 mm PABBO BB/
PULPROG                   zg30
TD                       32768
SOLVENT                  CDCl3
NS                           16
DS                            2
SWH                12019.230 Hz
FIDRES              0.366798 Hz
AQ                  1.3631488 sec
RG                       99.36
DW                     41.600 usec
DE                      6.00 usec
TE                     296.9 K
D1                 1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1               600.1737063 MHz
NUC1                        1H
P1                       12.00 usec
PLW1               21.00000000 W

F2 - Processing parameters
SI                        16384
SF                 600.1700138 MHz
WDW                         EM
SSB                  0
LB                        0.30 Hz
GB                   0
PC                        1.00
```

SP-676-13C



2g

BRUKER

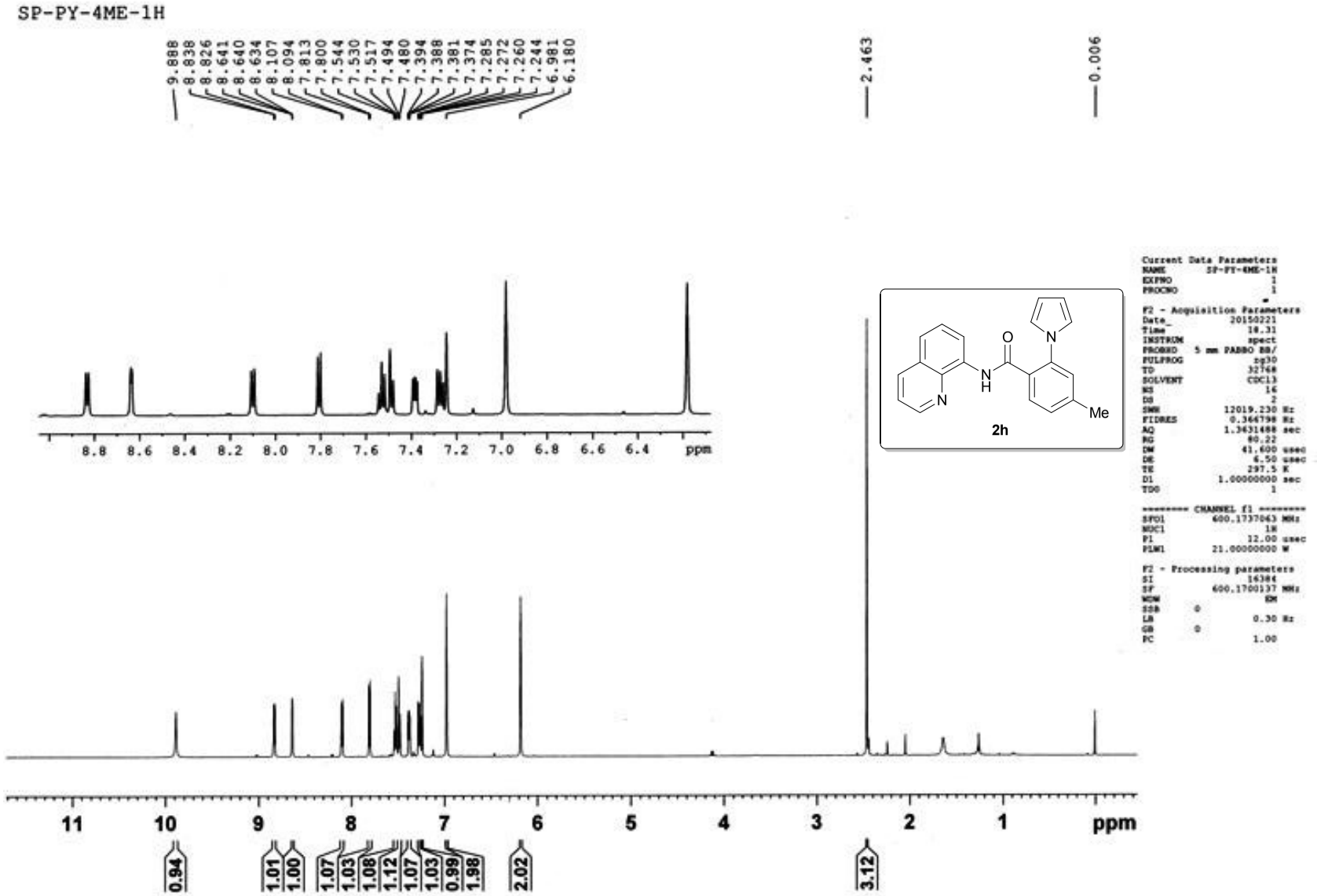
Current Data Parameters
NAME SP-676-13C
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150302
Time 19.09
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 158
DS 2
SWH 36057.691 Hz
FIDRES 1.100393 Hz
AQ 0.4543829 sec
RG 65.24
DW 13.867 usec
DE 6.50 usec
TE 297.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

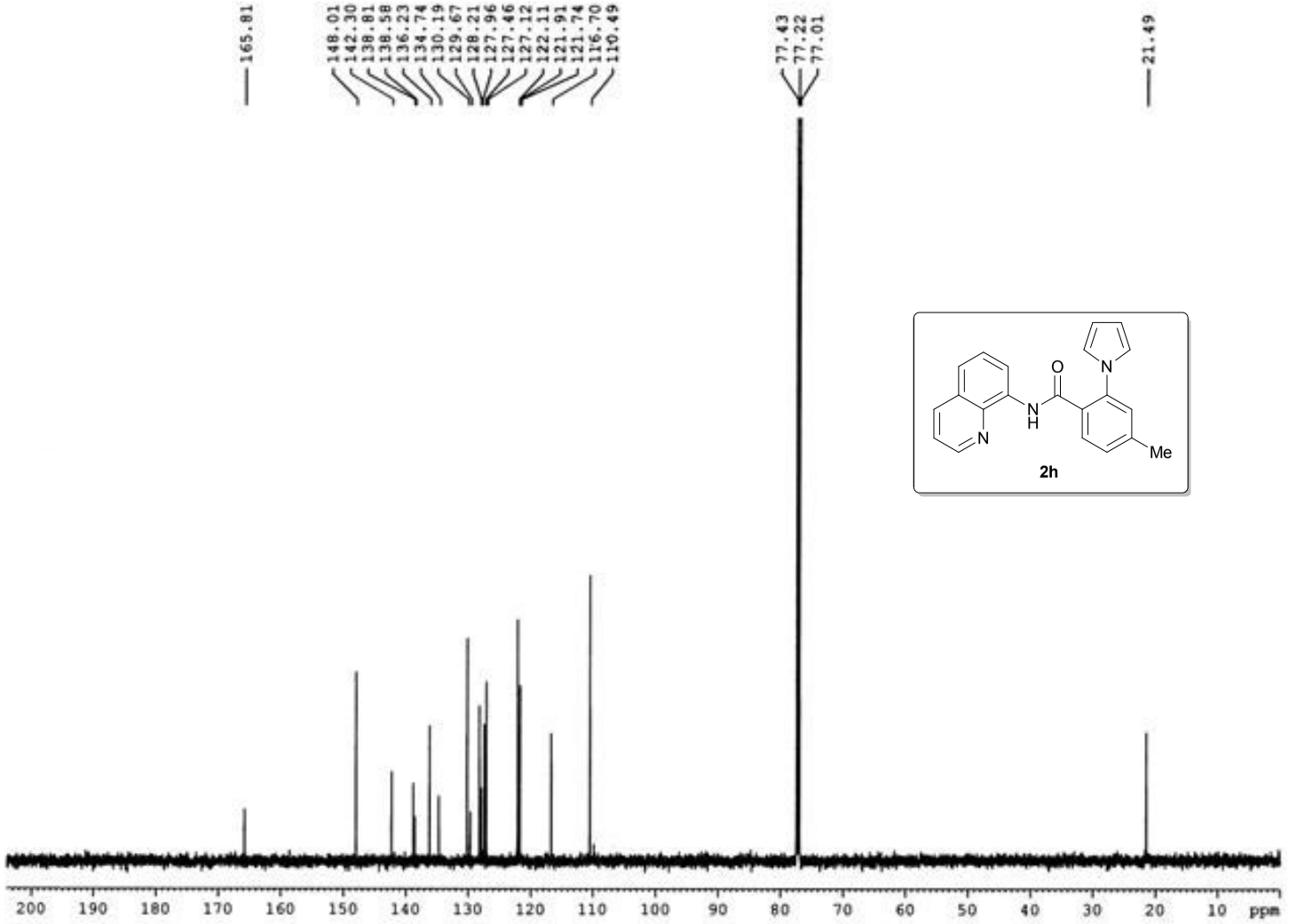
======== CHANNEL f1 ========
SFO1 150.9279571 MHz
NUC1 13C
P1 10.50 usec
PLW1 95.00000000 W

======== CHANNEL f2 ========
SFO2 600.1724007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 21.00000000 W
PLW12 0.61714000 W
PLW13 0.30239999 W

F2 - Processing parameters
SI 16384
SF 150.9128374 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

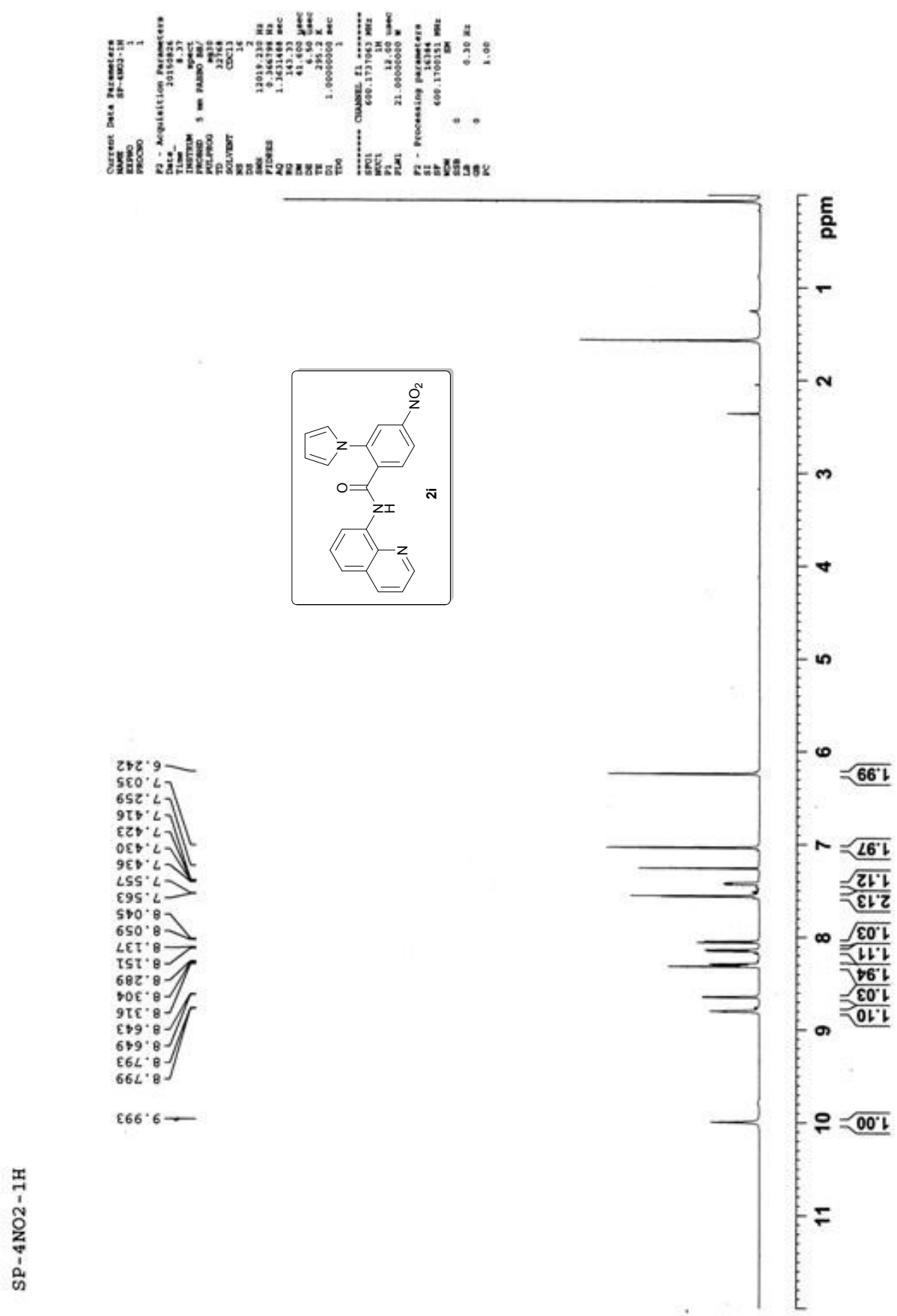

SP-PY-4ME-1H
9.888
8.838
8.826
8.641
8.640
8.634
8.107
8.094
7.813
7.800
7.544
7.530
7.517
7.494
7.480
7.394
7.388
7.381
7.374
7.285
7.272
7.260
7.244
6.981
6.180
2.463
0.006
2h
Current Data Parameters
NAME SP-PY-4ME-1H
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150221
Time 18.31
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 80.22
DW 41.600 usec
DE 6.50 usec
TE 297.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 600.1737063 MHz
NUC1 1H
P1 12.00 usec
PLW1 21.00000000 W
F2 - Processing parameters
SI 16384
SF 600.1700137 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.94
1.01
1.00
1.07
1.03
1.08
1.12
1.07
1.03
0.99
1.98
2.02
3.12
ppm

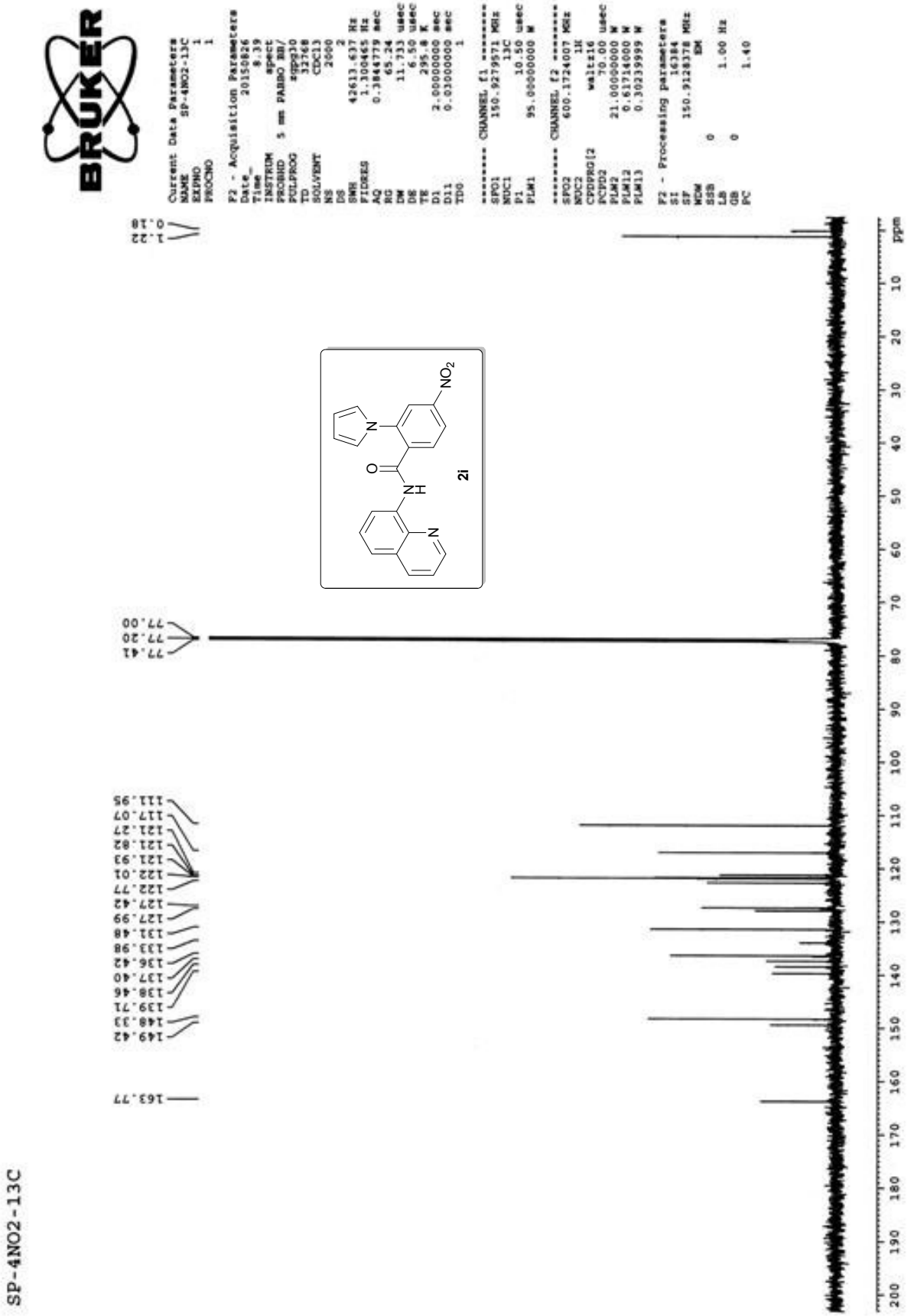
SP-PY-4ME-13C



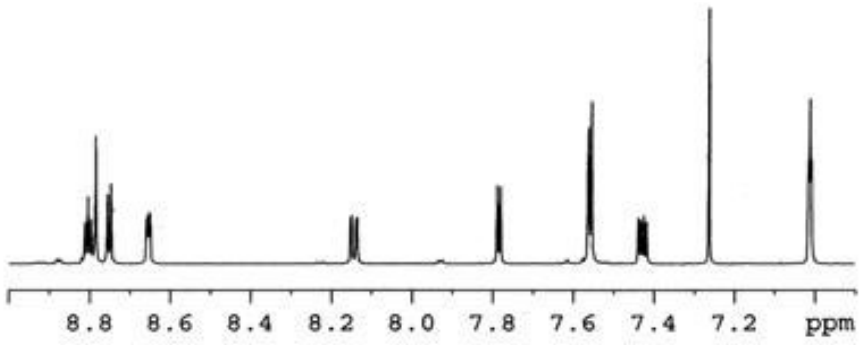
2h

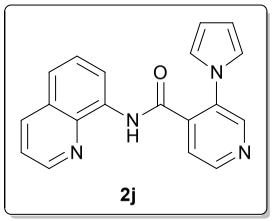
```
Current Data Parameters
NAME        SP-PY-4ME-13C
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20150221
Time                18.38
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG             zgpg30
TD                  32768
SOLVENT             CDCl3
NS                    115
DS                      2
SWH             36057.691 Hz
FIDRES          1.100393 Hz
AQ              0.4543829 sec
RG                 200.18
DW                 13.867 usec
DE                   6.50 usec
TE                 298.4 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          150.9279571 MHz
NUC1                  13C
P1                  10.50 usec
PLW1         95.00000000 W

======== CHANNEL f2 ========
SFO2          600.1724007 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2               70.00 usec
PLW2         21.00000000 W
PLW12         0.61714000 W
PLW13         0.30239999 W

F2 - Processing parameters
SI                    16384
SF            150.9128397 MHz
WDW                    EM
SSB          0
LB                  1.00 Hz
GB           0
PC                  1.40
```


SP-4NO2-1H
2i
ppm


SP-4NO2-13C
2i
Current Data Parameters
NAME SP-4NO2-13C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150826
Time_ 8.39
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 2600
DS 2
SWH 42613.637 Hz
FIDRES 1.300465 Hz
AQ 0.3844779 sec
RG 65.24
DW 11.733 usec
DE 6.50 usec
TE 295.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 150.9279571 MHz
NUC1 13C
P1 10.50 usec
PLW1 95.00000000 W
======== CHANNEL f2 ========
SFO2 600.1724007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 21.00000000 W
PLW12 0.61714000 W
PLW13 0.30239999 W
F2 - Processing parameters
SI 16384
SF 150.9128378 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
0.18
1.22
77.00
77.20
77.41
111.95
117.07
121.27
121.82
121.93
122.01
122.77
127.42
127.99
131.48
133.98
136.42
137.40
138.46
139.71
148.33
149.42
163.77
ppm

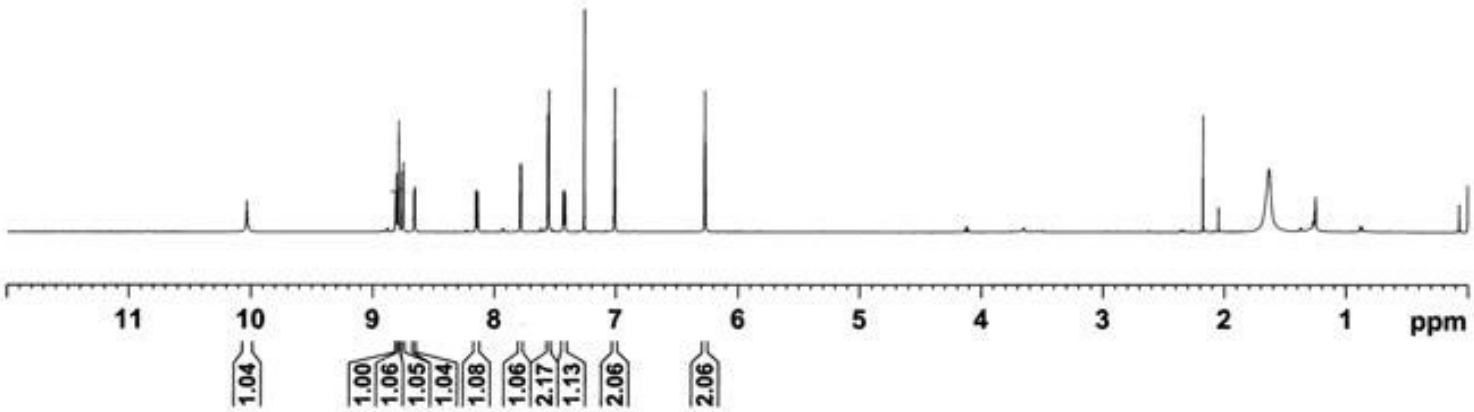
SP-801-A_1H



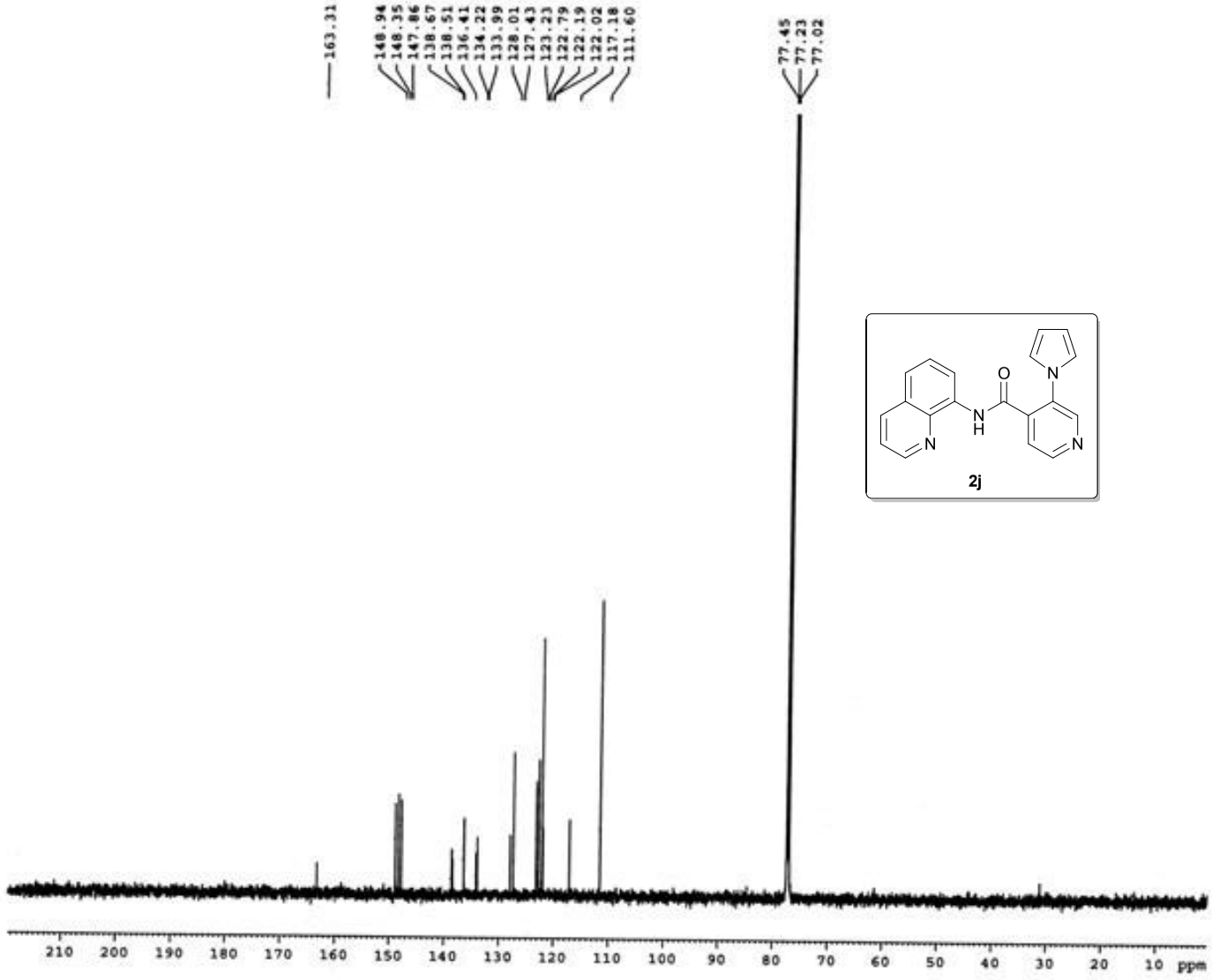


2j

```
Current Data Parameters
NAME            SP-801-A_1H
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20150911
Time                  12.36
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      16
DS                       2
SWH              12019.230 Hz
FIDRES            0.366798 Hz
AQ                1.3631488 sec
RG                   143.33
DW                 41.600 usec
DE                  6.50 usec
TE                  293.8 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1           600.1737063 MHz
NUC1                     1H
P1                    12.00 usec
PLW1           21.00000000 W

F2 - Processing parameters
SI                    16384
SF             600.1700126 MHz
WDW                      EM
SSB          0
LB                     0.30 Hz
GB           0
PC                     1.00
```



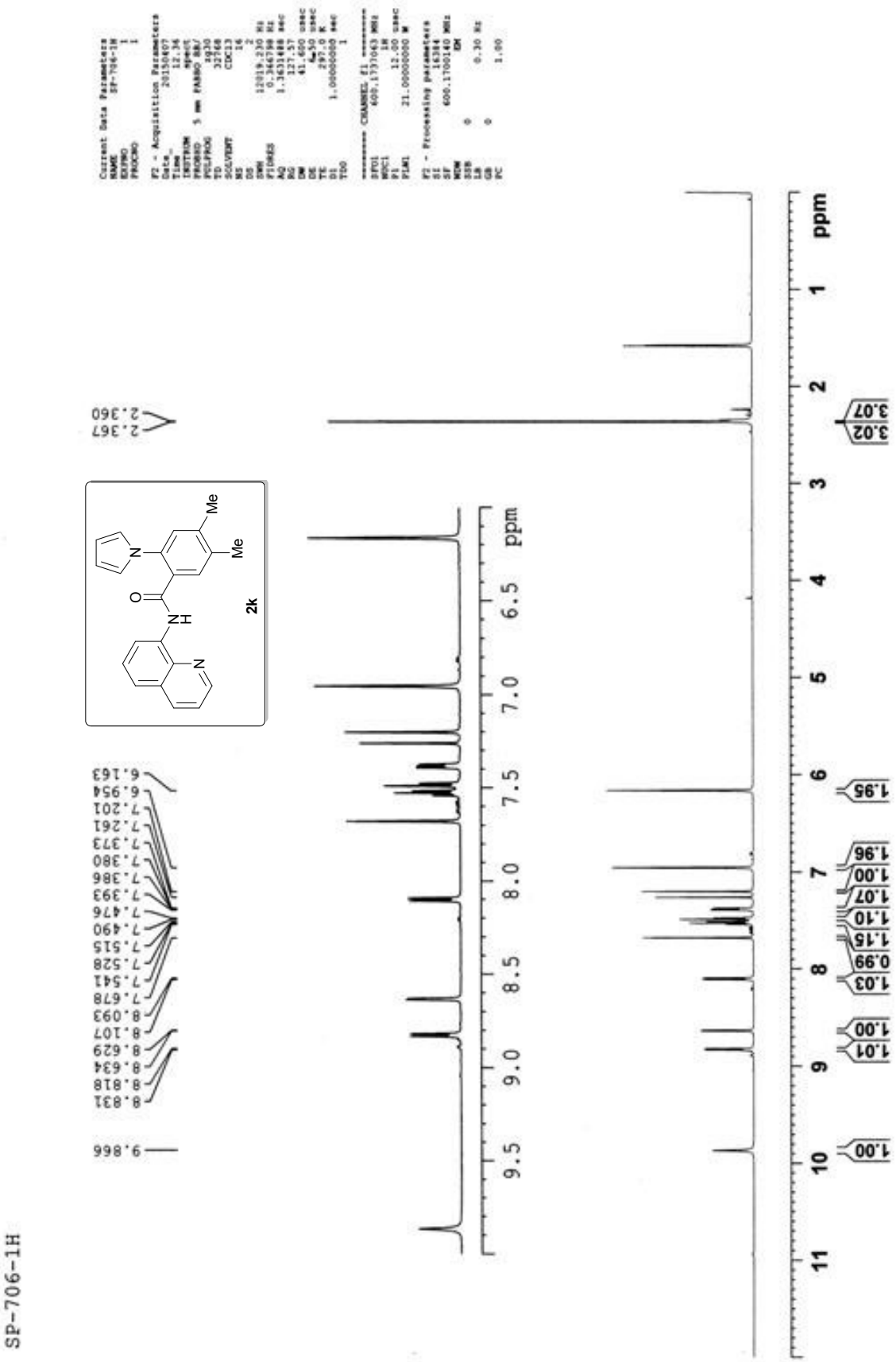
SP-801-A_13C

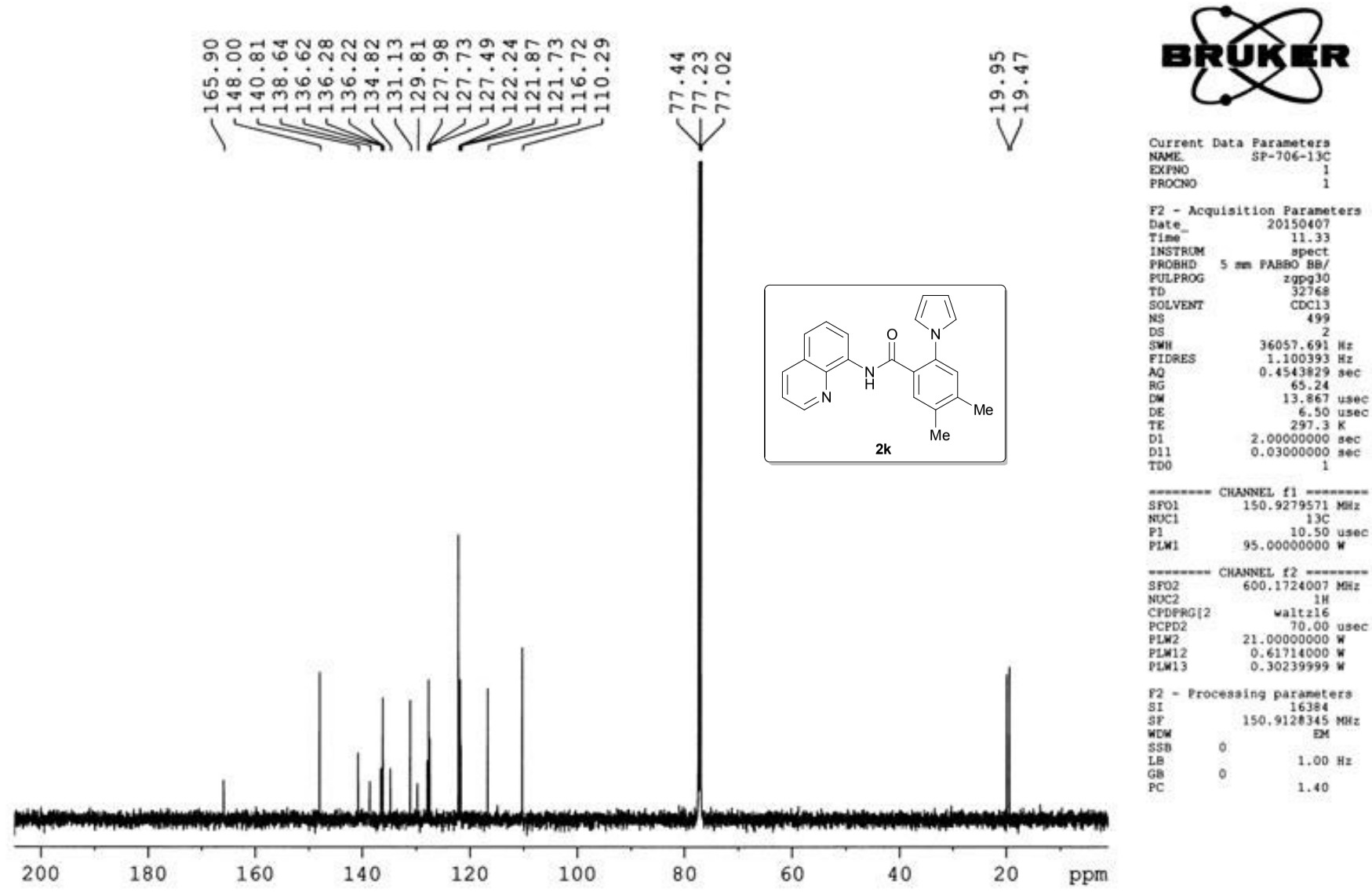


2j

BRUKER

```
Current Data Parameters
NAME          SP-801-A_13C
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_              20150911
Time                  12.38
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG             zgpg30
TD                   32768
SOLVENT              CDCl3
NS                    2000
DS                       2
SWH              36057.691 Hz
FIDRES           1.100393 Hz
AQ               0.4543829 sec
RG                   65.24
DW                 13.867 usec
DE                  6.50 usec
TE                 294.2 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          150.9279571 MHz
NUC1                   13C
P1                   10.50 usec
PLW1          95.00000000 W

======== CHANNEL f2 ========
SFO2          600.1724007 MHz
NUC2                   1H
CPDPRG[2           waltz16
PCPD2                70.00 usec
PLW2          21.00000000 W
PLW12          0.61714000 W
PLW13          0.30239999 W

F2 - Processing parameters
SI                    16384
SF            150.9128346 MHz
WDW                      EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```


SP-706-1H
Current Data Parameters
NAME SP-706-1H
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Data_ 20150407
Time 12.34
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 127.57
DW 41.600 usec
DE 6.50 usec
TE 297.0 K
D1 1.00000000 sec
TD0 1
---------- CHANNEL f1 ----------
SFO1 600.1737043 MHz
NUC1 1H
P1 12.00 usec
PLW1 21.00000000 W
F2 - Processing parameters
SI 16384
SF 600.1700140 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
Me
N
O
NH
N
2k
9.866
8.831
8.818
8.634
8.629
8.107
8.093
7.678
7.541
7.528
7.515
7.490
7.476
7.393
7.386
7.380
7.373
7.261
7.201
6.954
6.163
2.367
2.360
ppm
3.07
3.02
1.95
1.96
1.00
1.07
1.10
1.15
0.99
1.03
1.00
1.01
1.00



BRUKER

Current Data Parameters
NAME SP-706-13C
EXPNO 1
PROCNO 1

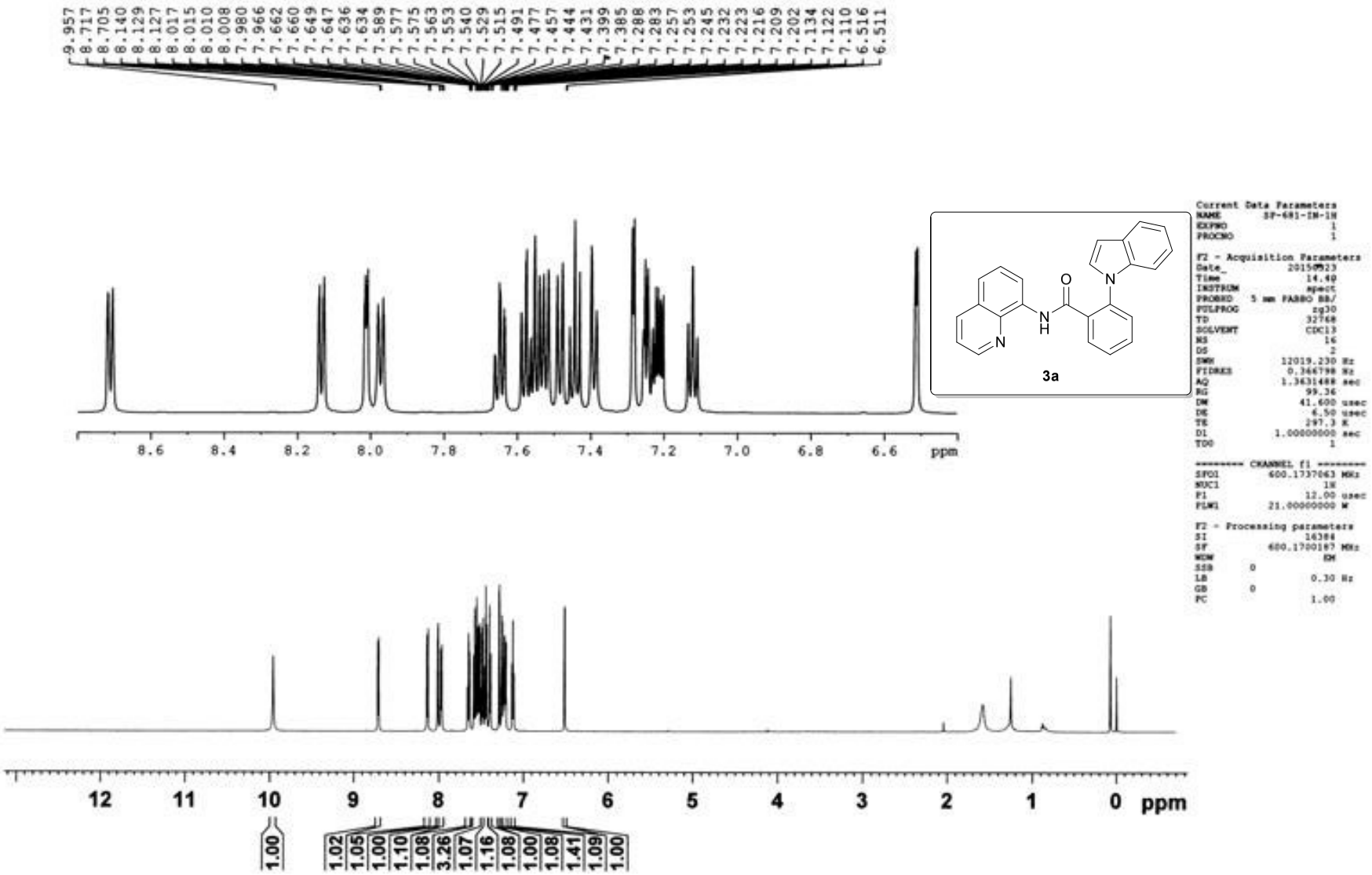
F2 - Acquisition Parameters
Date_ 20150407
Time 11.33
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 499
DS 2
SWH 36057.691 Hz
FIDRES 1.100393 Hz
AQ 0.4543829 sec
RG 65.24
DW 13.867 usec
DE 6.50 usec
TE 297.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9279571 MHz
NUC1 13C
P1 10.50 usec
PLW1 95.00000000 W

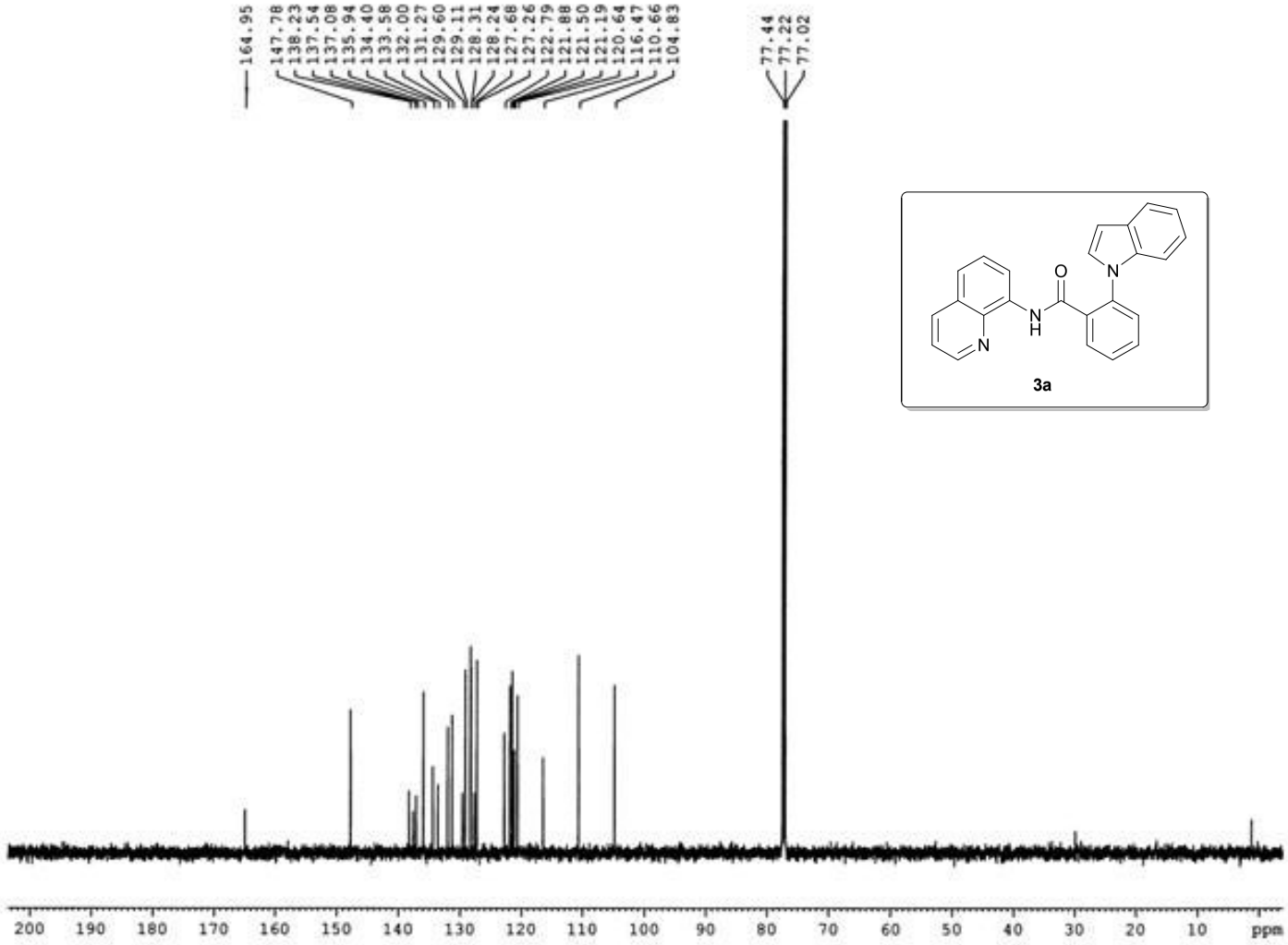
======== CHANNEL f2 ========
SFO2 600.1724007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 21.00000000 W
PLW12 0.61714000 W
PLW13 0.30239999 W

F2 - Processing parameters
SI 16384
SF 150.9128345 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

SP-681-IN-1H

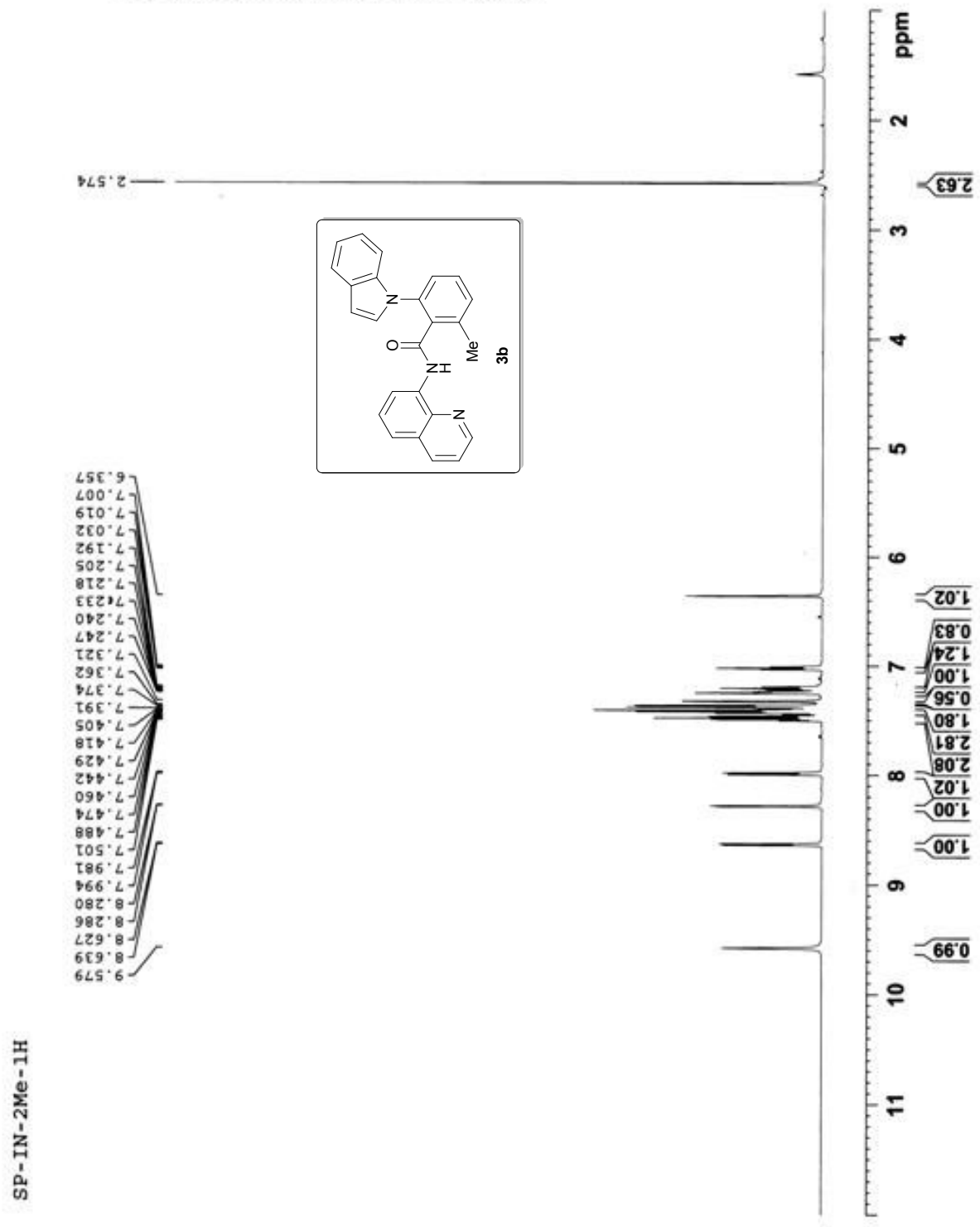

9.957
8.717
8.705
8.140
8.129
8.127
8.017
8.015
8.010
8.008
7.980
7.966
7.662
7.660
7.649
7.647
7.636
7.634
7.589
7.577
7.575
7.563
7.553
7.540
7.529
7.515
7.491
7.477
7.457
7.444
7.431
7.399
7.385
7.288
7.283
7.257
7.253
7.245
7.232
7.223
7.216
7.209
7.202
7.134
7.122
7.110
6.516
6.511
8.6
8.4
8.2
8.0
7.8
7.6
7.4
7.2
7.0
6.8
6.6
ppm
O
N
H
N
N
3a
Current Data Parameters
NAME SP-681-IN-1H
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150923
Time 14.40
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 99.36
DW 41.600 usec
DE 6.50 usec
TE 297.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 600.1737063 MHz
NUC1 1H
P1 12.00 usec
PLW1 21.00000000 W
F2 - Processing parameters
SI 16384
SF 600.1700187 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.02
1.05
1.00
1.10
1.08
3.26
1.07
1.16
1.08
1.00
1.08
1.41
1.09
1.00

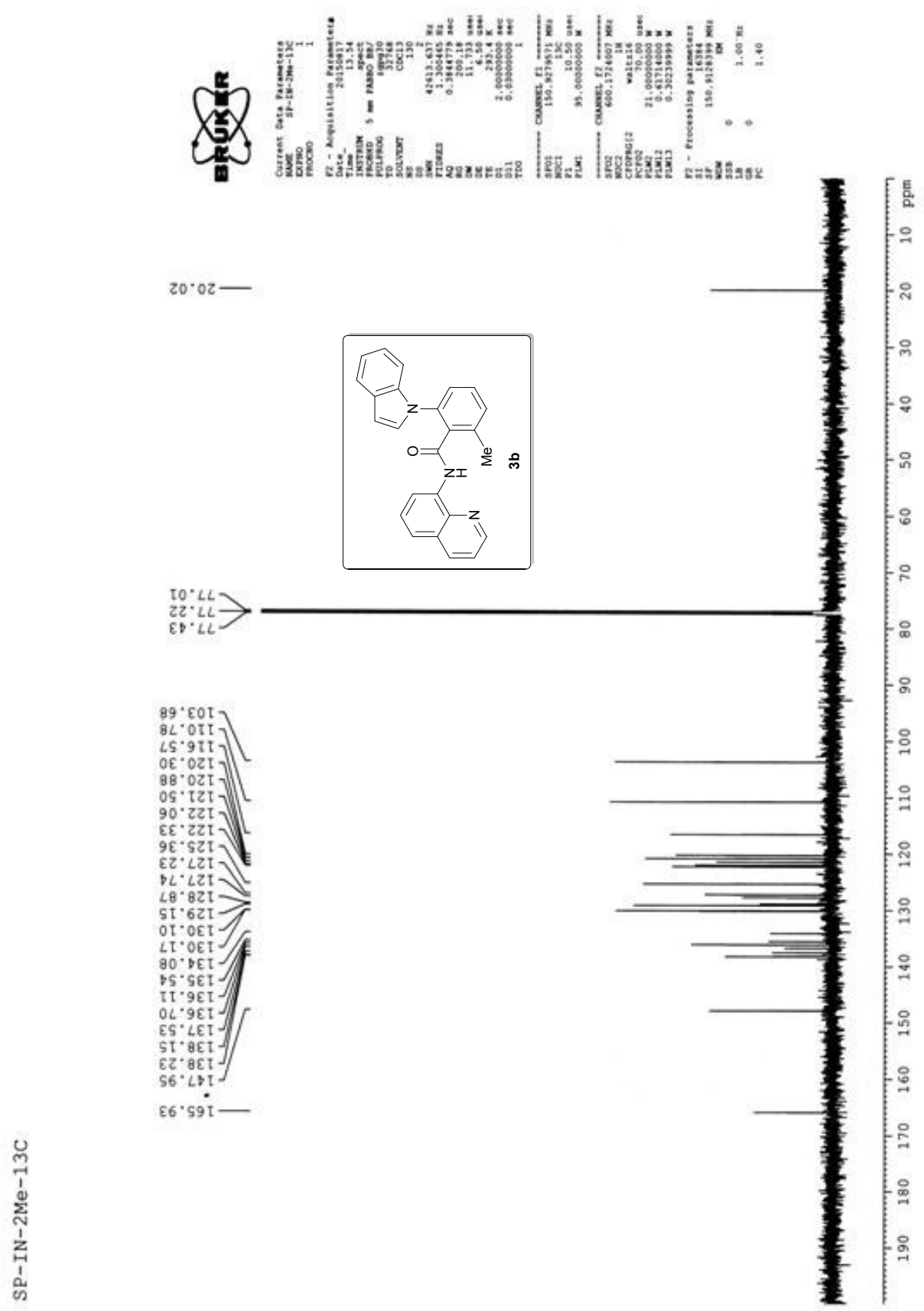
SP-681-In_13C



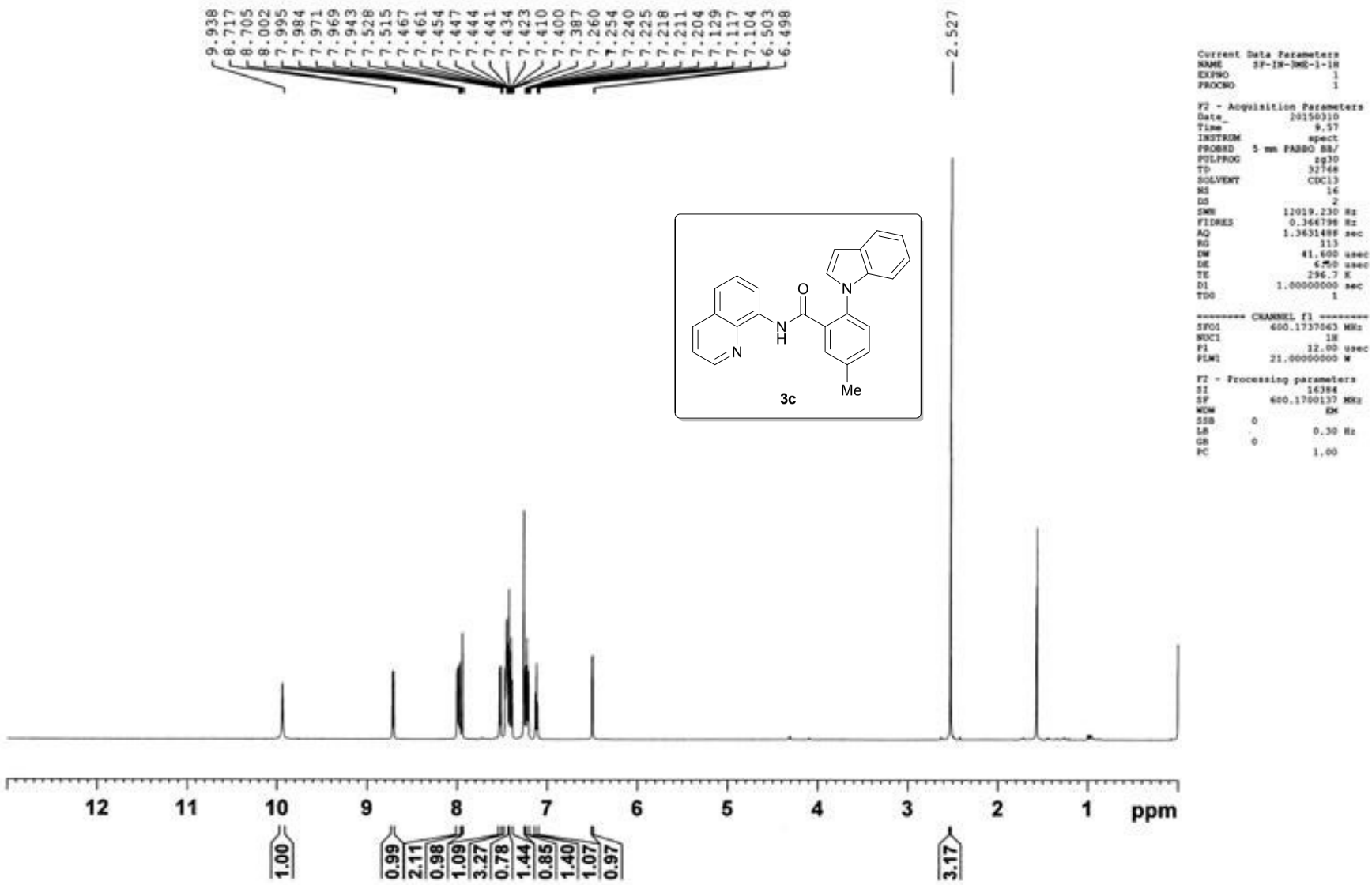
3a

```
Current Data Parameters
NAME       SP-681-In_13C
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20150401
Time              12.08
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               32768
SOLVENT          CDCl3
NS                 235
DS                   2
SWH          36057.691 Hz
FIDRES        1.100393 Hz
AQ            0.4543829 sec
RG               200.18
DW               13.867 usec
DE                6.50 usec
TE                298.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1       150.9279571 MHz
NUC1               13C
P1               10.50 usec
PLW1       95.00000000 W

======== CHANNEL f2 ========
SFO2       600.1724007 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            70.00 usec
PLW2       21.00000000 W
PLW12       0.61714000 W
PLW13       0.30239999 W

F2 - Processing parameters
SI                16384
SF         150.9128380 MHz
WDW                EM
SSB       0
LB               1.00 Hz
GB        0
PC               1.40
```

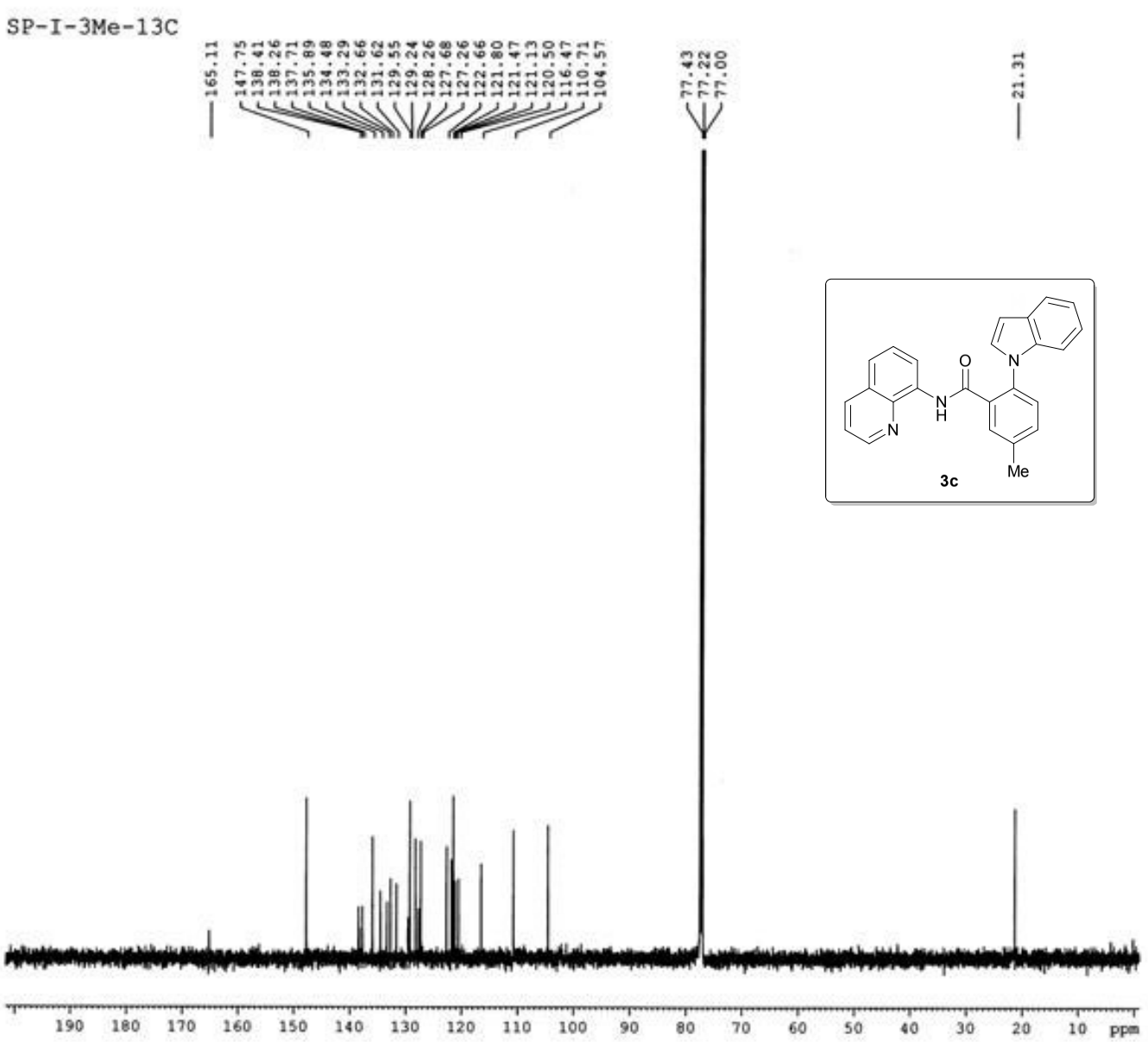

SP-IN-2Me-1H
ppm
3b
Me
N
O
H
2.574
2.63
1.02
0.83
1.24
1.00
0.56
1.80
2.81
2.08
1.02
1.00
1.00
0.99
6.357
7.007
7.019
7.032
7.192
7.205
7.218
7.233
7.240
7.247
7.321
7.362
7.374
7.391
7.405
7.418
7.429
7.442
7.460
7.474
7.488
7.501
7.981
7.994
8.280
8.286
8.627
8.639
9.579


SP-IN-2Me-13C
3b
Me
N
O
NH
N
20.02
77.43
77.22
77.01
103.68
110.78
116.57
120.30
120.88
121.50
122.06
122.33
125.36
127.23
127.74
128.87
129.15
130.10
130.17
134.08
135.54
136.11
136.70
137.53
138.15
138.23
147.95
165.93
ppm

SP-IN-3ME-1-1H




```
Current Data Parameters
NAME          SP-IN-3ME-1-1H
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_               20150310
Time                    9.57
INSTRUM                spect
PROBHD   5 mm PABBO BB/
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                12019.230 Hz
FIDRES              0.366796 Hz
AQ                  1.3631488 sec
RG                      111
DW                   41.600 usec
DE                    6.50 usec
TE                   296.7 K
D1                  1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1             600.1737063 MHz
NUC1                      1H
P1                     12.00 usec
PLW1            21.00000000 W

F2 - Processing parameters
SI                        16384
SF               600.1700137 MHz
WDW                       EM
SSB          0
LB                      0.30 Hz
GB           0
PC                      1.00
```



BRUKER

Current Data Parameters
NAME SP-I-3Me-13C
EXPNO 1
PROCNO 1

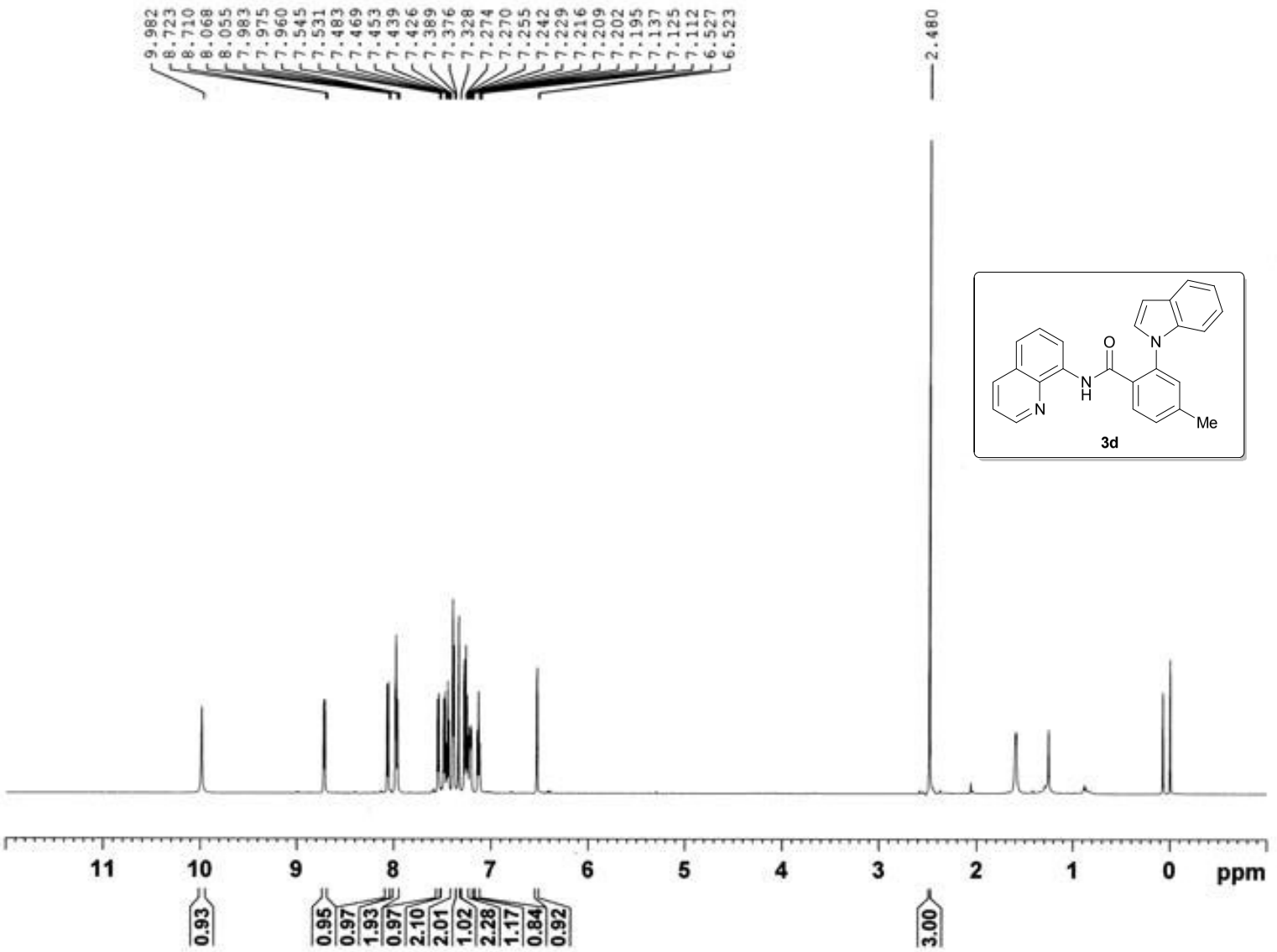
F2 - Acquisition Parameters
Date_ 20150320
Time 10.39
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 529
DS 2
SWH 36057.691 Hz
FIDRES 1.100393 Hz
AQ 0.4543829 sec
RG 65.24
DW 13.867 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9279571 MHz
NUC1 13C
P1 10.50 usec
PLW1 95.00000000 W

======== CHANNEL f2 ========
SFO2 600.1724007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 21.00000000 W
PLW12 0.61714000 W
PLW13 0.30239999 W

F2 - Processing parameters
SI 16384
SF 150.9128360 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

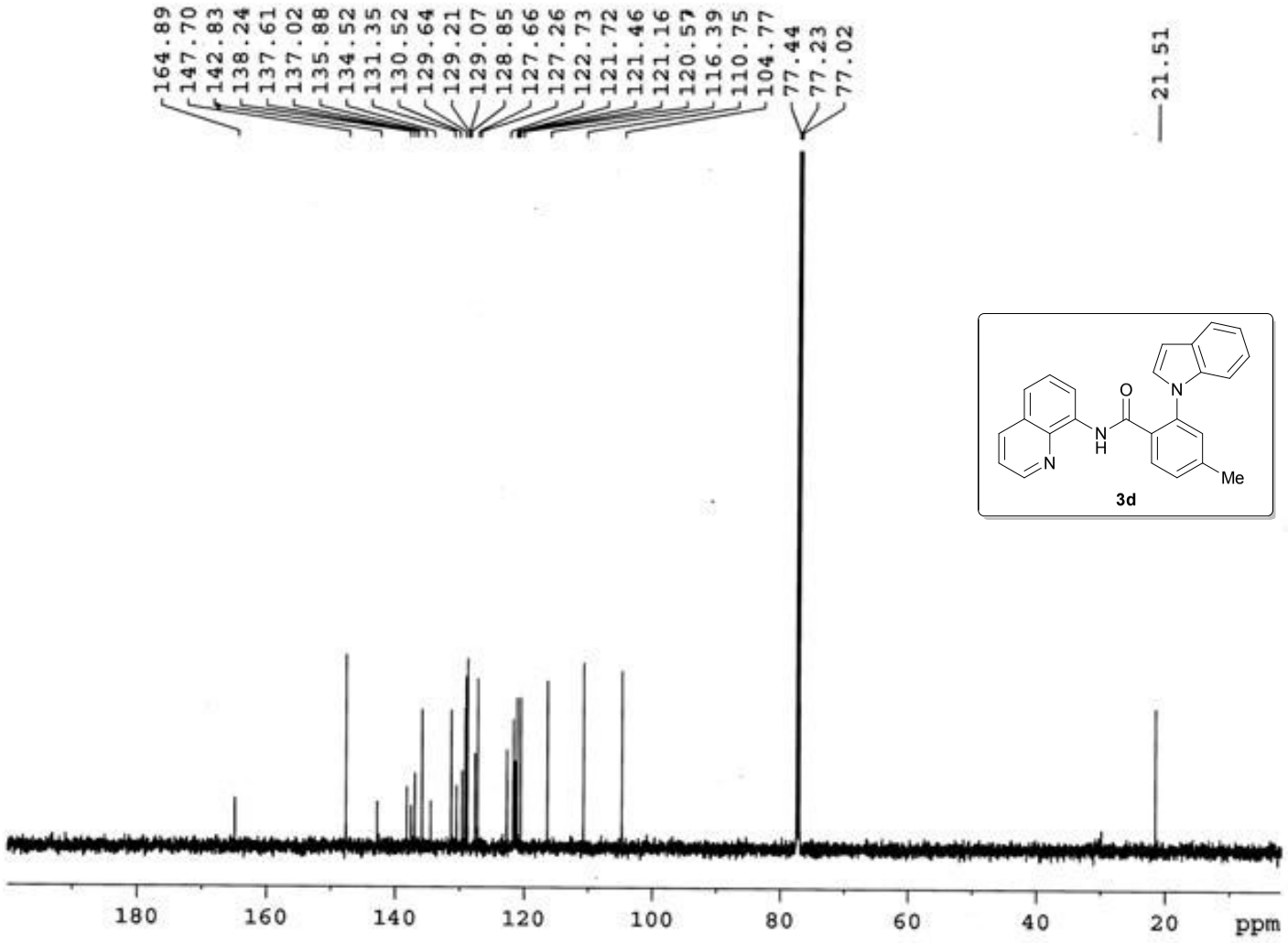
SP-682-1-1H



3d

```
Current Data Parameters
NAME                  SP-682-1-1H
EXPNO                           1
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20150803
Time                        13.51
INSTRUM                      spect
PROBHD    5 mm PABBO BB/
PULPROG                     zg30
TD                         32768
SOLVENT                    CDCl3
NS                            16
DS                             2
SWH                    12019.230 Hz
FIDRES                  0.366798 Hz
AQ                      1.3631488 sec
RG                          89.67
DW                         41.600 usec
DE                          6.50 usec
TE                         293.3 K
D1                      1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1                 600.1737063 MHz
NUC1                         1H
P1                         12.00 usec
PLW1                21.00000000 W

F2 - Processing parameters
SI                         16384
SF                   600.1700172 MHz
WDW                           EM
SSB                 0
LB                          0.30 Hz
GB                  0
PC                          1.00
```

SP-682-1-13C



3d

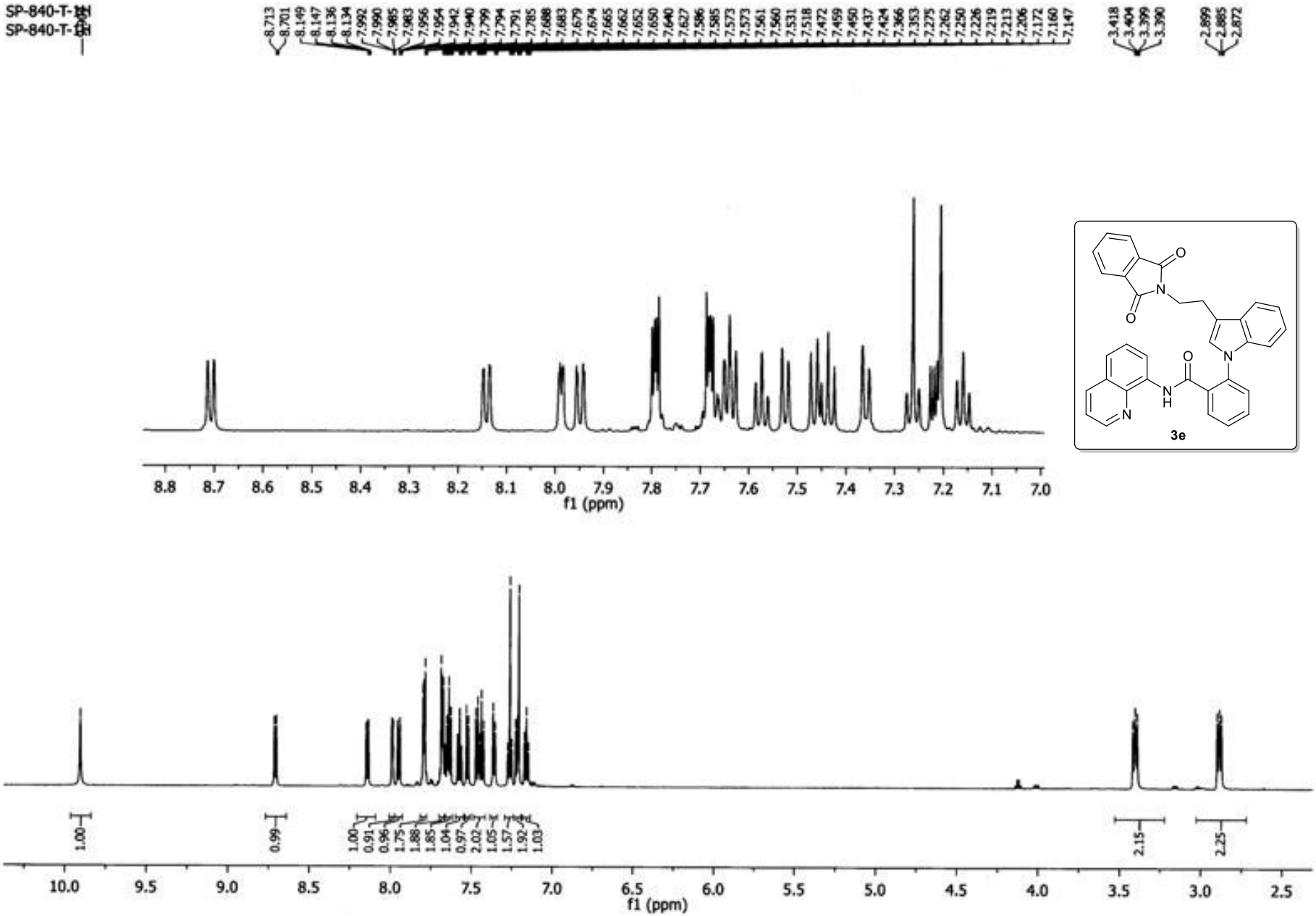
Current Data Parameters
NAME SP-682-1-13C
EXPNO 1
PROCNO 1

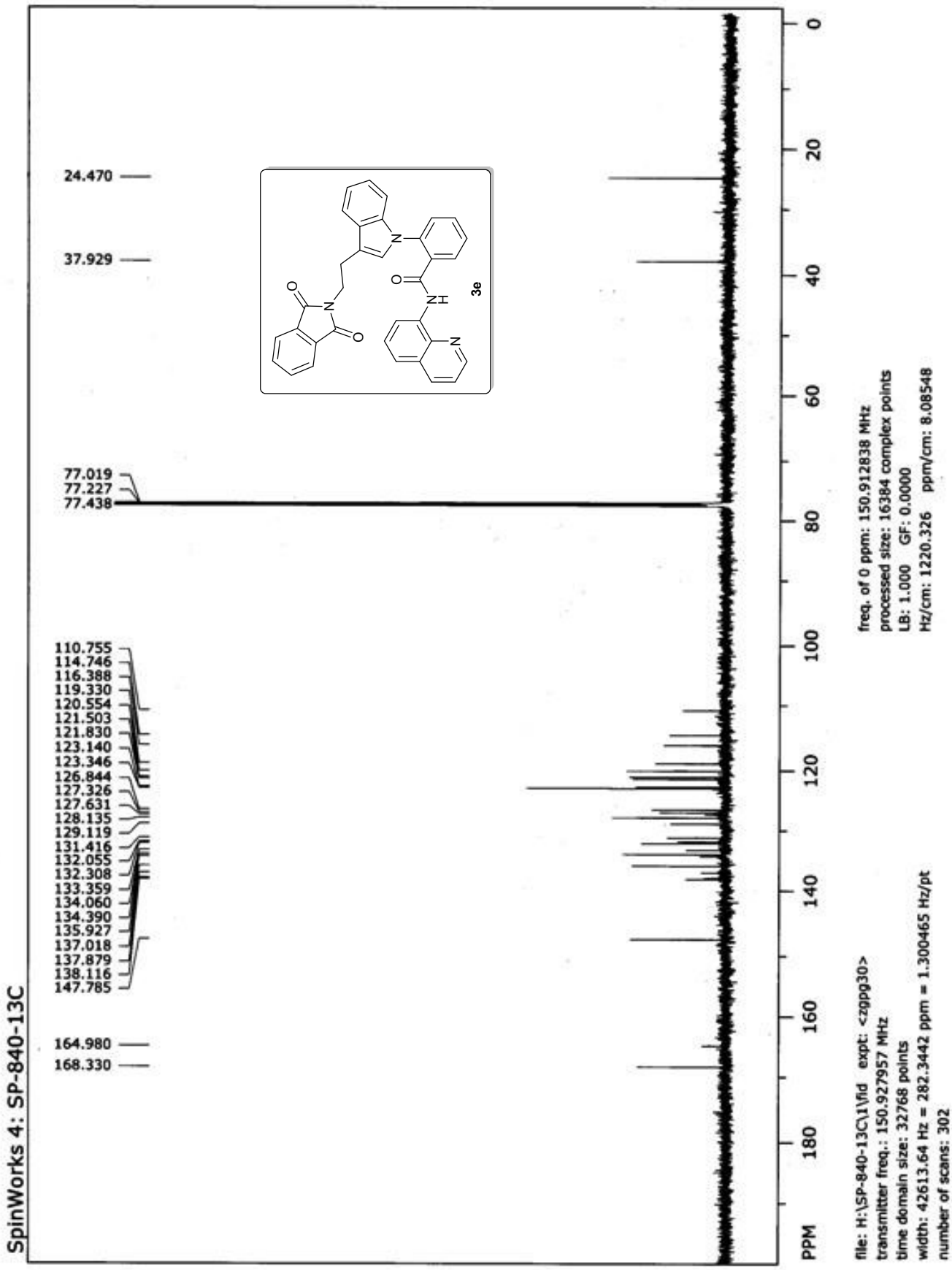
F2 - Acquisition Parameters
Date_ 20150902
Time 9.47
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 242
DS 2
SWH 42613.637 Hz
FIDRES 1.300465 Hz
AQ 0.3844779 sec
RG 65.24
DW 11.733 usec
DE 6.50 usec
TE 293.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9279571 MHz
NUC1 13C
P1 10.50 usec
PLW1 95.00000000 W

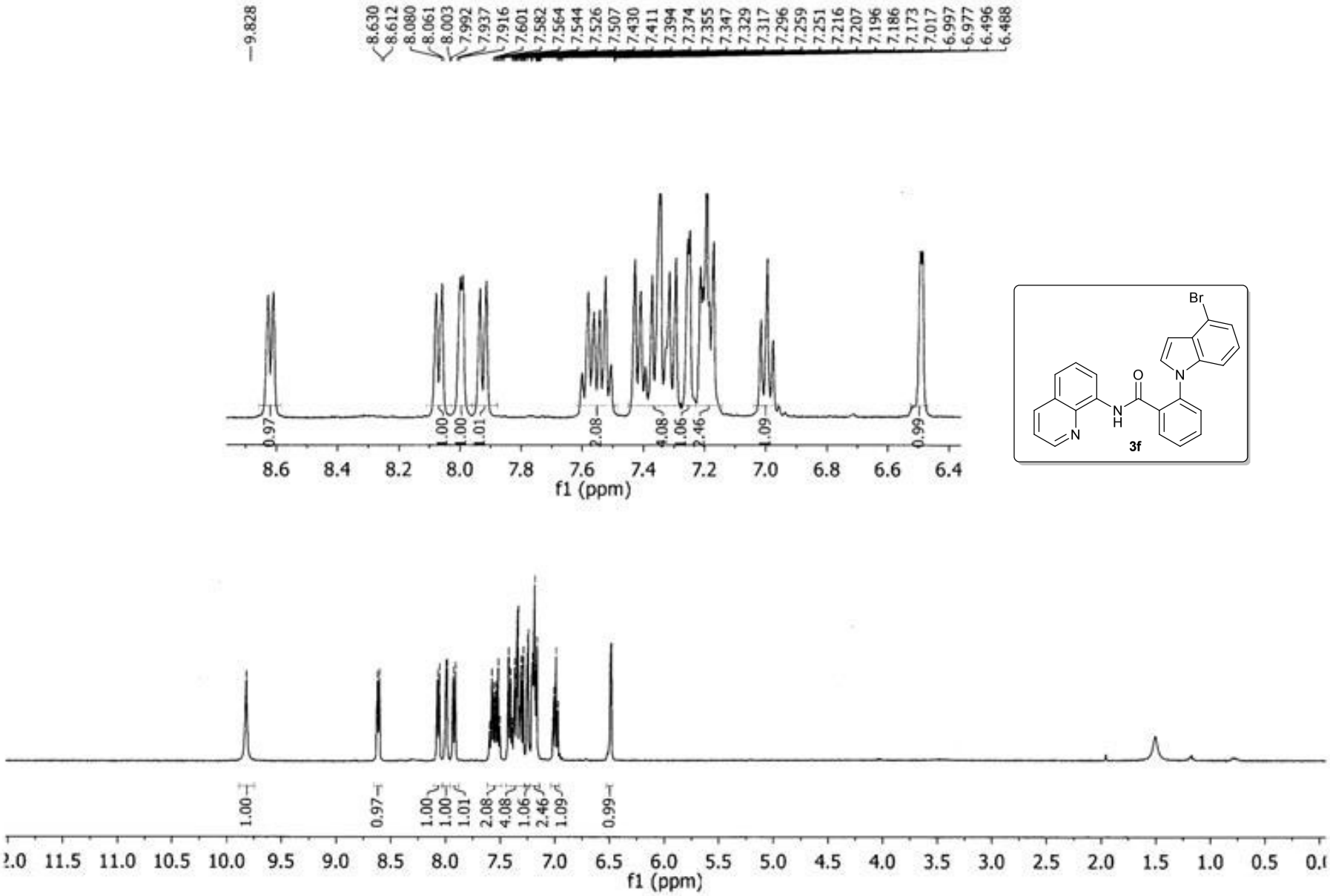
======== CHANNEL f2 ========
SFO2 600.1724007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 21.00000000 W
PLW12 0.61714000 W
PLW13 0.30239999 W

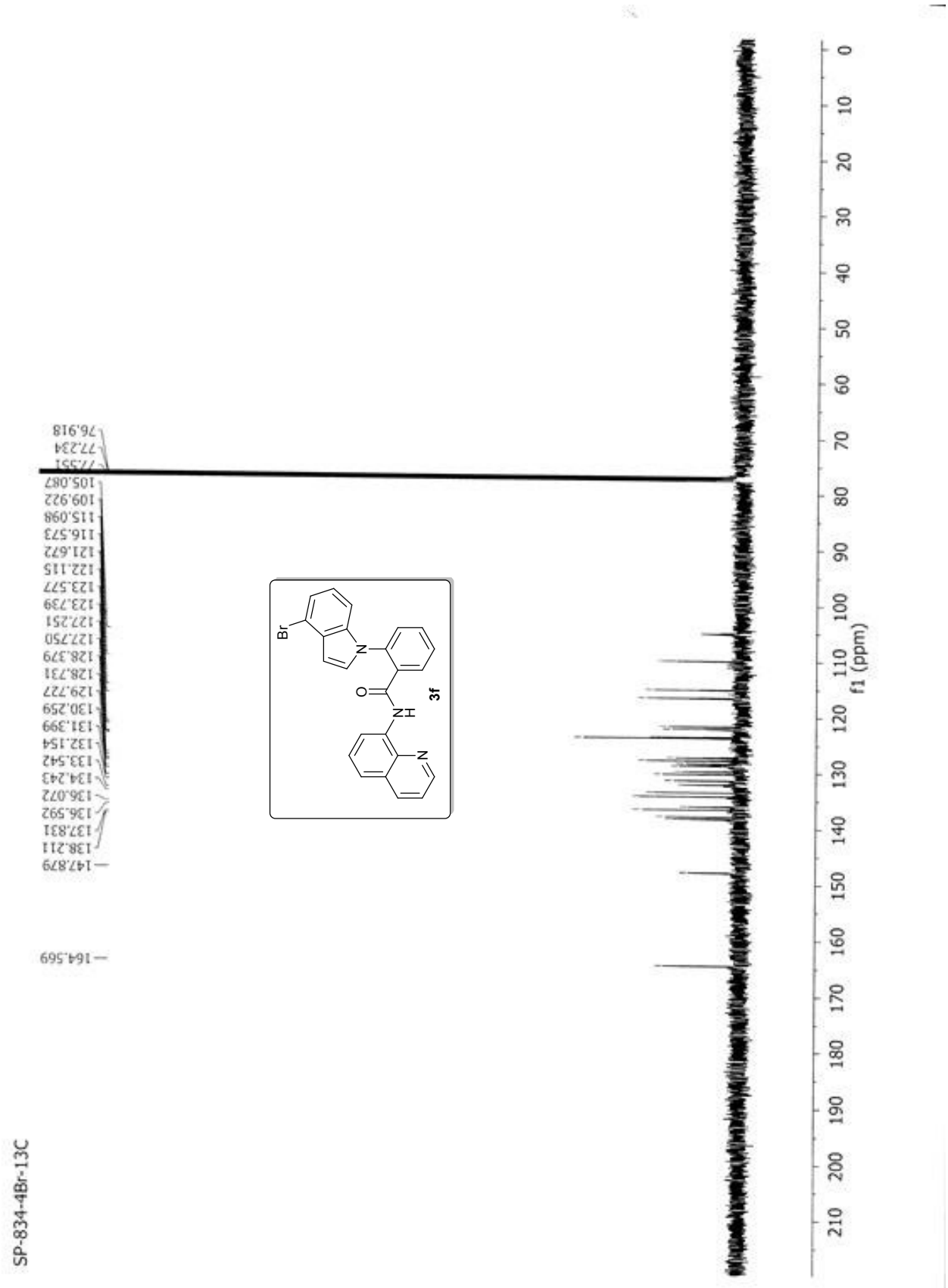
F2 - Processing parameters
SI 16384
SF 150.9128390 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

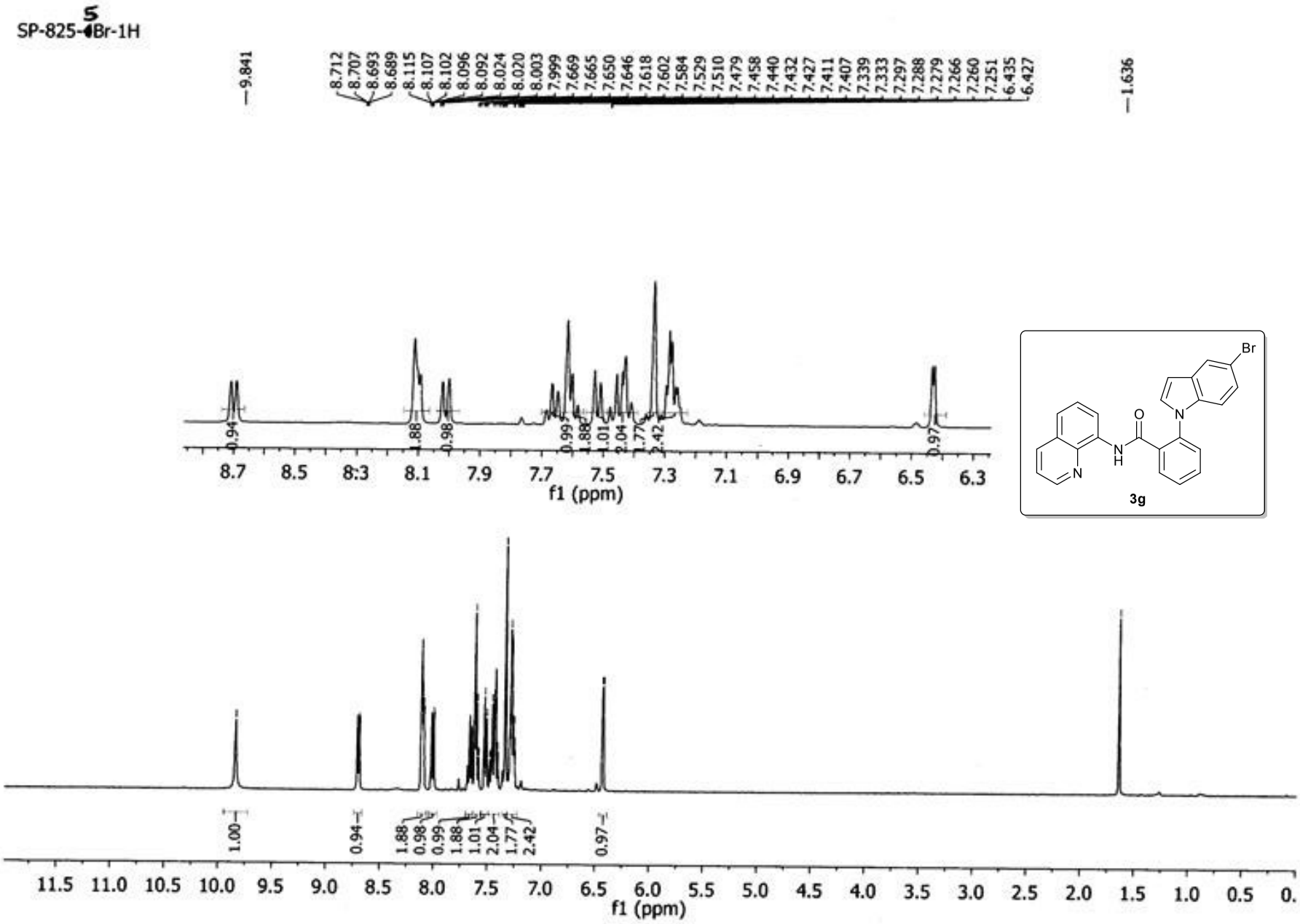

SP-840-T-1H
SP-840-T-13C
8.713
8.701
8.149
8.147
8.136
8.134
7.992
7.990
7.985
7.983
7.956
7.954
7.942
7.940
7.799
7.794
7.791
7.785
7.688
7.683
7.679
7.674
7.665
7.662
7.652
7.650
7.640
7.627
7.586
7.585
7.573
7.573
7.561
7.560
7.531
7.518
7.472
7.459
7.450
7.437
7.424
7.366
7.353
7.275
7.262
7.250
7.226
7.219
7.213
7.206
7.172
7.160
7.147
3.418
3.404
3.399
3.390
2.899
2.885
2.872
8.8 8.7 8.6 8.5 8.4 8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0
f1 (ppm)
O
O
N
N
O
N
H
N
3e
1.00
0.99
1.00
0.91
0.96
1.75
1.88
1.85
1.04
0.97
2.02
1.05
1.57
1.92
1.03
2.15
2.25
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5
f1 (ppm)

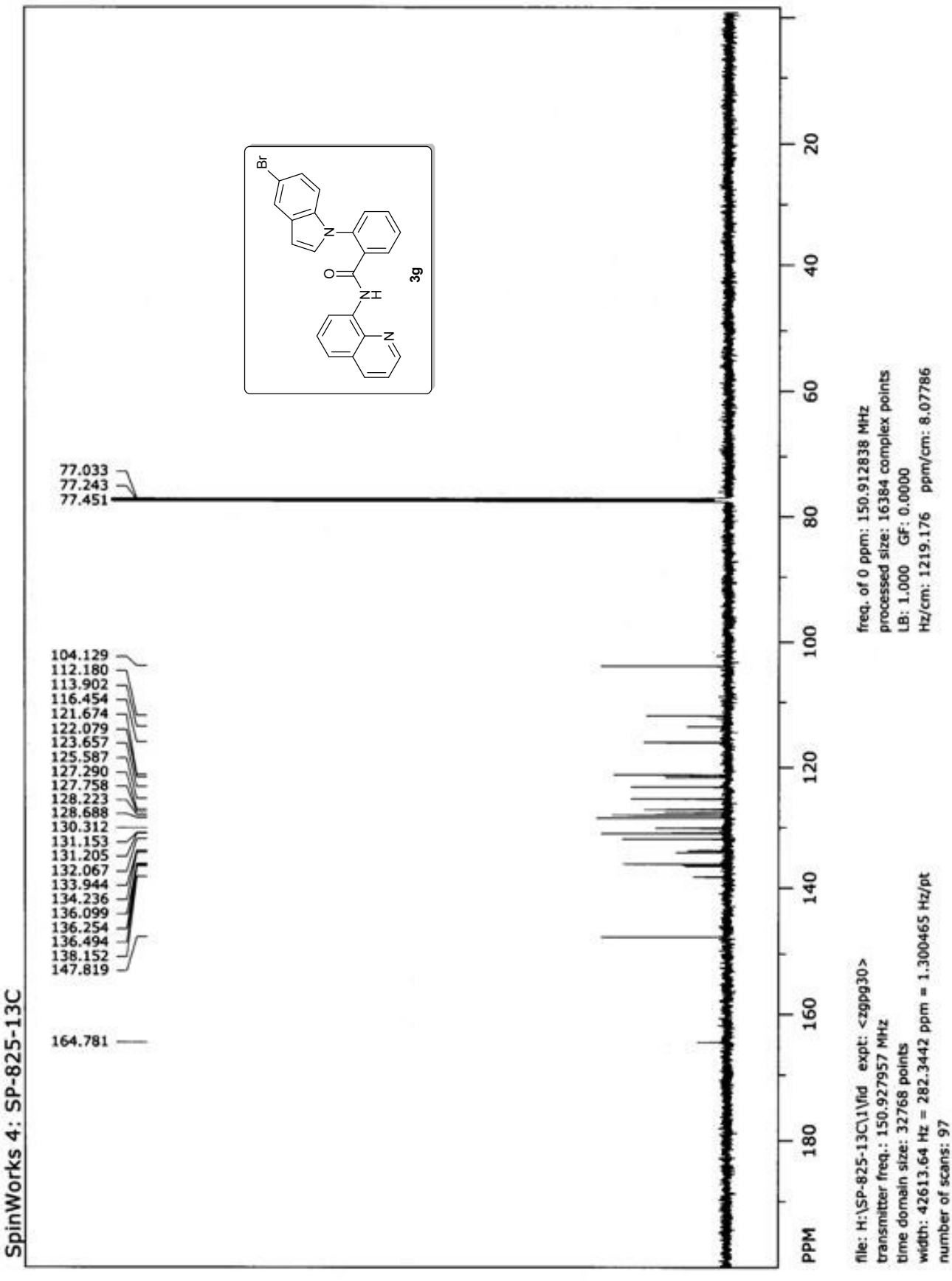

SpinWorks 4: SP-840-13C
24.470
37.929
77.019
77.227
77.438
110.755
114.746
116.388
119.330
120.554
121.503
121.830
123.140
123.346
126.844
127.326
127.631
128.135
129.119
131.416
132.055
132.308
133.359
134.060
134.390
135.927
137.018
137.879
138.116
147.785
164.980
168.330
3e
PPM
0
20
40
60
80
100
120
140
160
180
file: H:\SP-840-13C\1\fid expt: <zgpg30>
transmitter freq.: 150.927957 MHz
time domain size: 32768 points
width: 42613.64 Hz = 282.3442 ppm = 1.300465 Hz/pt
number of scans: 302
freq. of 0 ppm: 150.912838 MHz
processed size: 16384 complex points
LB: 1.000 GF: 0.0000
Hz/cm: 1220.326 ppm/cm: 8.08548

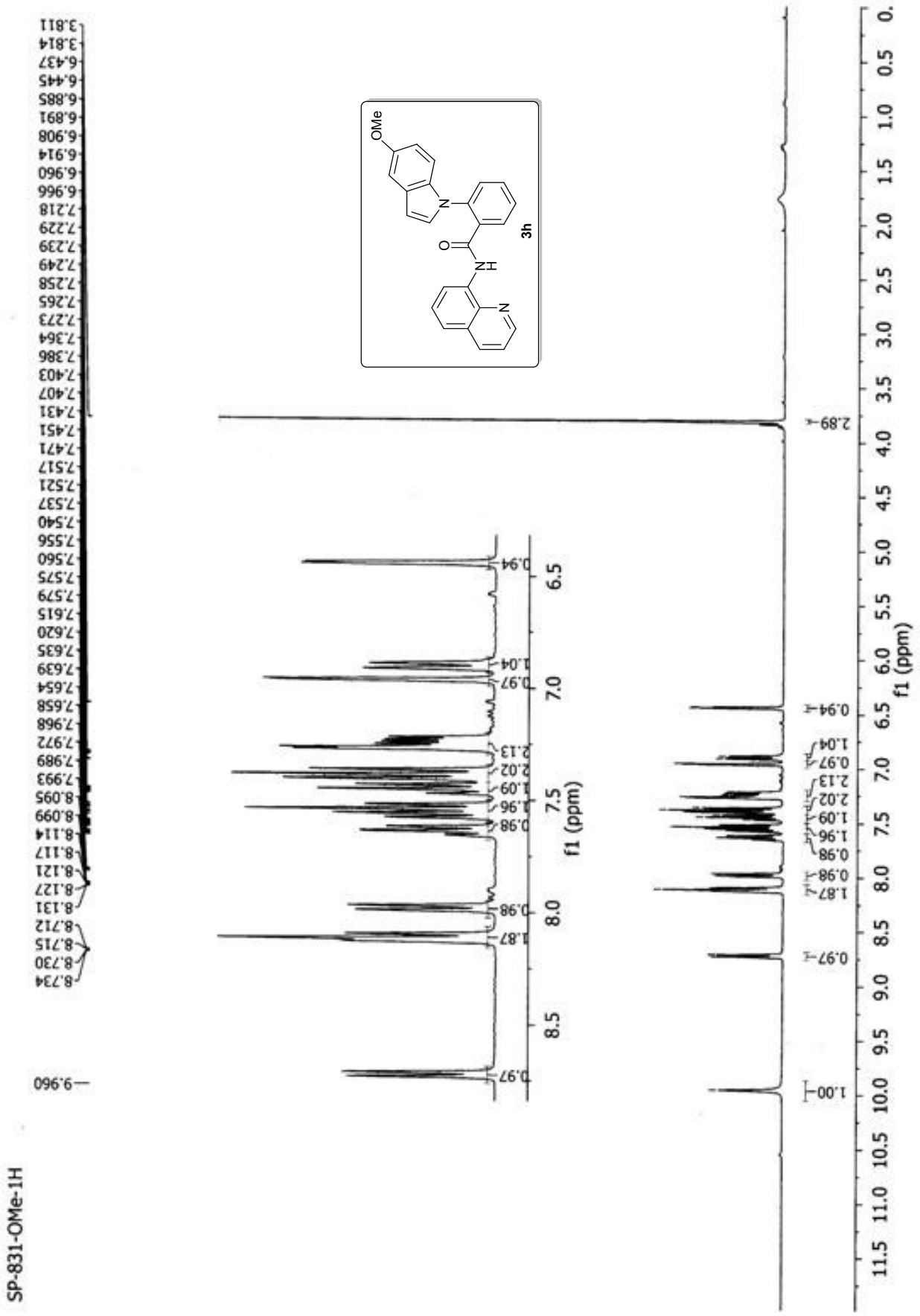
SP-834-4Br-1H

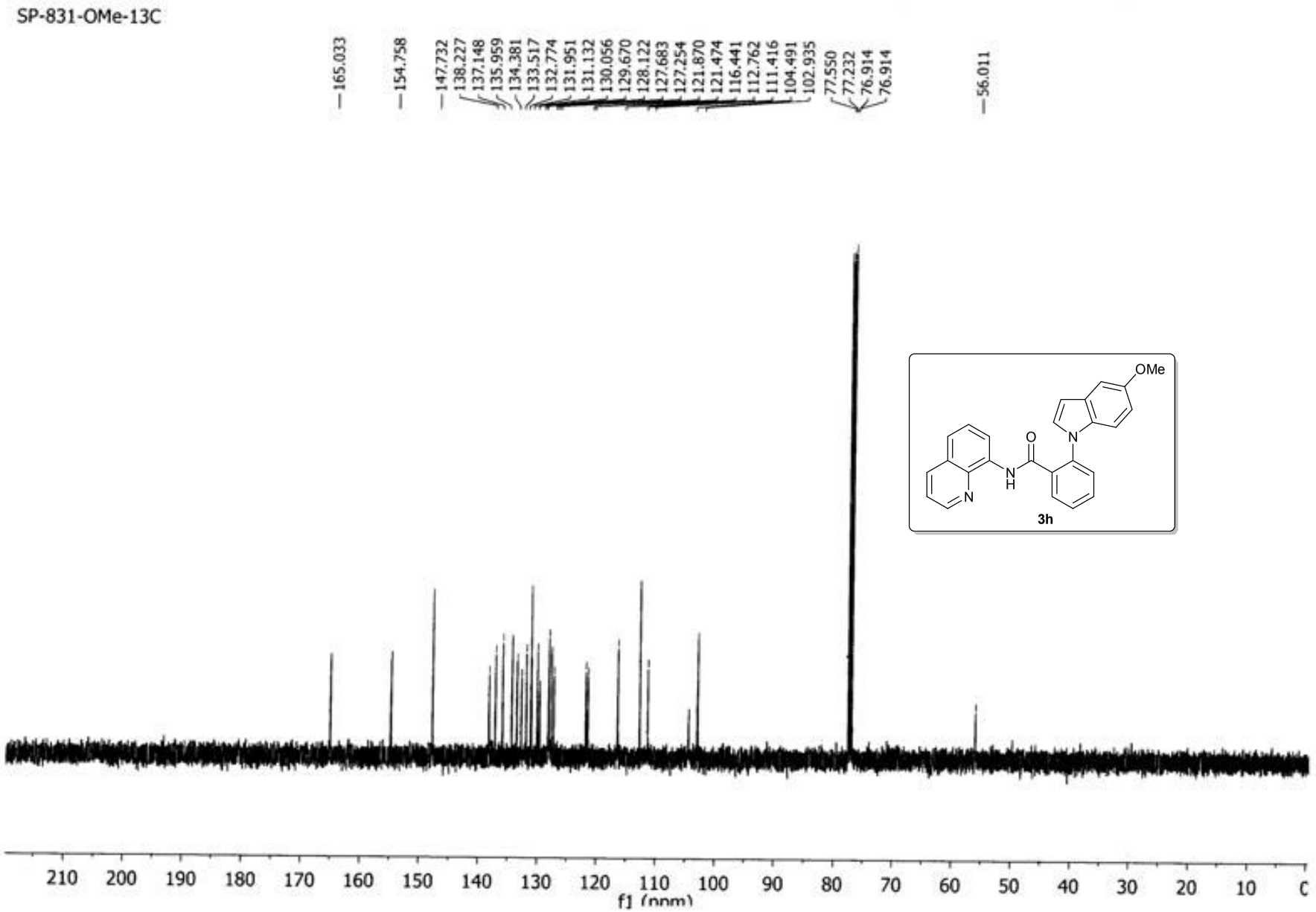

9.828
8.630
8.612
8.080
8.061
8.003
7.992
7.937
7.916
7.601
7.582
7.564
7.544
7.526
7.507
7.430
7.411
7.394
7.374
7.355
7.347
7.329
7.317
7.296
7.259
7.251
7.216
7.207
7.196
7.186
7.173
7.017
6.997
6.977
6.496
6.488
0.97
1.00
1.00
1.01
2.08
4.08
1.06
2.46
1.09
0.99
f1 (ppm)
Br
N
O
N
H
3f


SP-834-4Br-13C
164.569
147.879
138.211
137.831
136.592
136.072
134.243
133.542
132.154
131.399
130.259
129.727
128.731
128.379
127.750
127.251
123.739
123.577
122.115
121.672
116.573
115.098
109.922
105.087
77.551
77.234
76.918
Br
N
O
H
N
3f
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210
f1 (ppm)

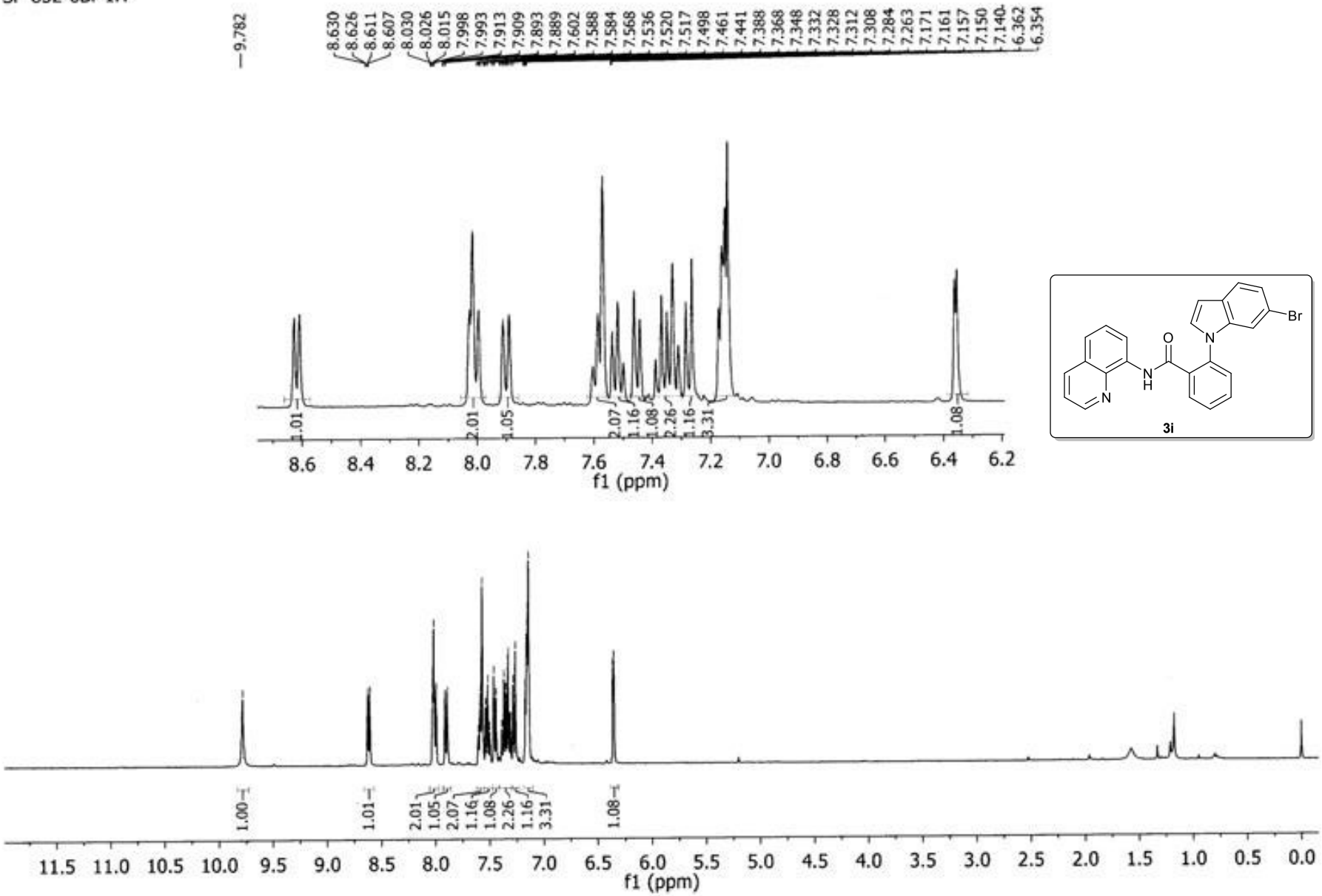

SP-825-5Br-1H
9.841
8.712
8.707
8.693
8.689
8.115
8.107
8.102
8.096
8.092
8.024
8.020
8.003
7.999
7.669
7.665
7.650
7.646
7.618
7.602
7.584
7.529
7.510
7.479
7.458
7.440
7.432
7.427
7.411
7.407
7.339
7.333
7.297
7.288
7.279
7.266
7.260
7.251
6.435
6.427
1.636
0.94
1.88
0.98
0.99
1.88
1.01
2.04
1.77
2.42
0.97
f1 (ppm)
Br
O
N
H
N
3g
1.00
0.94
1.88
0.98
0.99
1.88
1.01
2.04
1.77
2.42
0.97
f1 (ppm)

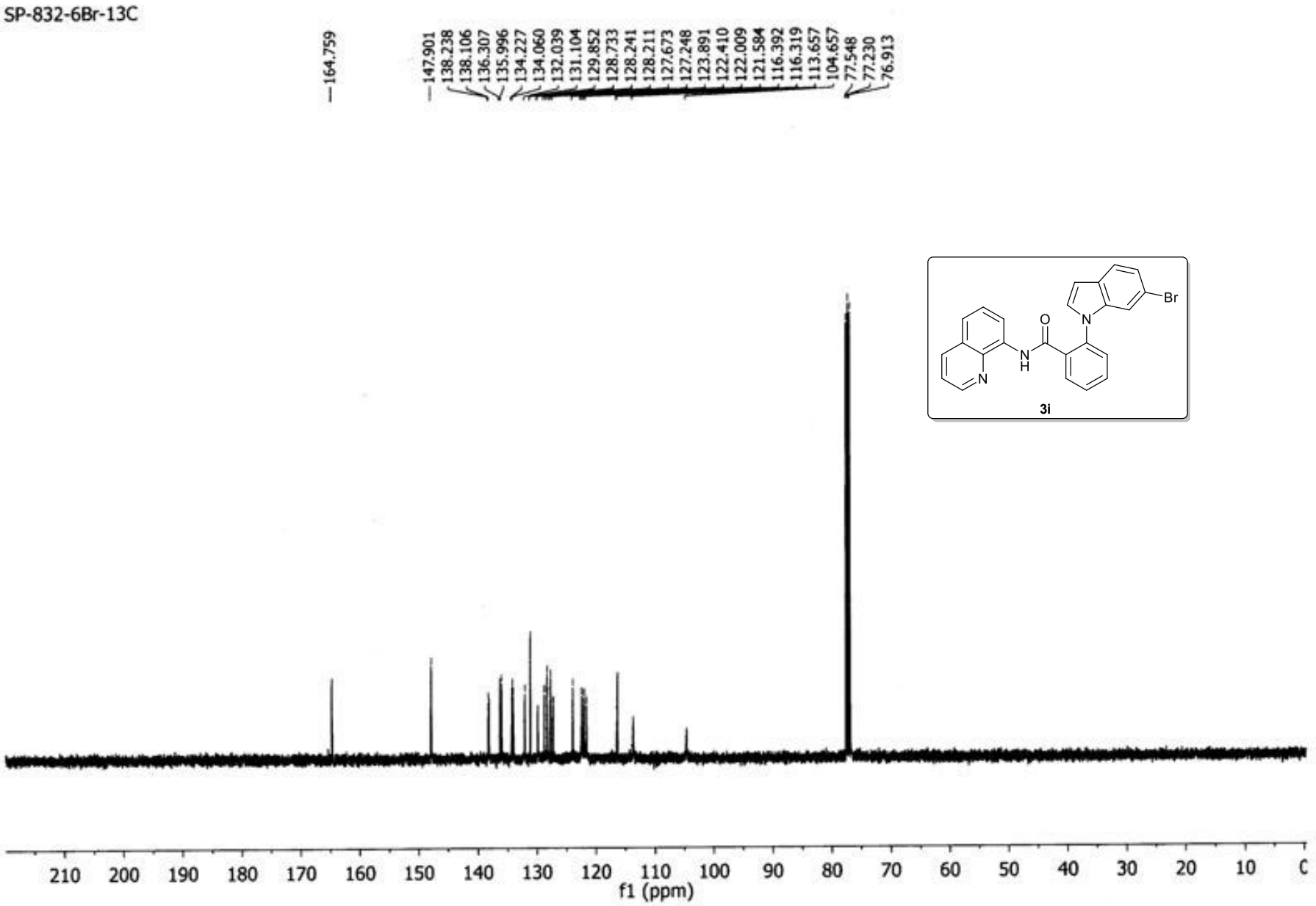

SpinWorks 4: SP-825-13C
77.033
77.243
77.451
104.129
112.180
113.902
116.454
121.674
122.079
123.657
125.587
127.290
127.758
128.223
128.688
130.312
131.153
131.205
132.067
133.944
134.236
136.099
136.254
136.494
138.152
147.819
164.781
Br
N
O
N
H
N
3g
PPM
180
160
140
120
100
80
60
40
20
file: H:\SP-825-13C\1\fid expt: <zgpg30>
transmitter freq.: 150.927957 MHz
time domain size: 32768 points
width: 42613.64 Hz = 282.3442 ppm = 1.300465 Hz/pt
number of scans: 97
freq. of 0 ppm: 150.912838 MHz
processed size: 16384 complex points
LB: 1.000 GF: 0.0000
Hz/cm: 1219.176 ppm/cm: 8.07786


SP-831-OMe-1H
OMe
3h
f1 (ppm)

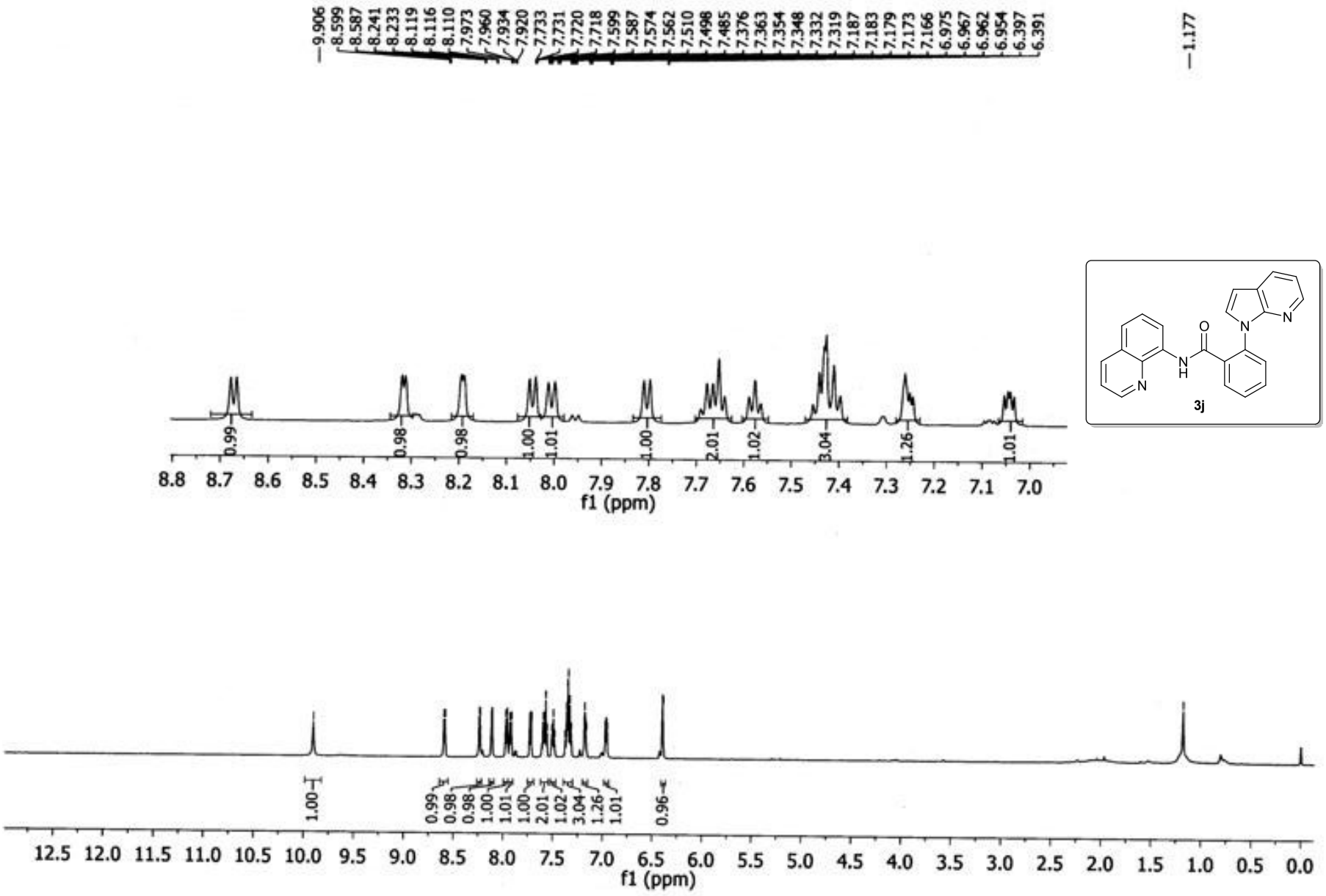

SP-831-OMe-13C
165.033
154.758
147.732
138.227
137.148
135.959
134.381
133.517
132.774
131.951
131.132
130.056
129.670
128.122
127.683
127.254
121.870
121.474
116.441
112.762
111.416
104.491
102.935
77.550
77.232
76.914
76.914
56.011
OMe
O
N
H
N
3h
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

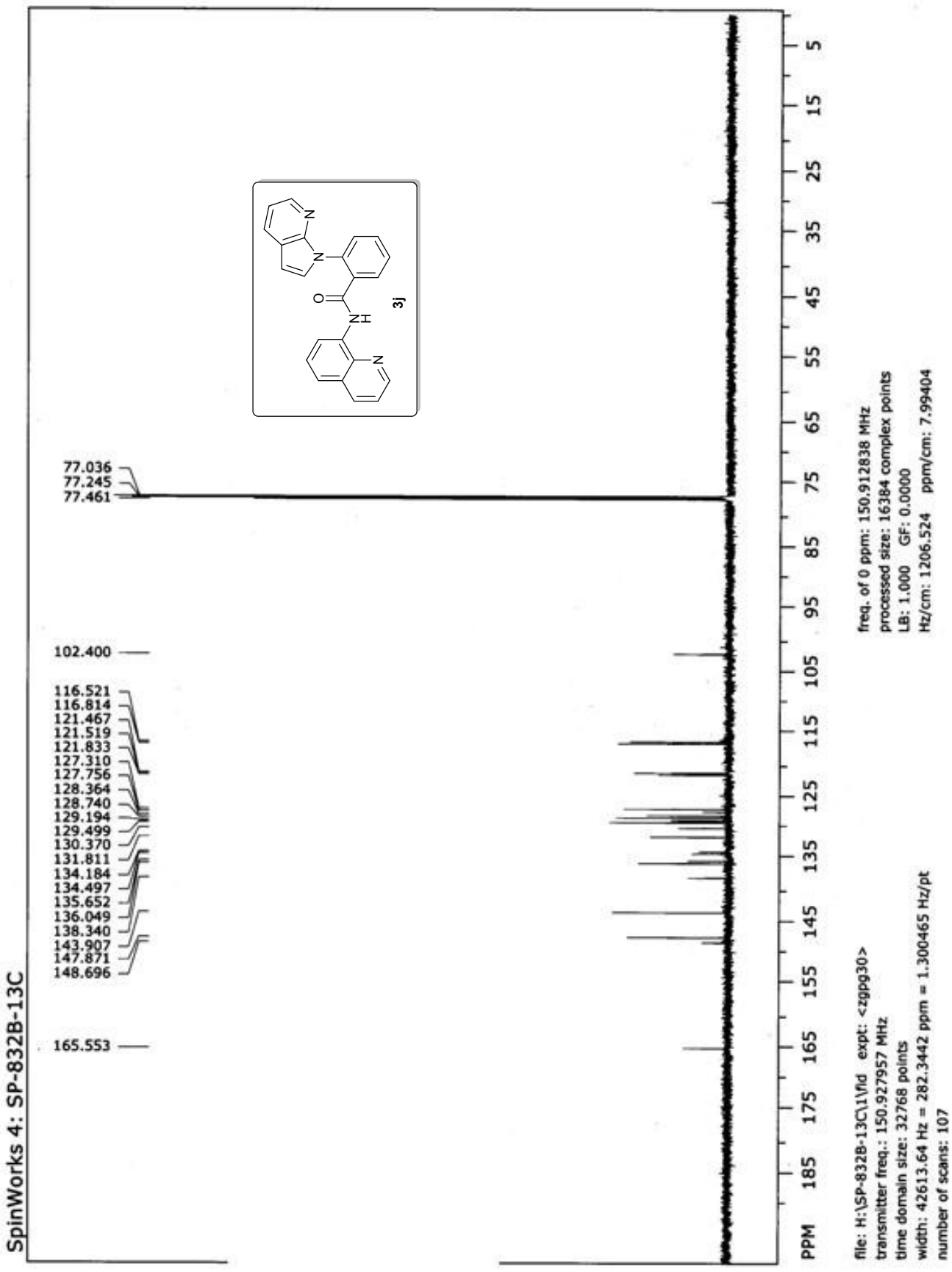
SP-832-6Br-1H


9.782
8.630
8.626
8.611
8.607
8.030
8.026
8.015
7.998
7.993
7.913
7.909
7.893
7.889
7.602
7.588
7.584
7.568
7.536
7.520
7.517
7.498
7.461
7.441
7.388
7.368
7.348
7.332
7.328
7.312
7.308
7.284
7.263
7.171
7.161
7.157
7.150
7.140
6.362
6.354
1.01
2.01
1.05
2.07
1.16
1.08
2.26
1.16
3.31
1.08
f1 (ppm)
Br
N
O
N
H
3i
1.00
1.01
2.01
1.05
2.07
1.16
1.08
2.26
1.16
3.31
1.08
f1 (ppm)

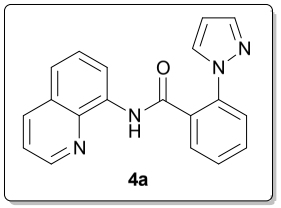

SP-832-6Br-13C
164.759
147.901
138.238
138.106
136.307
135.996
134.227
134.060
132.039
131.104
129.852
128.733
128.241
128.211
127.673
127.248
123.891
122.410
122.009
121.584
116.392
116.319
113.657
104.657
77.548
77.230
76.913
Br
N
O
N
H
3i
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

SP-832-aza

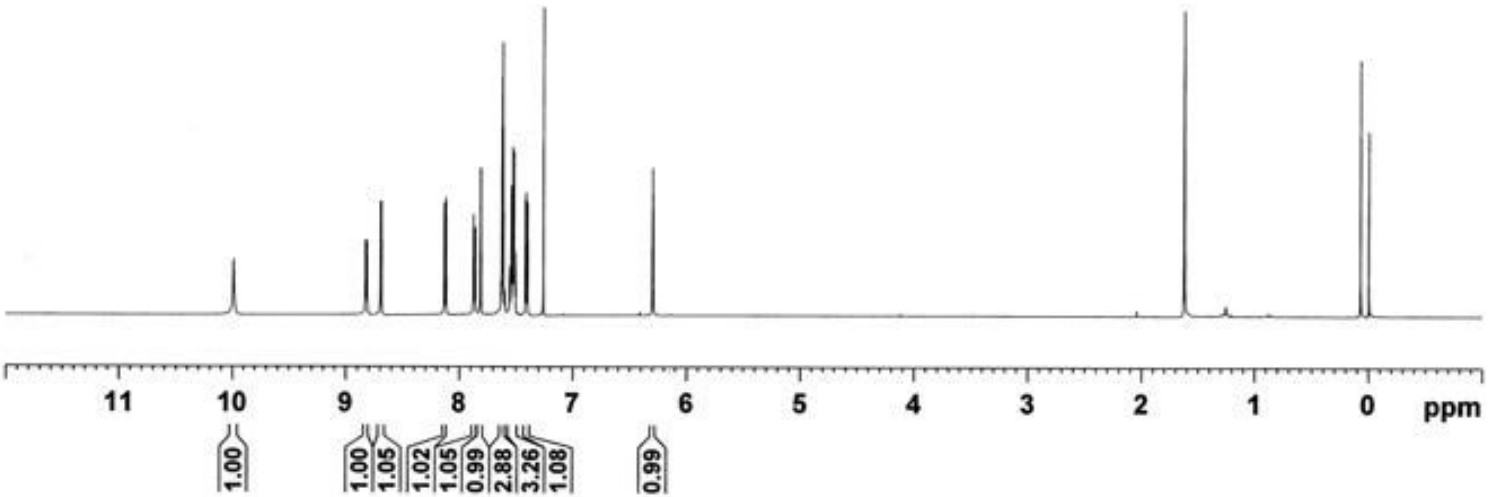

9.906
8.599
8.587
8.241
8.233
8.119
8.116
8.110
7.973
7.960
7.934
7.920
7.733
7.731
7.720
7.718
7.599
7.587
7.574
7.562
7.510
7.498
7.485
7.376
7.363
7.354
7.348
7.332
7.319
7.187
7.183
7.179
7.173
7.166
6.975
6.967
6.962
6.954
6.397
6.391
1.177
0.99
0.98
0.98
1.00
1.01
1.00
2.01
1.02
3.04
1.26
1.01
f1 (ppm)
3j
1.00
0.99
0.98
0.98
1.00
1.01
1.00
2.01
1.02
3.04
1.26
1.01
0.96
f1 (ppm)


SpinWorks 4: SP-832B-13C
77.036
77.245
77.461
102.400
116.521
116.814
121.467
121.519
121.833
127.310
127.756
128.364
128.740
129.194
129.499
130.370
131.811
134.184
134.497
135.652
136.049
138.340
143.907
147.871
148.696
165.553
3j
PPM
file: H:\SP-832B-13C\1\fid expt: <zgpg30>
transmitter freq.: 150.927957 MHz
time domain size: 32768 points
width: 42613.64 Hz = 282.3442 ppm = 1.300465 Hz/pt
number of scans: 107
freq. of 0 ppm: 150.912838 MHz
processed size: 16384 complex points
LB: 1.000 GF: 0.0000
Hz/cm: 1206.524 ppm/cm: 7.99404

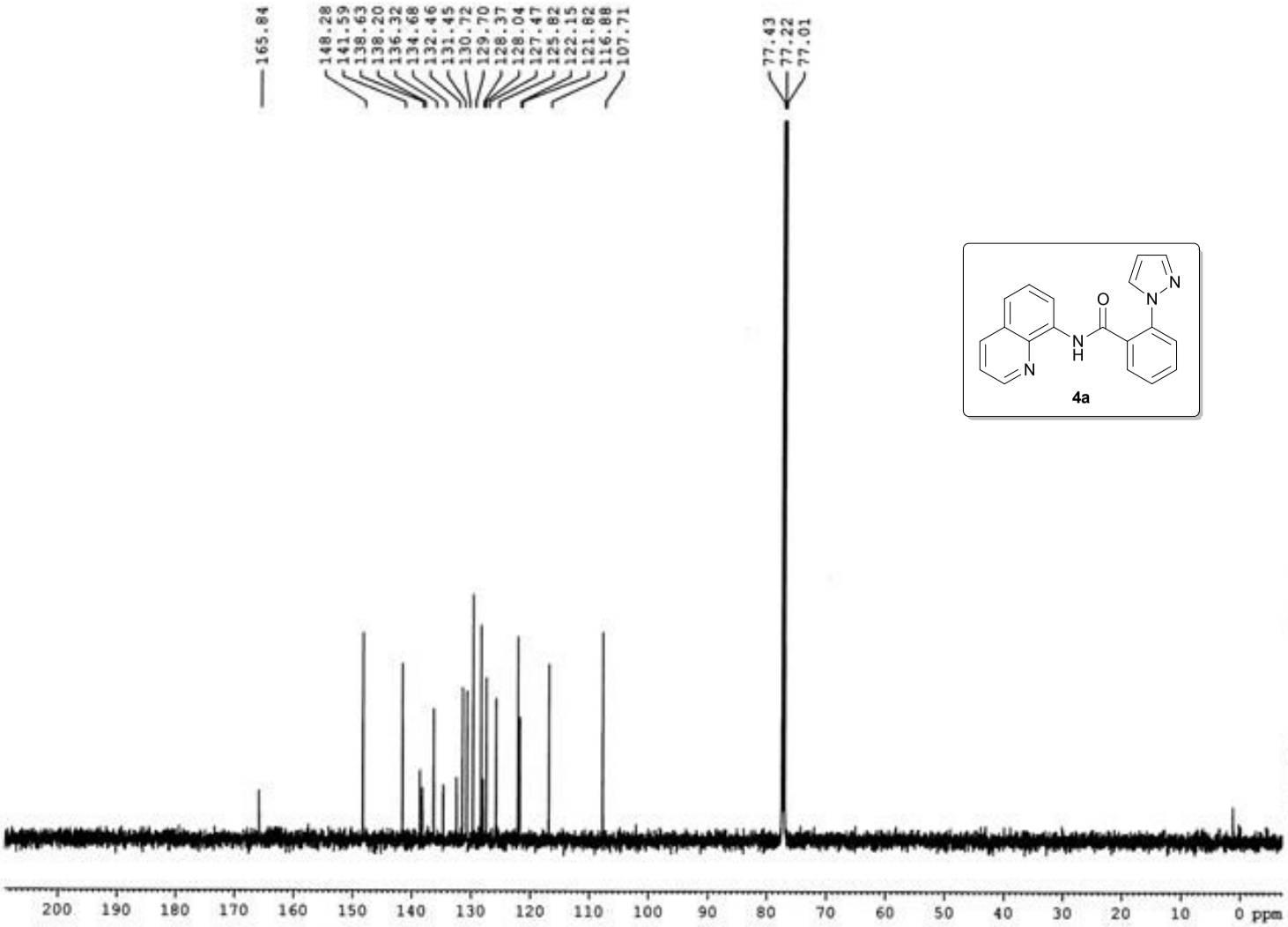
SP-689-PR-1H




```
Current Data Parameters
NAME          SP-689-PR-1H
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_          20150323
Time              14.33
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               32768
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           12019.230 Hz
FIDRES        0.366798 Hz
AQ            1.3631488 sec
RG                99.36
DW               41.600 usec
DE                6.50 usec
TE               297.3 K
D1            1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1          600.1737063 MHz
NUC1                 1H
P1                12.00 usec
PLW1          21.00000000 W

F2 - Processing parameters
SI                  16384
SF            600.1700152 MHz
WDW                    EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



SP-689-Pr_13C



4a

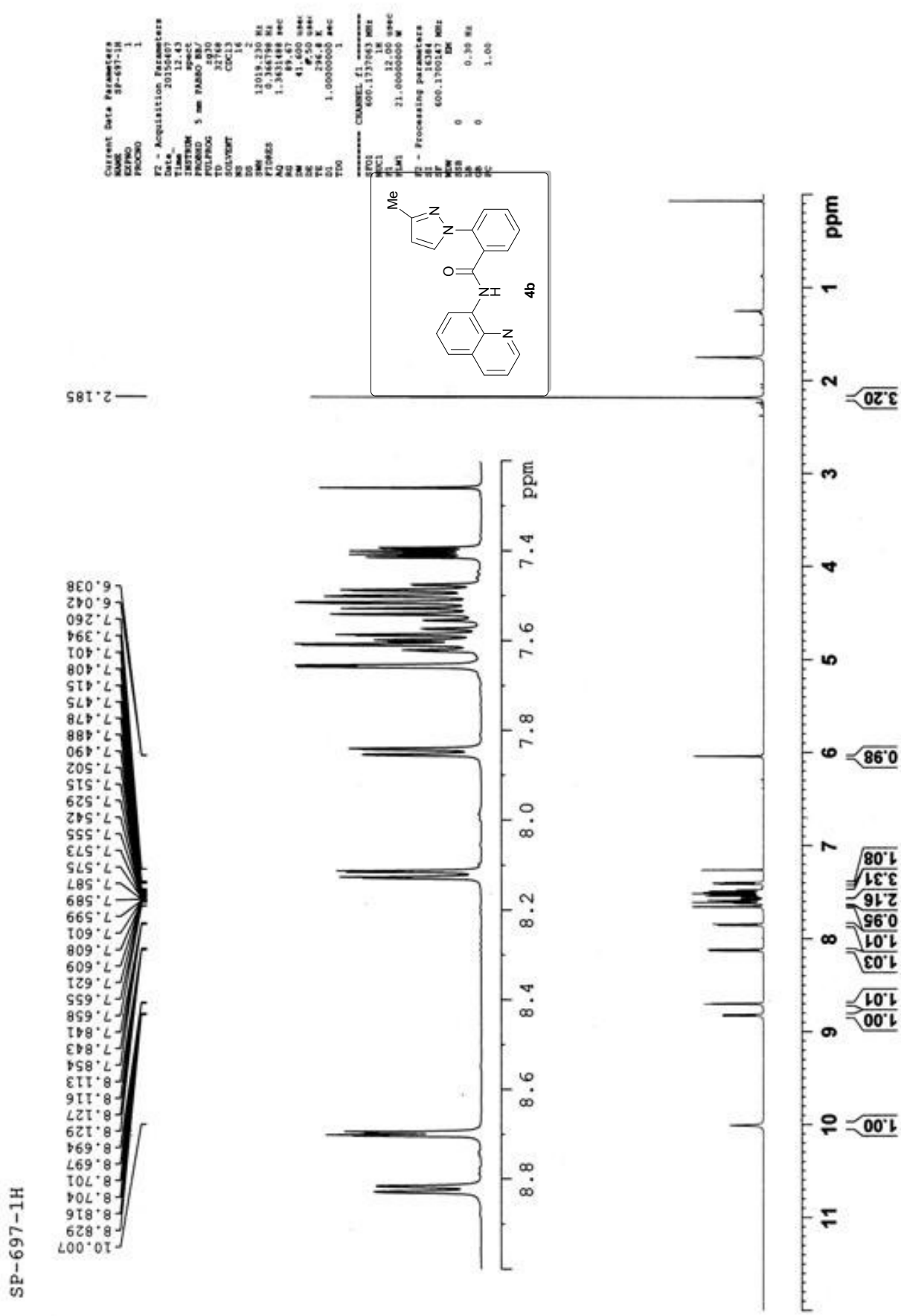
Current Data Parameters
NAME SP-689-Pr_13C
EXPNO 1
PROCNO 1

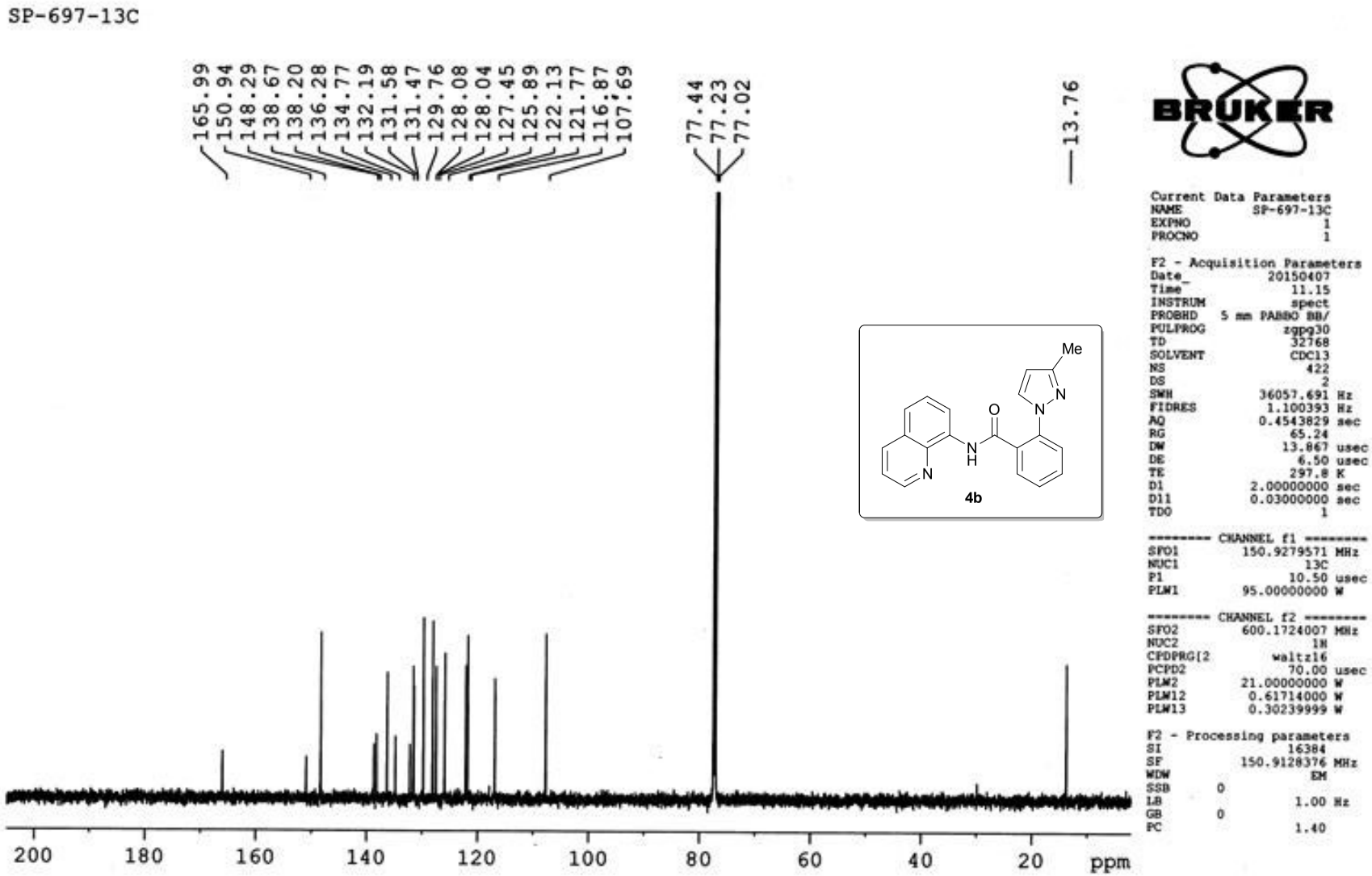
F2 - Acquisition Parameters
Date_ 20150401
Time 12.22
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 480
DS 2
SWH 36057.691 Hz
FIDRES 1.100393 Hz
AQ 0.4543829 sec
RG 65.24
DW 13.867 usec
DE 6.50 usec
TE 297.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

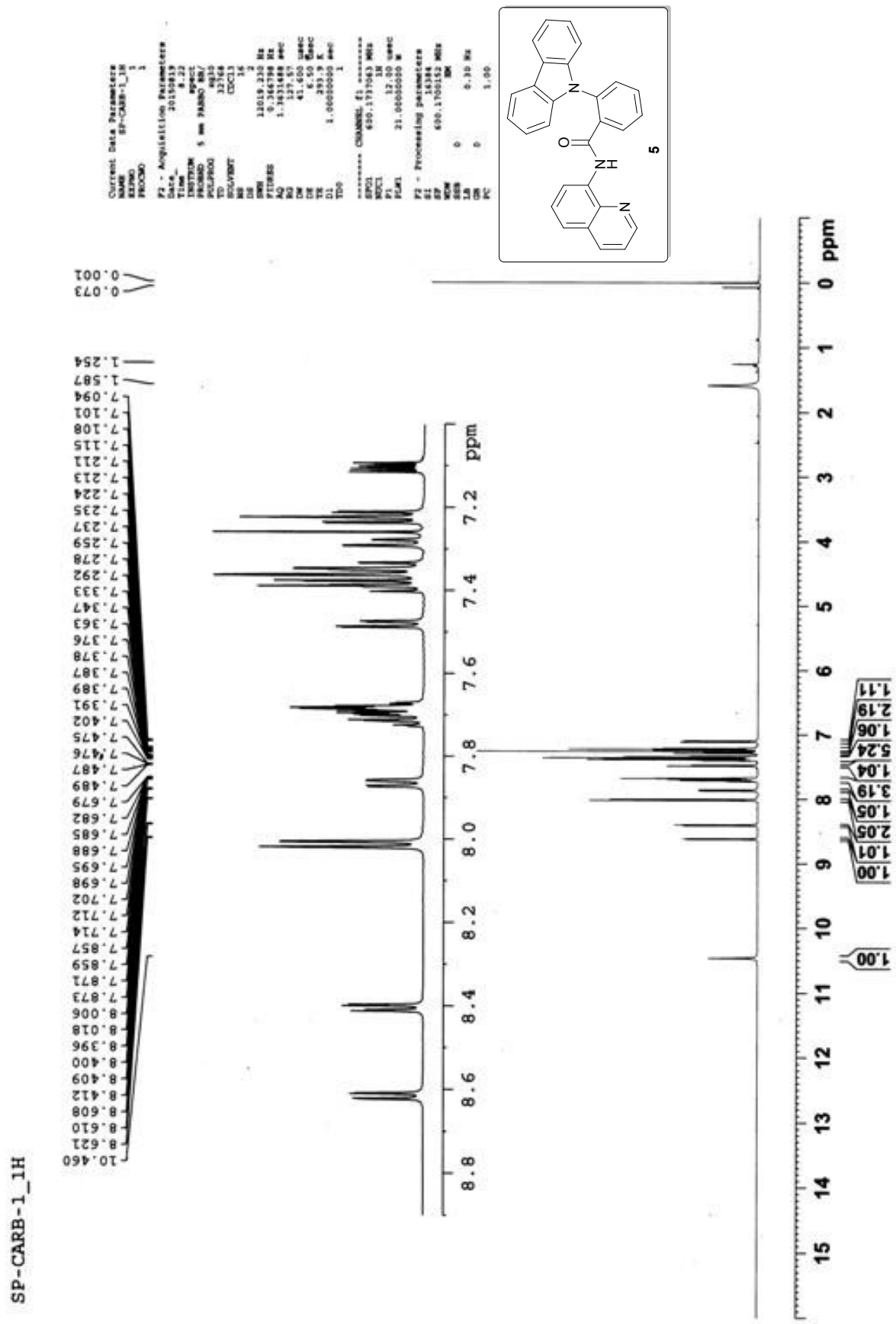
======== CHANNEL f1 ========
SFO1 150.9279571 MHz
NUC1 13C
P1 10.50 usec
PLW1 95.00000000 W

======== CHANNEL f2 ========
SFO2 600.1724007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 21.00000000 W
PLW12 0.61714000 W
PLW13 0.30239999 W

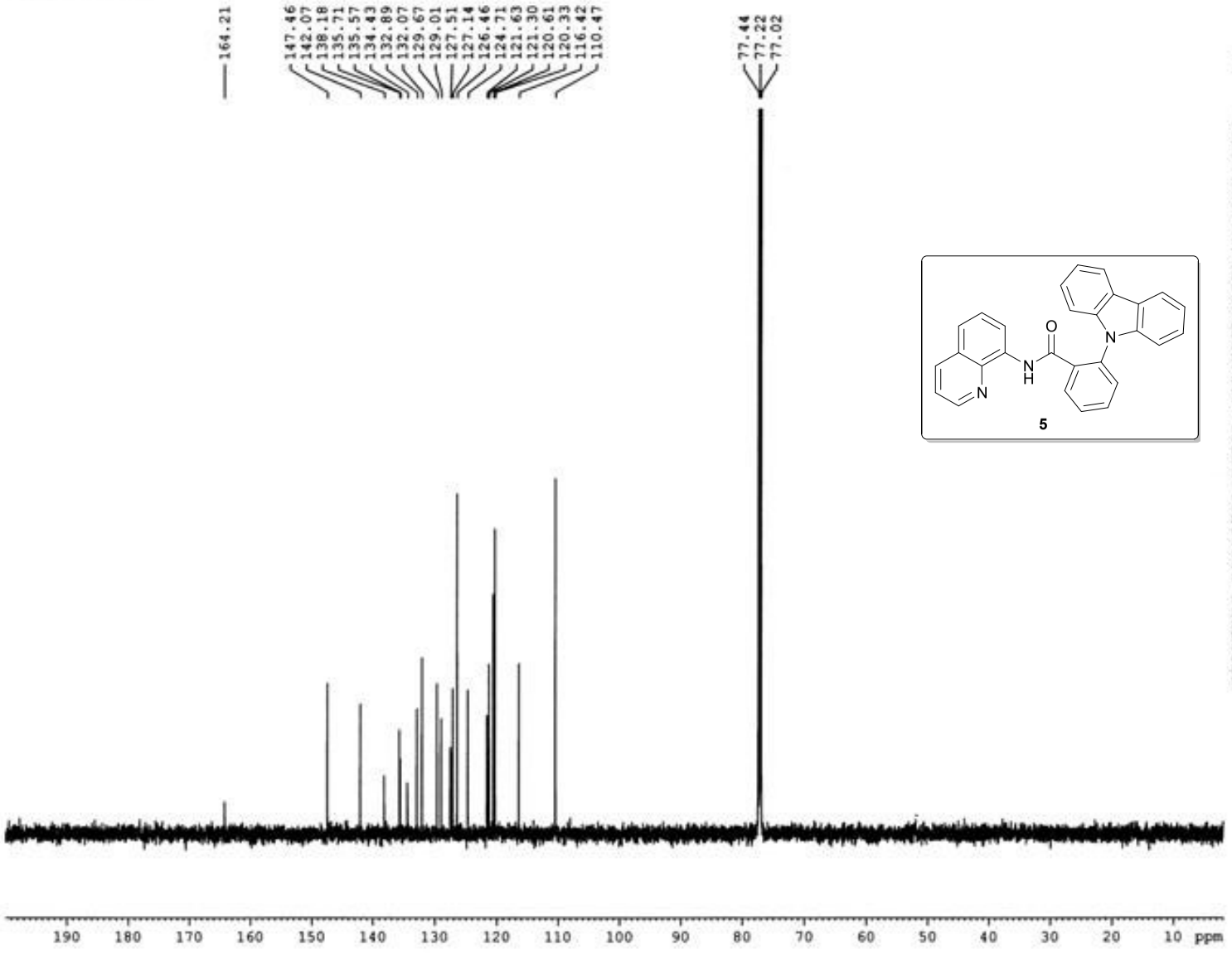
F2 - Processing parameters
SI 16384
SF 150.9128368 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


SP-697-1H
4b
Me
ppm


SP-697-13C
165.99
150.94
148.29
138.67
138.20
136.28
134.77
132.19
131.58
131.47
129.76
128.08
128.04
127.45
125.89
122.13
121.77
116.87
107.69
77.44
77.23
77.02
13.76
Me
N
N
O
N
H
4b
200
180
160
140
120
100
80
60
40
20
ppm
BRUKER
Current Data Parameters
NAME SP-697-13C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150407
Time 11.15
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 422
DS 2
SWH 36057.691 Hz
FIDRES 1.100393 Hz
AQ 0.4543829 sec
RG 65.24
DW 13.867 usec
DE 6.50 usec
TE 297.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
SFO1 150.9279571 MHz
NUC1 13C
P1 10.50 usec
PLW1 95.00000000 W
CHANNEL f2
SFO2 600.1724007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 21.00000000 W
PLW12 0.61714000 W
PLW13 0.30239999 W
F2 - Processing parameters
SI 16384
SF 150.9128376 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


SP-CARB-1_1H
Current Data Parameters
NAME SP-CARB-1_1H
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150819
Time 8.22
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 137.57
DW 41.600 usec
DE 6.50 usec
TE 293.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 600.1717043 MHz
NUC1 1H
P1 12.00 usec
PLW1 21.00000000 W
F2 - Processing parameters
SI 16384
SF 600.1700152 MHz
WDW EM
SSB 0 0.30 Hz
LB
GB 0
PC 1.00
5
ppm
SP-CARB-1_1H

SP-CARB_13C



BRUKER

Current Data Parameters
NAME SP-CARB_13C
EXPNO 1
PROCNO 1

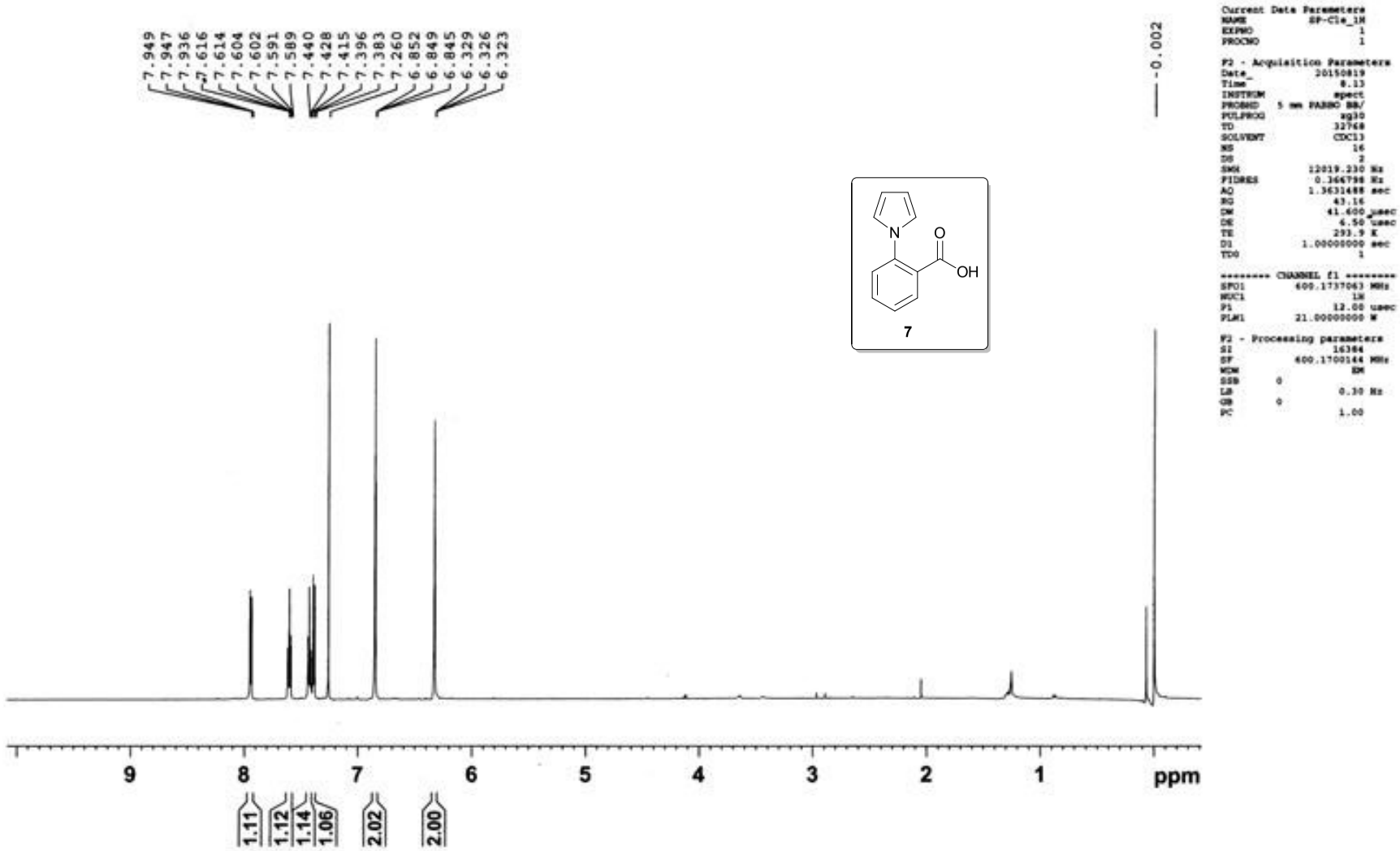
F2 - Acquisition Parameters
Date_ 20150807
Time 11.58
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 475
DS 2
SWH 36057.691 Hz
FIDRES 1.100393 Hz
AQ 0.4543829 sec
RG 200.18
DW 13.867 usec
DE 6.50 usec
TE 298.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9279571 MHz
NUC1 13C
P1 10.50 usec
PLW1 95.00000000 W

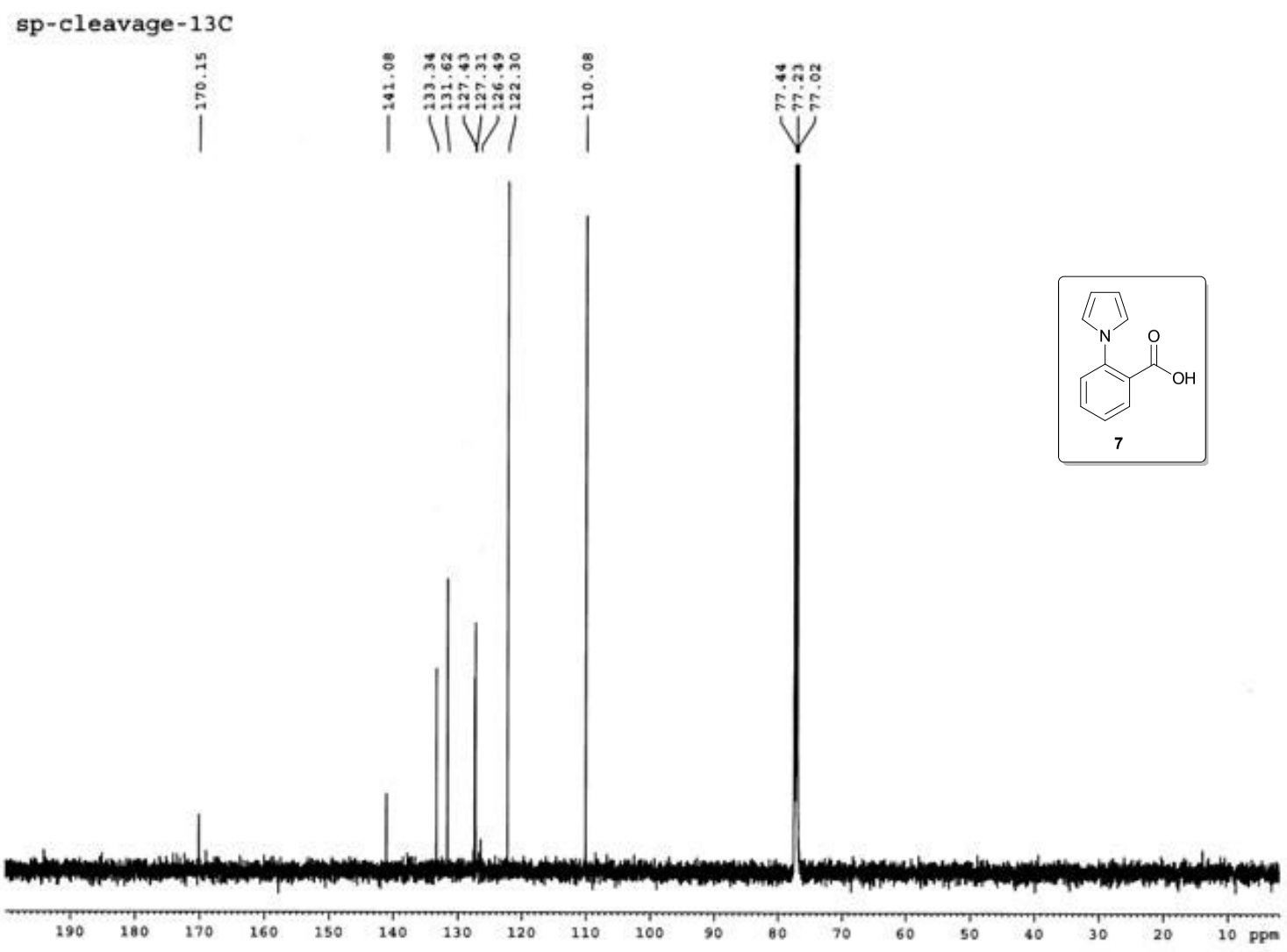
======== CHANNEL f2 ========
SFO2 600.1724007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 21.00000000 W
PLW12 0.61714000 W
PLW13 0.30239999 W

F2 - Processing parameters
SI 16384
SF 150.9128356 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

SP-Cle_1H




```
Current Data Parameters
NAME                  SP-Cle_1H
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_               20150819
Time                      8.13
INSTRUM                   spect
PROBHD     5 mm PABBO BB/
PULPROG                   zg30
TD                       32768
SOLVENT                  CDCl3
NS                          16
DS                           2
SWH                12019.230 Hz
FIDRES              0.366796 Hz
AQ                  1.3631488 sec
RG                      43.16
DW                    41.600 usec
DE                     6.50 usec
TE                     293.9 K
D1                 1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1               600.1737063 MHz
NUC1                       1H
P1                      12.00 usec
PLW1               21.00000000 W

F2 - Processing parameters
SI                       16384
SF                 600.1700144 MHz
WDW                         EM
SSB          0
LB                      0.30 Hz
GB           0
PC                      1.00
```



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BRUKER

Current Data Parameters
NAME sp-cleavage-13C
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150820
Time 19.48
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 1926
DS 2
SWH 42613.637 Hz
FIDRES 1.300465 Hz
AQ 0.3844779 sec
RG 200.18
DW 11.733 usec
DE 6.50 usec
TE 295.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9279571 MHz
NUC1 13C
P1 10.50 usec
PLW1 95.00000000 W

======== CHANNEL f2 ========
SFO2 600.1724007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 21.00000000 W
PLW12 0.61714000 W
PLW13 0.30239999 W

F2 - Processing parameters
SI 16384
SF 150.9128338 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40