Palladium-Catalyzed Domino C,N-Coupling/Carbonylation/Suzuki Coupling Reaction: An efficient synthesis of 2-Aroyl/Heteroaroyl-Indoles

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Supporting information

General Remarks:

CH_2Cl_2 was dried over P_2O_5, toluene over CaH_2 and dioxane over Na/benzophenone. 2-*gem*dibromovinylanilines **1a-1g** were prepared in a two-step sequence (Ramirez olefination, reduction using $SnCl_2.2H_2O$) from *o*-nitrobenzaldehyde derivatives according to Fang and Lautens.[1] All others commercial reagents and solvents were used as received without additional purification. NMR spectra were recorded with a BRUCKER ACP 300 spectrometer at 300 MHz for ^1H NMR and 75 MHz for ^{13}C NMR spectroscopy. Chemical shifts are given as δ values in ppm relative to the residual solvent peak ($CHCl_3$) as the internal reference, coupling constants are given in Hertz. All ^{13}C NMR spectra were recorded with complete proton decoupling. Peak assignment was unambiguously performed using HMQC, HMBC and NOESY technics. IR spectra were measured with FT-IR Perkin-Elmer spectrometer. Melting points were determined by the capillary method using an Electrothermal 9200 apparatus and are uncorrected. Mass spectra were recorded with a WATERS ZQ 2000 (ESI). High-resolution mass spectra were obtained on a Jeol 700 (DCI) or on a Waters LCT (ESI). Reactions were followed with Merck TLC silica gel 60 F_{254}. Flash chromatographies were carried out on Merck silica gel (320-400 mesh).

CAUTION: CO is a highly toxic odorless and colorless gas. Reactions involving Carbone Monoxide must be performed in a well ventilated hood with a Carbon Monoxide detector nearby.

[1] Fang, Y. –Q. ; Lautens, M. *J. Org. Chem*. **2008**, *73*, 538.

Synthetic Procedures

I) Synthesis of anilines

All anilines were prepared from *o*-nitrobenzaldehyde derivatives by a two-step reaction, following the procedure describe by Fang and Lautens.[1] Anilines **1c-1f** showed identical spectroscopic properties to those previously reported.[1]

2-(2,2-dibromovinyl)-3-chlorobenzenamine (1b)

Aniline **1b** was obtained from 2-chloro-6-nitrobenzaldehyde as an orange oil (82 % over the 2 steps): IR (CH_2Cl_2, cm^{-1}) 3426, 3395, 1067, 1047, 1009, 2925, 1616, 1569, 1471, 1448, 1305, 1292; ^1H NMR (300 MHz, $CDCl_3$) δ 7.27 (s, 1H), 7.07 (td, J = 8.0, 0.5 Hz, 1H), 6.79 (dd, J = 7.9, 0.9 Hz, 1H), 6.60 (dd, J = 8.1, 0.9 Hz, 1H), 3.87 (brs, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 144.8, 133.6, 133.0, 130.0, 120.5, 118.9, 113.7, 96.3; MS (ES^+) *m/z* 310 (45 %), 312 (100 %), 314 (70 %), 315 (15%) $[M+H]^+$; HRMS calcd for $C_8H_7N^{35}Cl^{79}Br^{81}Br$ $[M+H]^+$ 311.8613, found 311.8628.

2-(2,2-dibromovinyl)-3,4,5-trimethoxyaniline (1g)

Aniline **1g** was obtained from 2,3,4-trimethoxy-6-nitrobenzaldehyde[2] as a brown oil (55 % over the 2 steps): IR (CH_2Cl_2, cm^{-1}) 3620, 3483, 3399, 3012, 2974, 2939, 1615, 1498, 1458, 1411, 1233, 1217, 1125 ^1H NMR (300 MHz, $CDCl_3$) δ 7.23 (s, 1H), 6.04 (s, 1H), 3.85 (s, 3H), 3.81 (s, 3H), 3.77 (s, 3H), 3.65 (brs, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 154.4, 151.4, 139.8, 134.2, 132.4, 108.8, 95.1, 94.3, 61.2, 61.1, 55.7; MS (ES^+) *m/z* 388 (55 %), 389 (100 %), 392 (45 %) $[M+Na]^+$; HRMS calcd for $C_{11}H_{14}NO_3{}^{79}Br^{81}Br$ $[M+H]^+$ 367.9320, found 367.9319.

[2] Cherkaoui, M. Z.; Scherowsky, G. *New. J. Chem.* **1997**, *21*, 1203.

General procedure for the domino C,N-coupling/carbonylation/Suzuki coupling reaction.

The autoclave and the magnetic stirring bar were dried in an oven and then cold to room temperature under an argon atmosphere. Boronic acid (1.1 mmol), K_2CO_3 (5 mmol, flamed dried prior to use) and $Pd(PPh_3)_4$ (0.05 mmol) were introduced then the autoclave was flushed with argon for 5 minutes. A degassed solution (argon bulbbing for 10 min) of aniline (1 mmol) in dry dioxane (10 mL) was added and the autoclave was flushed three times with CO and pressurized to 12 bar.

After heating for the appropriate time and temperature in an oil bath, the autoclave was cooled to room temperature and then cautionary discharged of the gas excess. Reaction mixture was diluted in ethyl acetate (10 mL) and washed with water (10 mL), saturated aqueous NH_4Cl (10 mL) and brine (10 mL). The aqueous layers were combined, saturated with NaCl, acidified (by adding HCl 1M until pH = 2) and extracted with ethyl acetate (2 x 20 mL). Organic layers were combined, dried over $MgSO_4$ and concentrated under reduce pressure. The crude product was purified by flash chromatography and recrystallized in the indicated solvents to give aroylindoles **2a-2t**.

(1*H*-indol-2-yl)(phenyl)methanone (2a)[3]

Following general procedure, a mixture of aniline **1a** (277 mg, 1 mmol) and phenyl boronic acid (134 mg, 1.1 mmol) was heated at 85 °C for 60 h. The residue was purified by flash chromatography (toluene 100 %) and recrystallization (dichloromethane/hexane) to afford **2a** as beige crystals (154 mg, 70%): mp 149-150 °C (lit. 147-148 °C); IR ($CHCl_3$, cm^{-1}) 3444, 3064, 1630, 1524, 1340, 1125; ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.32 (brs, 1H), 7.99 (d, J = 8.4 Hz, 2H), 7,72 (dd, J = 8.1, 0.8 Hz, 1H), 7,63 (tt, J = 7.3, 1.4 Hz, 1H), 7.53 (tt, J = 6.6, 1.6 Hz, 2H), 7.48 (dd, J = 8.4, 0.8 Hz, 1H), 7.38 (td, J = 8.0, 1.1 Hz, 1H), 7.20-7.14 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 187.3 138.0, 137.7, 134.4, 132.4, 129.3, 128.5, 127.7, 126.5, 123.2, 121.0, 112.9, 112.3; MS (ES^+) *m/z* 244 $[M+Na]^+$.

(4-chloro-1*H*-indol-2-yl)(phenyl)methanone (2b)

Following general procedure, a mixture of aniline **1b** (311 mg, 1 mmol) and phenyl boronic acid (134 mg, 1.1 mmol) was heated at 85 °C for 24 h. The residue was purified by flash chromatography (toluene/ethyl acetate 98/2) and recrystallization (dichloromethane/hexane)

[3] Abbiati, G.; Casoni, A.; Canevari, V.; Nava, D.; Rossi, E. *Org. Lett*. **2006**, *8*, 4839.

to afford **2b** as yellow crystals (174 mg, 68%): mp 191-192 °C ; IR (CH_2Cl_2, cm^{-1}) 3437, 3053, 1633, 1566, 1518, 1336, 1247, 1131; ^1H NMR (300 MHz, $CDCl_3$) δ 9.66 (brs, 1H), 8.02 (dd, *J* = 6.9, 1.4 Hz, 2H), 7.65 (td, *J* = 7.3, 1.4 Hz, 1H), 7.57 (m, 2H), 7.41 (d, *J* = 8.2 Hz, 1H), 7.29 (t, *J* = 7.5, 8.2 Hz, 1H), 7.25 (dd, *J* = 2.1, 0.8 Hz, 1H), 7.18 (dd, *J* = 7.5, 0.5 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 187.1, 138.0, 137.5, 134.5, 132.7, 129.2, 128.6, 128.4, 126.9, 120.7, 110.9, 110.7; MS (ES^+) *m/z* 256 $[M+H]^+$, 258 $[M+H]^+$; HRMS calcd for $C_{15}H_{11}N^{35}Cl$ $[M+H]^+$ 256.0529, found 256.0518.

Methyl-2-(phenylcarbonyl)-1*H*-indole-5-carboxylate (2c)

Following general procedure, a mixture of aniline **1c** (335 mg, 1 mmol) and phenyl boronic acid (134 mg, 1.1 mmol) was heated at 85 °C for 24 h. The residue was purified by flash chromatography (toluene/ethyl acetate 98/2) and recrystallization (dichloromethane/hexane) to afford **2c** as white crystals (140 mg, 50%): mp 201-202 °C; IR ($CHCl_3$,cm^{-1}) 3346, 2925, 1713, 1633, 1620, 1531, 1336, 1257; ^1H NMR (300 MHz, $CDCl_3$) δ 9.55 (brs, 1H), 8.50 (s, 1H), 8.06 (dd, *J* = 8.7, 1.5 Hz, 1H), 7.99 (d, *J* = 7.0 Hz, 2H), 7.65 (t, *J* = 7.3 Hz, 1H), 7.54 (t, *J* = 7.6 Hz, 2H), 7.50 (d, *J* = 8.7 Hz, 1H), 7.26 (s, 1H), 3.94 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 187.1, 167.5, 139.7, 137.6, 135.5, 132.7, 129.2, 128.6, 127.3 (2C), 126.5, 123.3, 113.7, 112.0, 52.1; MS (ES^+) *m/z* 280 $[M+H]^+$;HRMS calcd for $C_{17}H_{14}NO_3$ $[M+H]^+$ 280.0974, found 280.0974.

(*5H*-[1,3]dioxolo[4,5-*f*]indol-6-yl)(phenyl)methanone (2d)[4]

Following general procedure, a mixture of aniline **1d** (321 mg, 1 mmol) and phenyl boronic acid (134 mg, 1.1 mmol) was heated at 85 °C for 24 h. The residue was purified by flash chromatography (toluene/ethyl acetate 98/2) and recrystallization (dichloromethane/hexane) to afford **2d** as yellow crystals (148 mg, 56%): mp 200-201 °C (lit. 200 °C); IR (CH_2Cl_2, cm^{-1}) 3442, 3062, 2978, 2888, 1621, 1528, 1498, 1471, 1279, 1245, 1177, 1122, 1040; ^1H NMR (300 MHz, $CDCl_3$) δ 9.35 (brs, 1H), 7.95 (d, *J* = 6.9 Hz, 2H), 7.59 (t, *J* = 7.4 Hz, 1H), 7.51 (dd, *J* = 7.4, 6.9 Hz, 2H), 7.04 (dd, *J* = 2.1, 0.5 Hz, 1H), 6.99 (s, 1H), 6.87 (s, 1H), 5.99 (s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 186.1, 149.0, 144.4, 138.3, 134.3, 133.7, 132.0, 129.1, 128.4, 122.1, 113.5, 101.2, 100.0, 92.0; MS (ES^+) *m/z* 266 $[M+H]^+$; HRMS calcd for $C_{16}H_{12}NO_3$ $[M+H]^+$ 266.0817, found 266.0808.

[4] Mali, R. S.; Tilve, S. G.; Desai, V. G. *J. Chem. Res. M. P.* **2000**, 150.

(5,6-dimethoxy-1*H*-indol-2-yl)(phenyl)methanone (2e)[4]

Following general procedure, a mixture of aniline **1e** (337 mg, 1 mmol) and phenyl boronic acid (134 mg, 1.1 mmol) was heated at 85 °C for 24 h. The residue was purified by flash chromatography (toluene/ethyl acetate 85/15) and recrystallization (dichloromethane/hexane) to afford **2e** as yellow needles (155 mg, 55%): mp 181-182 °C (lit. 180 °C); IR (CH_2Cl_2 ,cm^{-1}) 3444, 3061, 3007, 2960, 2835, 1621, 1522, 1498, 1279, 1251, 1218, 1203, 1123; ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.43 (brs, 1H), 7.98 (dt, J = 7.0, 1.5 Hz, 2H), 7.60 (ttd, J = 7.3, 7.0, 1.4 Hz, 1H), 7.51 (tt, J = 7.3, 1.3 Hz, 2H), 7.06 (dd, J = 2.1, 0.7 Hz, 1H), 7.03 (s, 1H), 6.91 (s, 1H), 3.95 (s, 3H), 3.91 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 186.1, 151.2, 146.5, 138.4, 133.5, 133.4, 132.0, 129.1, 128.4, 120.9, 113.2, 102.7, 93.7, 56.1, 56.0; MS (ES^+) *m/z* 282.1 $[M+H]^+$ 304 $[M+Na]^+$; HRMS calcd for $C_{17}H_{16}NO_3$ $[M+H]^+$ 282.1130, found 282.1139.

(5-(benzyloxy)-1*H*-indol-2-yl)(phenyl)methanone (2f)[5]

Following general procedure, a mixture of aniline **1f** (383 mg, 1 mmol) and phenyl boronic acid (134 mg, 1.1 mmol) was heated at 85 °C for 24 h. The residue was purified by flash chromatography (toluene/ethyl acetate 98/2) and recrystallization (dichloromethane/hexane) to afford **2f** as yellow powder (240 mg, 73%): mp 190-191 °C (lit.: 187-188 °C); IR (CH_2Cl_2, cm^{-1}) 3444, 3035, 1629, 1522, 1244, 1233, 1167, 1122; ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.32 (brs, 1H), 7.98 (dt, J = 6.9, 1.5 Hz, 2H), 7.62 (tt, J = 7.3, 1.5, 1H), 7.53 (tt, J = 6.9, 7.3, 1.4 Hz, 2H), 7.42-7.30 (m, 6H), 7.16-7.12 (m, 2H), 7.07 (dd, J = 2.1, 0.8 Hz, 1H), 5.10 (s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 187.0, 154.0, 138.0, 137.2, 134.8, 133.2, 132.3, 129.2, 128.6, 128.4, 128.0, 127.9, 127.5, 119.0, 113.1, 112.3, 104.5, 70.7; MS (ES^+) *m/z* 328 $[M+H]^+$.

(4,5,6-trimethoxy-1*H*-indol-2-yl)(phenyl)methanone (2g)

Following general procedure, a mixture of aniline **1g** (367 mg, 1 mmol) and phenyl boronic acid (134 mg, 1.1 mmol) was heated at 85 °C for 24 h. The residue was purified by flash chromatography (cyclohexane/ethyl acetate 8/2) and recrystallization (dichloromethane/hexane) to afford **2g** as yellow needles (202 mg, 65%): mp 172-173 °C; IR ($CHCl_3$, cm^{-1}) 3444, 3057, 2937, 2832, 1622, 1573, 1510, 1504, 1272, 1143; ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.42 (brs, 1H), 7.97 (dd, J = 8.4, 1.5 Hz, 2H), 7.60 (t, J = 7.4 Hz, 1H), 7.52 (t, J = 7.6 Hz, 2H), 7.19 (d, J = 1.6 Hz, 1H), 6.64 (s, 1H), 4.01 (s, 3H), 3.91 (s, 3H), 3.86 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 186.2, 155.2, 147.1, 138.2, 136.3, 135.3, 133.1, 132.1, 129.1,

[5] Mahboobi, S.; Pongratz, H.; Hufsky, H.; Hockemeyer, J.; Frieser, M. *et al. J. Med. Chem*. **2001**, *44*, 4535.

128.4, 116.2, 111.3, 88.9, 61.5, 61.0, 56.2; MS (ES^+) *m/z* 312 $[M+H]^+$; HRMS calcd for $C_{18}H_{18}NO_4$ $[M+H]^+$ 312.1236, found 312.1223.

(1*H*-indol-2-yl)(4-methoxyphenyl)methanone (2h)[6]

Following general procedure, a mixture of aniline **1a** (277 mg, 1 mmol) and 4-methoxyphenyl boronic acid **3h** (167 mg, 1.1 mmol) was heated at 100 °C for 16 h. The residue was purified by flash chromatography (toluene/ethyl acetate 97/3) and recrystallization (dichloromethane/hexane) to afford **2h** as yellow needles (154 mg, 65%): mp 189-190 °C (lit. 190-191 °C) ; IR (CH_2Cl_2, cm^{-1}) 3443, 3054, 2841, 1626, 1604, 1524, 1508, 1340, 1310, 1270, 1250, 1170, 1124, 1031; ^1H NMR (300 MHz, $CDCl_3$) δ 9.52 (brs, 1H), 8.05 (dt, *J* = 8.8, 1.9 Hz, 2H), 7.72 (d, *J* = 7.9 Hz, 1H), 7.49 (dd, *J* = 8.0, 0.5 Hz, 1H), 7.37 (td, *J* = 8.0, 0.9 Hz, 1H), 7.17 (td, *J* = 7.9, 0.7 Hz, 1H), 7.17 (s, 1H), 7.03 (dt, *J* = 8.8, 1.9, 2H), 3.91 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 185.8, 163.2, 137.3, 134.5, 131.6, 130.6, 127.8, 126.2, 123.1, 121.0, 113.8, 112.2, 111.8, 55.5; MS (ES^+) *m/z* 274 $[M+Na]^+$; HRMS calcd for $C_{16}H_{14}NO_2$ $[M+H]^+$ 252.1025, found 252.1014.

(1*H*-indol-2-yl)(3,4,5-trimethoxyphenyl)methanone (2i)[5]

Following general procedure, a mixture of aniline **1a** (277 mg, 1 mmol) and 3,4,5-trimethoxyphenyl boronic acid **3i** (233 mg, 1.1 mmol) was heated at 100 °C for 16 h. The residue was purified by flash chromatography (toluene/ethyl acetate 95/5) and recrystallization (dichloromethane/hexane) to afford **2i** as beige needles (196 mg, 63%): mp 153-154 °C (lit. 148-150 °C); IR (CH_2Cl_2, cm^{-1}) 3442, 2941, 2839, 1625, 1582, 1522, 1503, 1464, 1413, 1340, 1328, 1220, 1129; ^1H NMR (300 MHz, $CDCl_3$) δ 9.22 (brs, 1H), 7.74 (dd, *J* = 8.1, 0.9 Hz, 1H), 7.48 (dd, *J* = 8.3, 0.9 Hz, 1H), 7.38 (ddd, *J* = 8.3, 8.1, 0.9 Hz, 1H), 7.27 (s, 2H), 7.18 (td, *J* = 8.1, 1.0 Hz, 1H), 7.18 (dd, *J* = 2.0, 0.9 Hz, 1H), 3.96 (s, 3H), 3.95 (s, 6H);); ^{13}C NMR (75 MHz, $CDCl_3$) δ 186.1, 153.1, 142.0, 137.4, 134.2, 133.1, 127.7, 126.5, 123.2, 121.1, 112.1, 106.8, 61.0, 56.4; MS (ES^+) *m/z* 312 $[M+H]^+$, 334 $[M+Na]^+$.

(1*H*-indol-2-yl)(2-methoxyphenyl)methanone (2j)[6]

Following general procedure, a mixture of aniline **1a** (277 mg, 1 mmol) and (2-methoxy)phenyl boronic acid **3j** (167 mg, 1.1 mmol) was heated at 100 °C for 16 h. The residue was purified by flash chromatography (toluene/ethyl acetate 95/5) and

[6] Katritzky, A. R.; Akutagawa, K. *Tetrahedron Lett.* **1985**, 26, 5935

recrystallization (dichloromethane/hexane) to afford **2j** as yellow crystals (100 mg, 40%): mp 133-134 °C (lit. 129-130 °C); IR (CH_2Cl_2, cm^{-1}) 3444, 3063, 3047, 2944, 2839, 1637, 1525, 1488, 1339, 1311, 1268, 1252, 1128, 1106; ^1H NMR (300 MHz, $CDCl_3$) δ 9.25 (brs, 1H), 7.65 (dd, J = 8.0, 0.8 Hz, 1H), 7.53-7.49 (m, 2H), 7.45 (dd, J = 8.3, 0.9 Hz, 1H), 7.35 (ddd, J = 8.3, 8.1, 1.1 Hz, 1H), 7.13 (ddd, J = 8.1, 8.0, 1.0 Hz, 1H), 7.05 (t, J = 7.4 Hz, 1H), 7.04 (d, J = 7.7 Hz, 1H), 6.92 (dd, J = 2.0, 0.9 Hz, 1H), 3.83 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 187.1, 157.4, 137.7, 135.8, 132.0, 129.7, 128.1, 127.6, 126.5, 123.3, 120.9, 120.1, 113.1, 112.2, 111.6, 55.8; MS (ES^+) *m/z* 252 $[M+H]^+$.

(4-(trifluoromethyl)phenyl)(1*H*-indol-2-yl)methanone (2l)

Following general procedure, a mixture of aniline **1a** (277 mg, 1 mmol) and (4-trifluoromethyl)phenyl boronic acid **3l** (209 mg, 1.1 mmol) was heated at 100 °C for 16 h. The residue was purified by flash chromatography (toluene 100%) and recrystallization (dichloromethane/hexane) to afford **2l** as pale yellow needles (211 mg, 73%): mp 188-189 °C; IR (CH_2Cl_2, cm^{-1}) 3442, 1635, 1524, 1408, 1325, 1270, 1244, 1172, 1126, 1066; ^1H NMR (300 MHz, $CDCl_3$) δ 9.39 (brs, 1H), 8.80 (d, J = 8.0 Hz, 2H), 7.81 (d, J = 8.0 Hz, 2H), 7.73 (dd, J = 8.1, 0.8 Hz, 1H), 7.49 (dd, J = 8.4, 0.9 Hz, 1H), 7.41 (ddd, J = 8.4, 8.0, 1.1 Hz, 1H), 7.19 (ddd, J = 8.1, 8.0, 1.0 Hz, 1H), 7.15 (dd, J = 2.1, 0.9 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 185.9, 141.0, 137.8, 133.9, 133.7 (32 Hz), 129.4, 127.7, 127.1, 125.5 (3.5 Hz), 123.4, 121.4, 119.9 (296 Hz), 113.4, 112.2; MS (ES^-) *m/z* 288 $[M-H]^+$; HRMS calcd for $C_{16}H_9NOF_3$ $[M+H]^+$ 288.0636, found 288.0639.

(4-chlorophenyl)(1*H*-indol-2-yl)methanone (2m)[7]

Following general procedure, a mixture of aniline **1a** (277 mg, 1 mmol) and 4-chlorophenyl boronic acid **3m** (172 mg, 1.1 mmol) was heated at 100 °C for 16 h. The residue was purified by flash chromatography (toluene 100%) and recrystallization (dichloromethane/hexane) to afford **2m** as bright yellow needles (179 mg, 70%): mp 196-197 °C (lit. 196-197 °C) ; IR (CH_2Cl_2, cm^{-1}) 3443, 1630, 1591, 1523, 1340, 1312, 1264, 1250, 1124; ^1H NMR (300 MHz, $CDCl_3$) δ 9.28 (brs, 1H), 7.95 (dt, J = 8.7, 2.3 Hz, 2H), 7.72 (dd, J = 8.1, 0.9 Hz, 1H), 7.51 (dt, J = 8.7, 2.0 Hz, 2H), 7.48 (dd, J = 8.3, 0.9 Hz, 1H), 7.39 (ddd, J = 8.3, 8.0, 1.1 Hz, 1H), 7.18 (ddd, J = 8.1, 8.0, 1.1 Hz, 1H), 7.14 (dd, J = 2.1, 0.9 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 185.8, 138.9, 137.6, 136.3, 134.0, 130.6, 128.8, 127.7, 126.8, 123.3, 121.2, 112.7,

[7] Takeda, Y.; Kikuchi, A.; Terashima, M. *Heterocycles*, **1993**, *35*, 573.

112.2; MS (ES^+) *m/z* 278 (^{35}Cl) $[M+Na]^+$ 280 (^{37}Cl) $[M+Na^+]$; HRMS calcd for $C_{15}H_{11}NO^{35}Cl$ $[M+H]^+$ 256.0529, found 256.0531.

(1*H*-indol-2-yl)(4-(N-methylaminocarbonyl)phenyl)methanone (2n)

Following general procedure, a mixture of aniline **1a** (277 mg, 1 mmol) and 4-(N-methylaminocarbonyl)phenyl boronic acid **3n** (197 mg, 1.1 mmol) was heated at 100 °C for 16 h. The residue was purified by flash chromatography (cyclohexane/ethyl acetate 1/1) and recrystallization (dichloromethane/hexane) to afford **2n** as white powder (80 mg, 29%): mp 232-234 °C ; IR (KBr, cm^{-1}) 3318, 2932, 1623, 1548, 1409, 1384, 1345, 1321, 1263, 1012; ^1H NMR (300 MHz, $CDCl_3$) δ 9.28 (brs, 1H), 8.0 (dt, *J* = 8.6, 2.0 Hz, 2H), 7.91 (dt, *J* = 8.6, 2.0 Hz, 2H), 7.72 (dd, *J* = 8.0, 0.9 Hz, 1H), 7.48 (dd, *J* = 8.4, 1.0 Hz, 1H), 7.39 (ddd, *J* = 8.4, 7.9, 1.1 Hz, 1H), 7.18 (ddd, *J* = 8.0, 7.9, 1.0 Hz, 1H), 7.14 (dd, *J* = 2.1, 1.0 Hz, 1H), 6.24 (brs, 1H), 3.07 (d, 4.9 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 186.2, 167.3, 140.3, 137.9, 137.6, 134.0, 129.4, 127.7, 127.0, 126.9, 123.4, 121.3, 113.1, 112.1, 27.0; MS (ES^+) *m/z* 279 $[M+H]^+$; HRMS calcd for $C_{17}H_{15}N_2O_2$ $[M+H]^+$ 279.1134, found 279.1121.

(*E*)-1-(1*H*-indol-2-yl)-3-phenylprop-2-en-1-one (2o)

Following general procedure, a mixture of aniline **1a** (277 mg, 1 mmol) and (*E*)-phenylvinyl boronic acid **3o** (163 mg, 1.1 mmol) was heated at 100 °C for 16 h. The residue was purified by flash chromatography (toluene 100%) and recrystallization (dichloromethane/hexane) to afford **2o** as bright yellow needles (166 mg, 67%): mp 219-220 °C ; IR (CH_2Cl_2, cm^{-1}) 3443, 3054, 1651, 1596, 1523, 1268, 1166, 1142; ^1H NMR (300 MHz, $CDCl_3$) δ 9.29 (brs, 1H), 7.92 (d, *J* = 15.7 Hz, 1H), 7.75 (dd, *J* = 8.0, 0.9 Hz, 1H), 7.70-7.67 (m, 2H), 7.53 (d, *J* = 15.7 Hz, 1H), 7.48-7.43 (m, 4H), 7.40-7.37 (m, 2H), 7.17 (dd, *J* = 8.0, 1.0 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 181.1, 143.3, 137.6, 136.5, 134.8, 130.6, 129.0, 128.5, 127.8, 126.5, 123.2, 121.5, 121.1, 112.2, 109.4; MS (ES^+) *m/z* 248 $[M+H]^+$; HRMS calcd for $C_{17}H_{14}NO$ $[M+H]^+$ 248.1075, found 248.1075.

(1*H*-indol-2-yl)(thiophen-3-yl)methanone (2p)[3]

Following general procedure, a mixture of aniline **1a** (277 mg, 1 mmol) and thiophene-3-boronic acid **3p** (140 mg, 1.1 mmol) was heated at 100 °C for 16 h. The residue was purified by flash chromatography (toluene/ethyl acetate 98/2) and recrystallization (dichloromethane/hexane) to afford **2p** as beige needles (152 mg, 67%): mp 153-154 °C (lit. 140-143 °C) ; IR (CH_2Cl_2, cm^{-1}) 3443, 3112, 3063, 1621, 1525, 1417, 1339, 1309, 1247; ^1H

NMR (300 MHz, $CDCl_3$) δ 9.38 (brs, 1H), 8.22 (dd, *J* = 2.9, 1.2 Hz, 1H), 7.73 (dd, *J* = 8.0, 0.9 Hz, 1H), 7.71 (dd, *J* =5.0, 1.2 Hz, 1H), 7.48 (dd, *J* = 8.3, 0.9 Hz, 1H), 7.44 (dd, *J* = 5.0, 2.9 Hz, 1H), 7.37 (ddd, J = 8.3, 8.1, 1.1 Hz, 1H), 7.30 (dd, *J* = 2.1, 0.9 Hz, 1H), 7.18 (ddd, *J* = 8.1, 8.0, 1.0 Hz, 1H);); ^{13}C NMR (75 MHz, $CDCl_3$) δ 180.3, 141.0, 137.4, 134.8, 132.0, 128.1, 127.8, 126.4, 123.2, 121.1, 112.1, 111.2; MS (ES^+) *m/z* 228 [M+H].

(benzofuran-2-yl)(1*H*-indol-2-yl)methanone (2q)[8]

Following general procedure, a mixture of aniline **1a** (277 mg, 1 mmol) and benzofuran-2-boronic acid **3q** (149 mg, 1.1 mmol) was heated at 100 °C for 16 h. The residue was purified by flash chromatography (toluene/ethyl acetate 98/2) and recrystallization (ethyl acetate) to afford **2q** as a bright yellow powder (152 mg, 58%): mp 240-241 °C (lit. 242 °C); IR (CH_2Cl_2, cm^{-1}) 3442, 3063, 1722, 1620, 1558, 1518, 1340, 1312, 1218, 1173, 1143; ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.39 (brs, 1H), 7.89 (dd, *J* = 2.1, 0.9 Hz, 1H), 7.81 (dq, *J* = 8.1, 1.0 Hz, 1H), 7.79 (d, *J* = 0.9 Hz, 1H), 7.77 (ddd, *J* = 7.9, 1.2, 0.8 Hz, 1H), 7.69 (dq, *J* = 8.4, 0.9 Hz, 1H), 7.53 (ddd, *J* = 8.4, 8.0, 1.2 Hz, 1H), 7.49 (dq, *J* = 8.3, 0.9 Hz, 1H), 7.40 (ddd, *J* = 8.3, 8.0, 1.1 Hz, 1H), 7.36 (ddd, *J* = 8.0, 7.9, 0.9 Hz, 1H), 7.19 (ddd, *J* = 8.1, 8.0, 1.0 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 174.0, 155.9, 152.9, 137.5, 133.7, 128.1, 128.0, 127.1, 126.9, 124.0, 123.5, 123.2, 121.2, 114.2, 112.4, 112.3, 112.1; MS (ES^-) *m/z* 260 $[M-H]^+$; HRMS calcd for $C_{17}H_{11}NO_2Na$ $[M+Na]^+$ 284.0687, found 284.0690.

(1*H*-indol-2-yl)(dibenzofuran-4-yl)methanone (2r)

Following general procedure, a mixture of aniline **1a** (277 mg, 1 mmol) and dibenzofuran-4-boronic acid **3r** (233 mg, 1.1 mmol) was heated at 100 °C for 16 h. The residue was purified by flash chromatography (toluene 100%) and recrystallization (dichloromethane/hexane) to afford **2r** as a white powder (221 mg, 71%): mp 180-181 °C ; IR (CH_2Cl_2, cm^{-1}) 3443, 3065, 3046, 1638, 1620, 1522, 1415, 1340, 1312, 1270, 1264, 1189, 1137; ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.55 (brs, 1H), 8.18 (td, *J* = 7.6, 1.3 Hz, 1H), 8.01 (ddd, *J* = 7.6, 1.3, 0.6 Hz, 1H), 7.95 (td, *J* = 7.6, 1.3 Hz, 1H), 7.70 (dq, *J* = 8.1, 0.8 Hz, 1H), 7.63 (dt, *J* = 8.2, 0.9 Hz, 1H), 7.55 (dq, *J* = 8.3, 0.9 Hz, 1H), 7.50 (ddd, *J* = 7.6, 7.3, 0.9 Hz, 1H), 7.40 (ddd, *J* = 8.2, 7.3, 1.3 Hz, 1H), 7.40 (ddd, *J* = 8.3, 8.0, 1.0 Hz, 1H), 7.18 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 184.5, 156.4, 153.7, 137.9, 135.0, 128.1, 127.9, 127.7, 126.8, 125.8, 124.1, 123.4, 123.3,

[8] Mahboobi, S.; Uecker, A.; Cenac, C.; Sellmer, A.; Eichhorn, E.; Elz, S.; Böhmer, F.-D.; Dove, S. *Bioorg. Med. Chem.* **2007**, *15*, 2187.

123.2, 122.4, 121.1, 120.7, 113.5, 112.3, 112.1; MS (ES^+) *m/z* 312 $[M+H]^+$; HRMS calcd for $C_{21}H_{13}NO_2Na$ $[M+Na]^+$ 334.0844, found 334.0837.

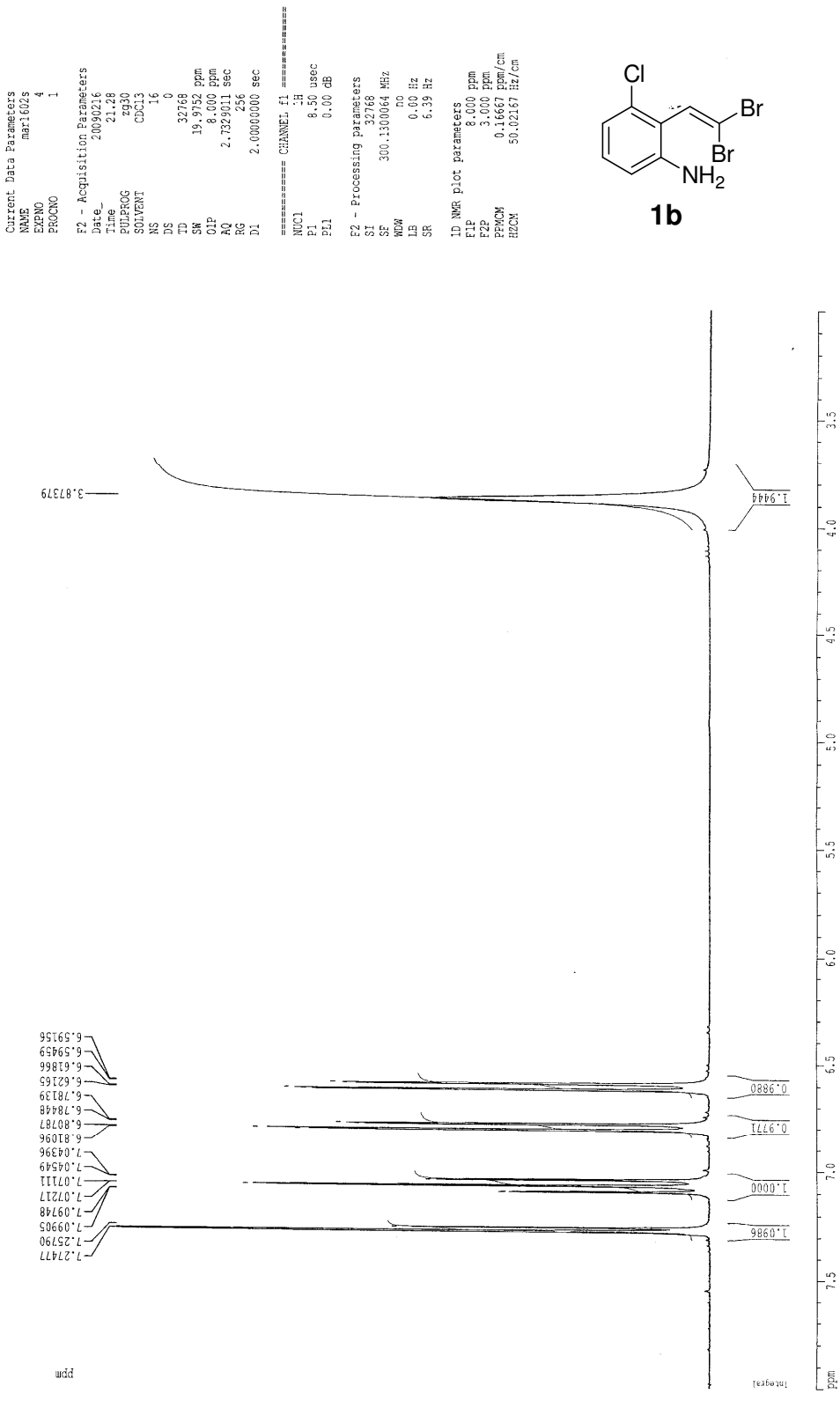
(1*H*-indol-2-yl)(isoquinolin-4-yl)methanone (2s)

Following general procedure, a mixture of aniline **1a** (277 mg, 1 mmol) and isoquinolin-3-boronic acid **3s** (190 mg, 1.1 mmol) was heated at 100 °C for 16 h. The residue was purified by flash chromatography (toluene/ethyl acetate 9/1) and recrystallization (dichloromethane/hexane) to afford **2s** as a yellow powder (57 mg, 21%): mp 226-227 °C ; IR (CH_2Cl_2, cm^{-1}) 3442, 3045, 1632, 1523, 1340, 1270, 1261, 1136 ; ^1H NMR (300 MHz, $CDCl_3$) δ 9.42 (brs, 2H), 8.96 (s, 1H), 8.32 (dd, *J* = 8.1, 0.7 Hz, 1H), 8.10, (d, *J* = 8.3 Hz, 1H), 7.80 (ddd, *J* = 8.4, 8.3, 1.4 Hz, 1H), 7.74-7.67 (m, 2H), 7.52 (dd, *J* = 8.4, 0.8 Hz, 1H), 7.41 (ddd, *J* = 8.4, 8.1, 1.0 Hz, 1H), 7.18 (ddd, *J* = 8.1, 8.0, 0.9 Hz, 1H), 7.08 (dd, *J* = 2.1, 0.9 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 186.4, 155.4, 143.9, 138.1, 135.6, 133.5, 131.9, 128.9, 128.5, 128.2, 128.1, 127.6, 127.2, 124.6, 123.6, 121.3, 114.2, 112.2; MS (ES^+) *m/z* 273$[M+H]^+$; HRMS calcd for $C_{18}H_{13}N_2O$ $[M+H]^+$ 273.1028, found 273.1021.

(1*H*-indol-2-yl)(naphthalen-3-yl)methanone (2t)[9]

Following general procedure, a mixture of aniline **1a** (277 mg, 1 mmol) and 2-naphtalene boronic acid **3t** (209 mg, 1.1 mmol) was heated at 100 °C for 16 h. The residue was purified by flash chromatography (toluene 100%) and recrystallization (dichloromethane/hexane) to afford **2t** as pale yellow needles (190 mg, 70%): mp 174-175 °C (lit. 165 °C) ; IR (CH_2Cl_2, cm^{-1}) 3443, 3061, 1621, 1523, 1339, 1310, 1263, 1228, 1181, 1132; ^1H NMR (300 MHz, $CDCl_3$) δ 9.41 (brs, 1H),8.55 (s, 1H), 8.05 (dd, *J* = 8.5, 1.7 Hz, 1H), 8.00-7.92 (m, 3H), 7.75 (dd, *J* = 8.0, 0.9 Hz, 1H), 7.61-7.57 (m, 2H), 7.51 (dd, *J* = 8.3, 0.9 Hz, 1H), 7.40 (ddd, *J* = 8.3, 8.1, 1.1 Hz, 1H), 7.26 (s, 1H), 7.19 (ddd, *J* = 8.1, 8.0, 1.1 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 187.1, 137.5, 135.3, 134.5, 132.4, 130.6, 129.4, 128.5, 128.2, 127.9, 127.8, 126.9, 126.6, 125.3, 123.3, 121.1, 112.8, 112.2; MS (ES^+) *m/z* 272 $[M+H]^+$, 310 $[M+K]^+$; HRMS calcd for $C_{19}H_{14}NO$ $[M+H]^+$ 272.1075, found 272.1074.

[9] Kar, S.; Lahiri, S. *J. Indian. Chem. Soc*. **1999**, *76*, 607.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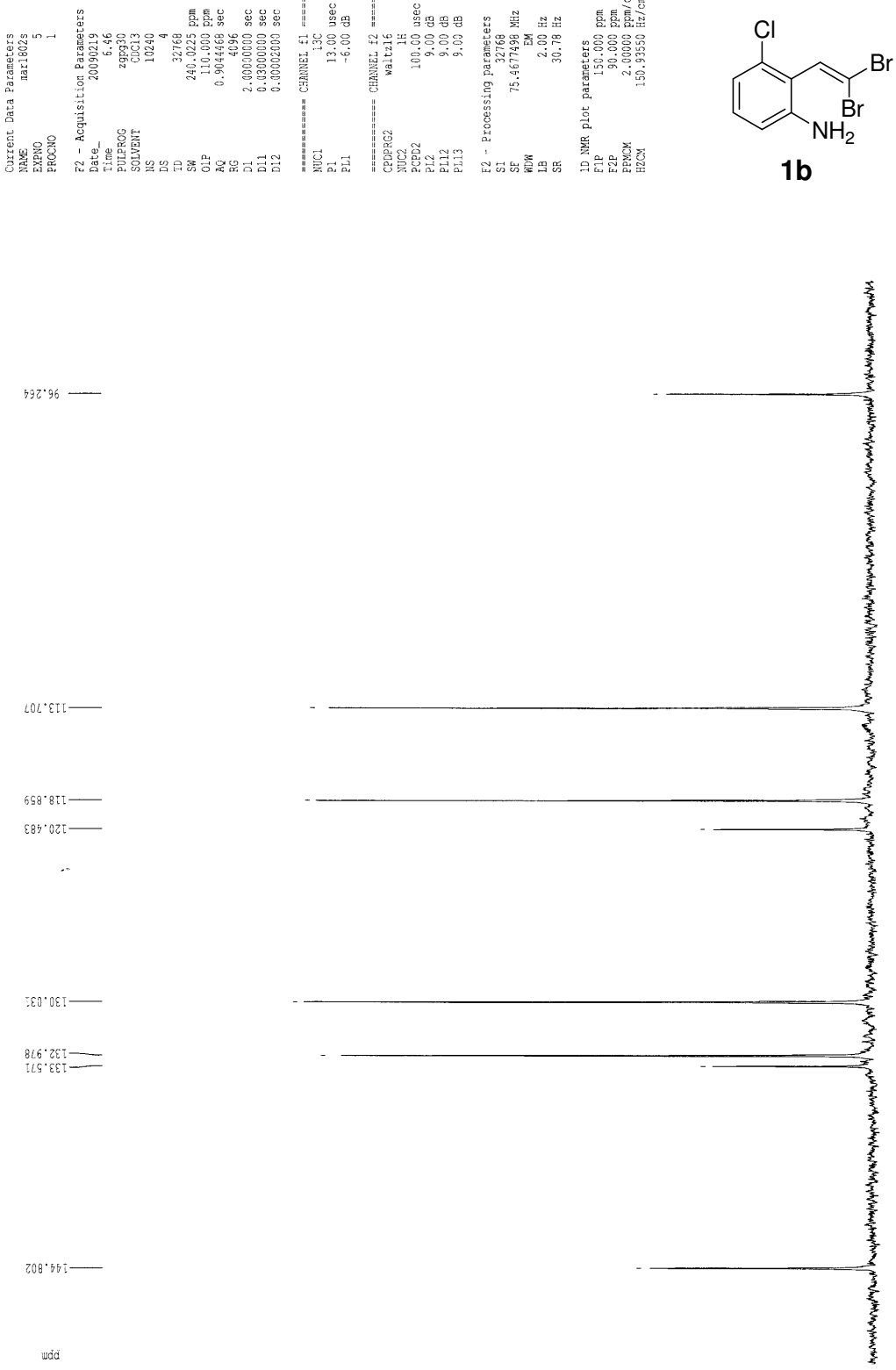
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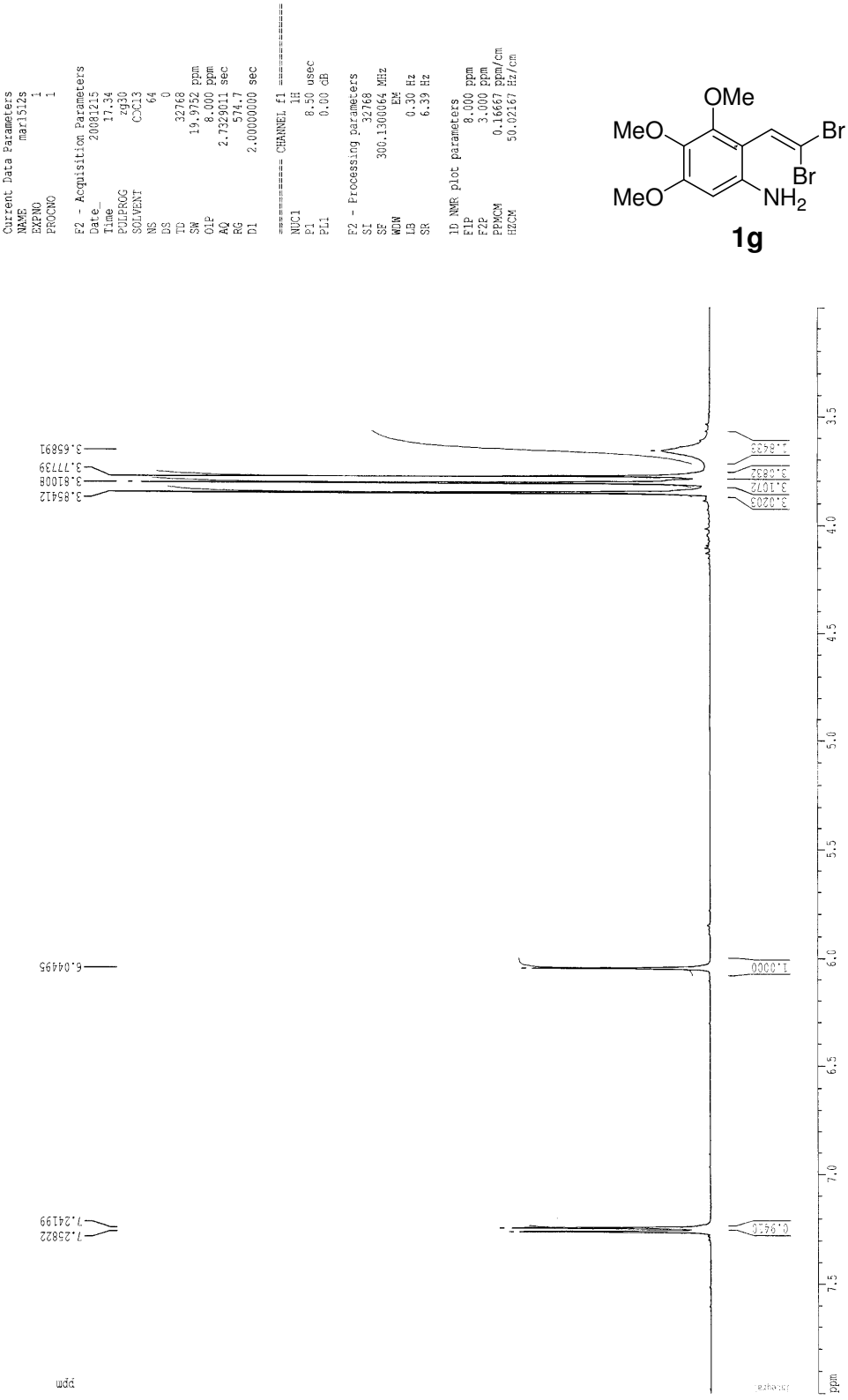
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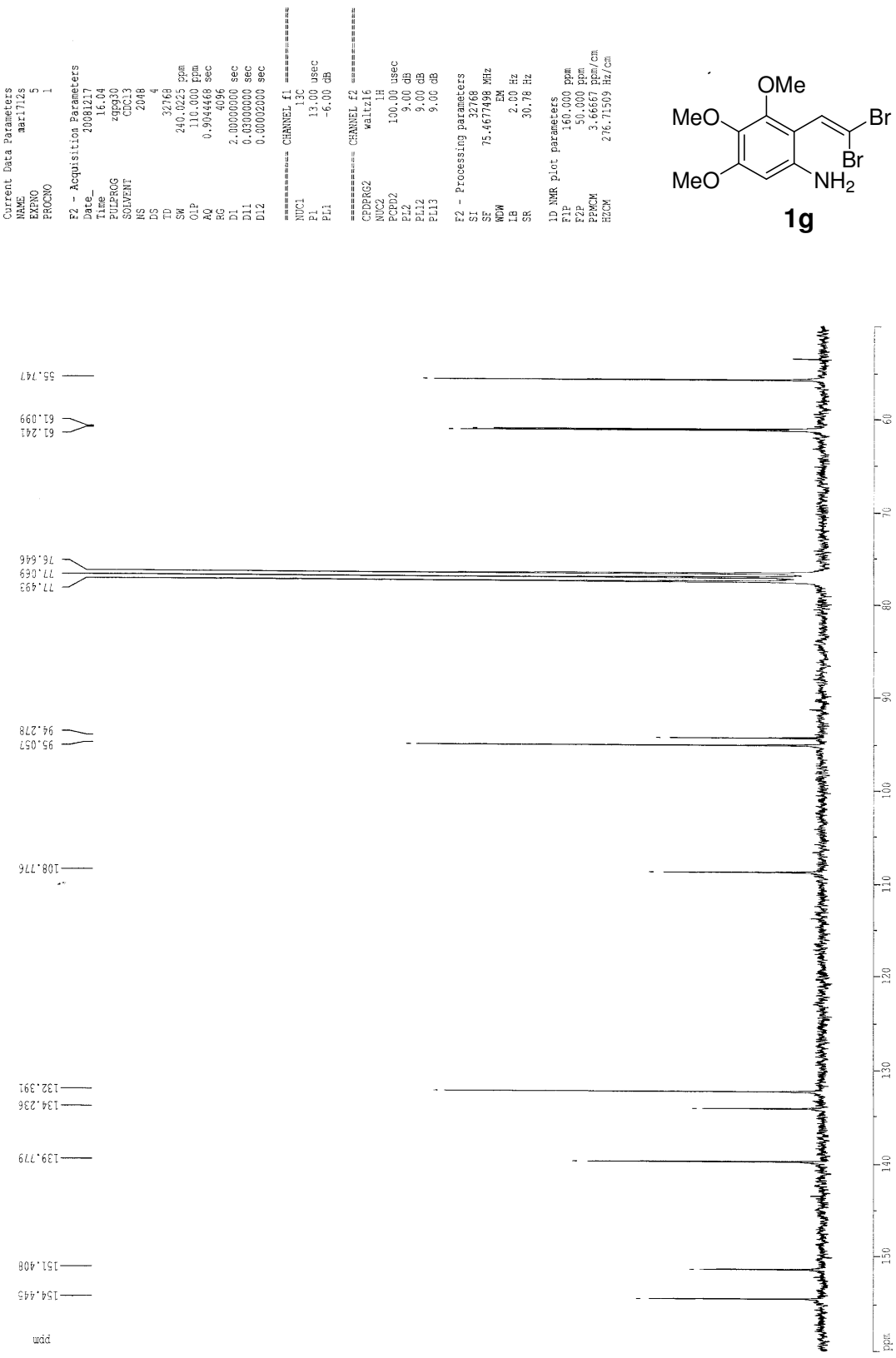
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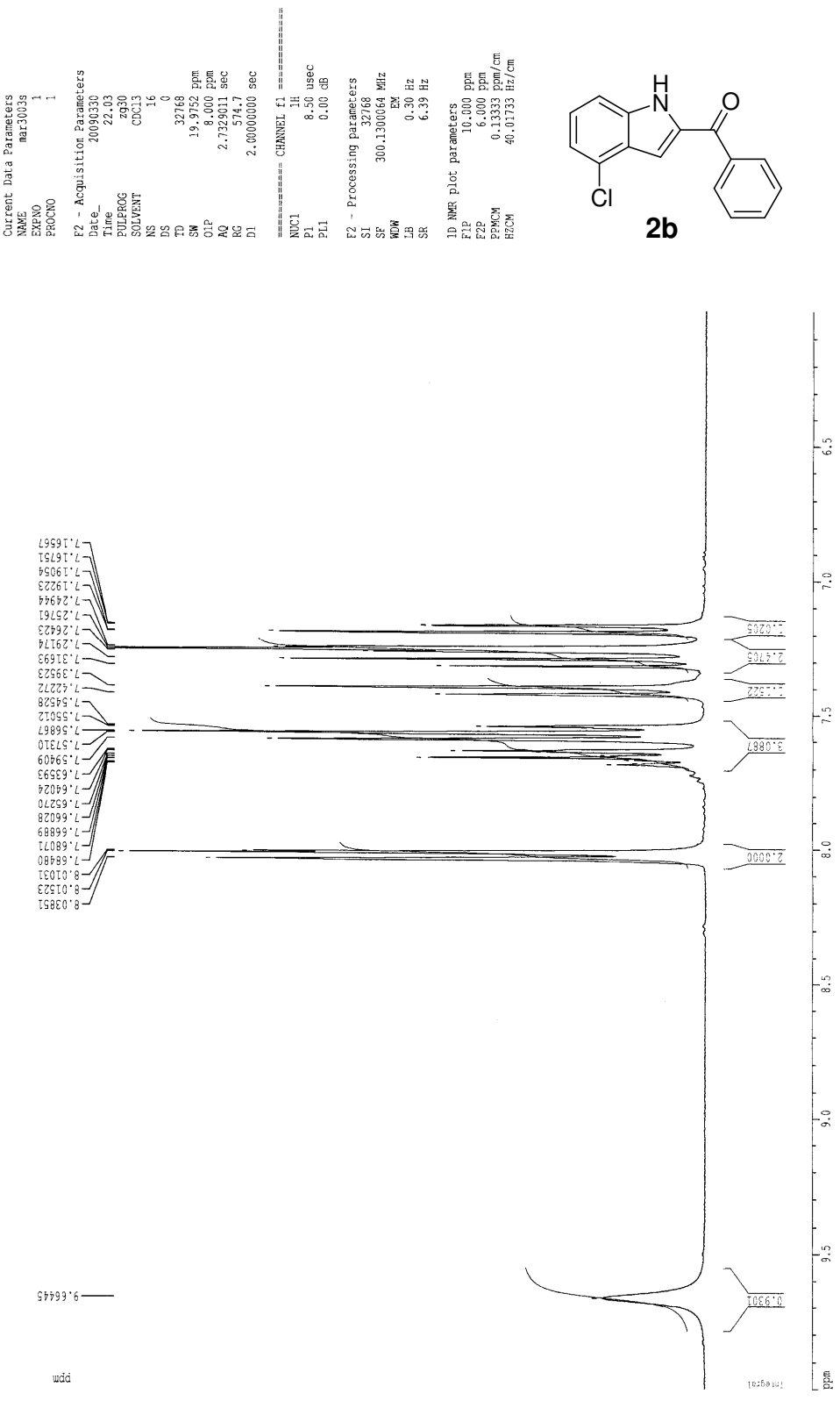
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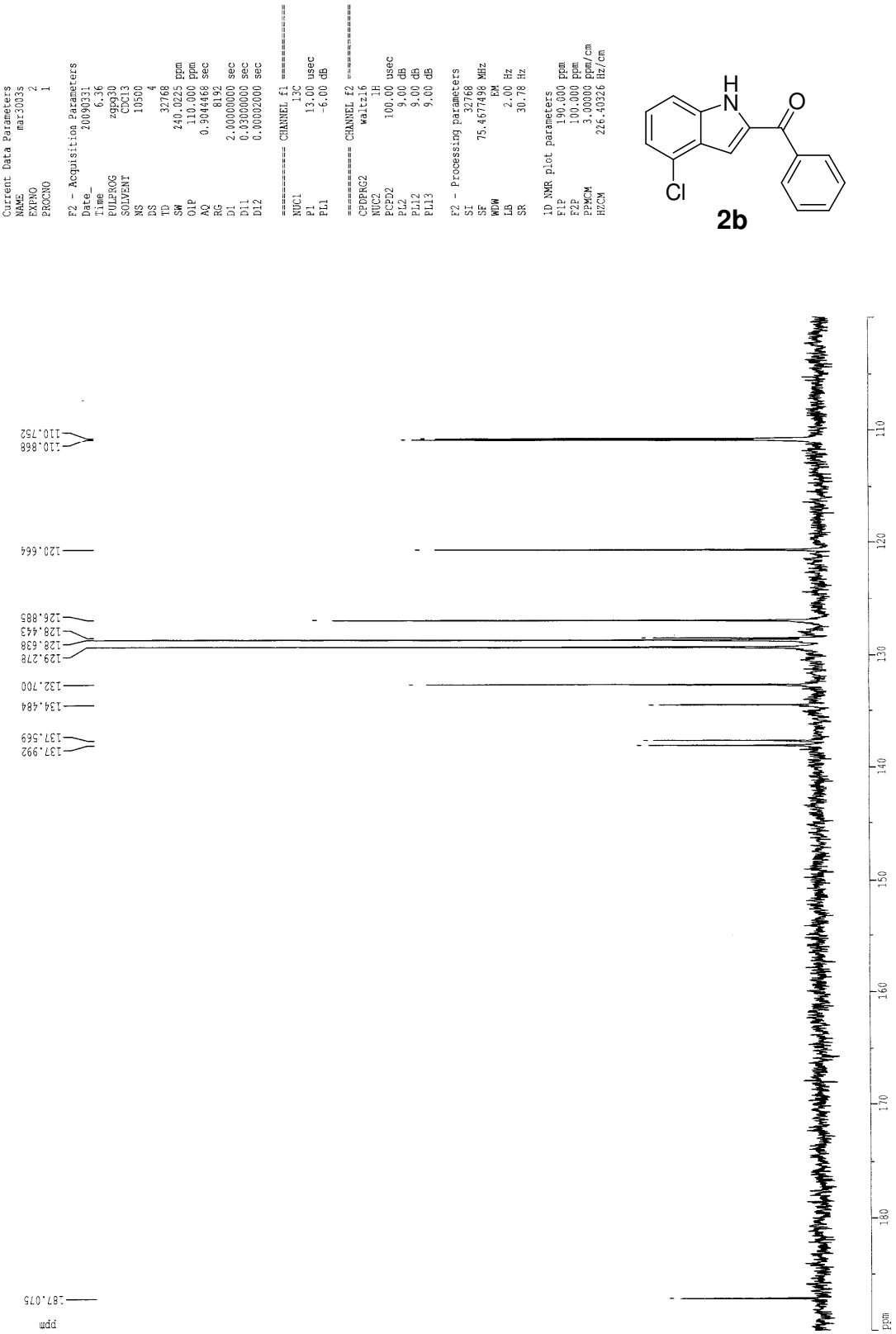
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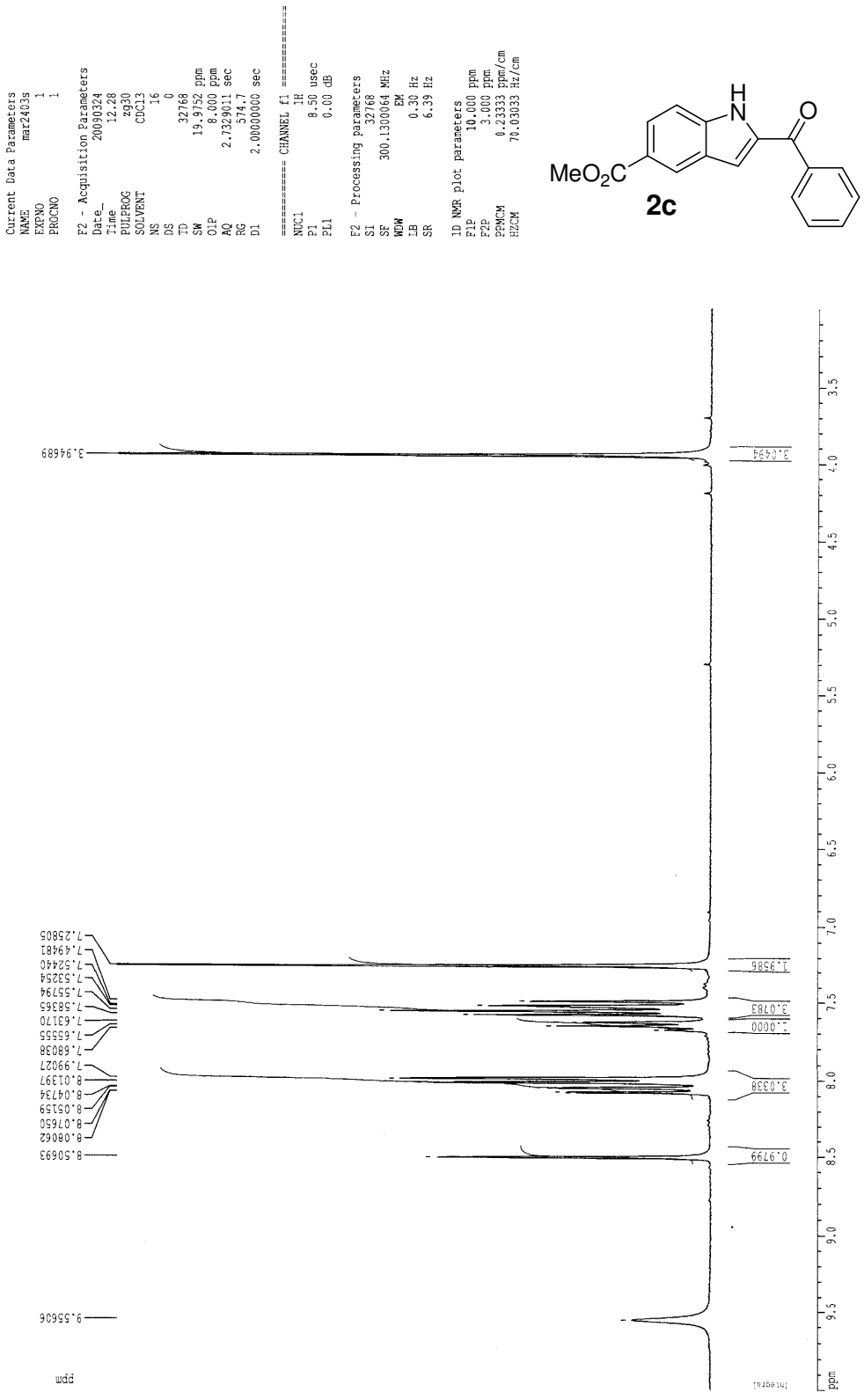
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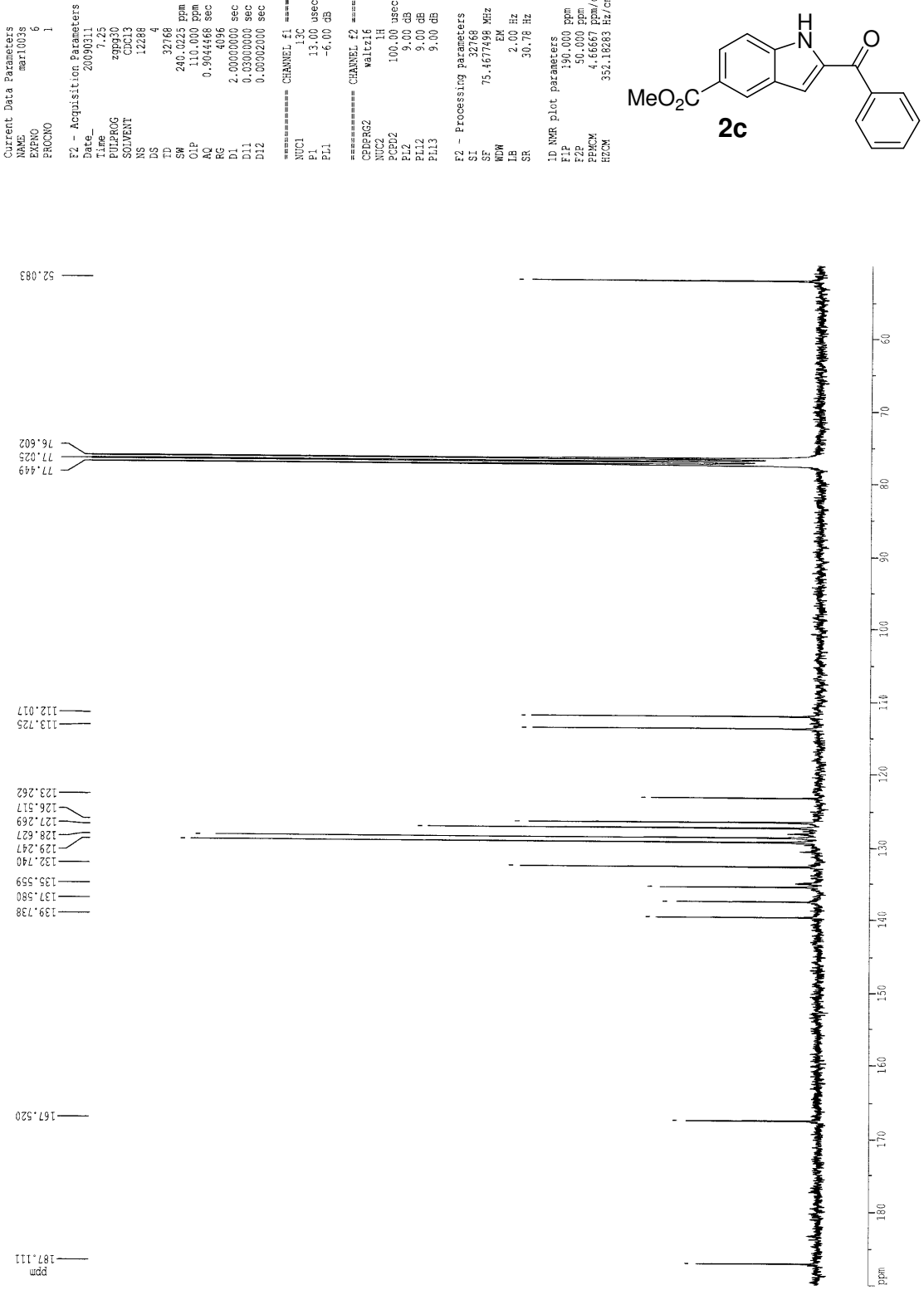
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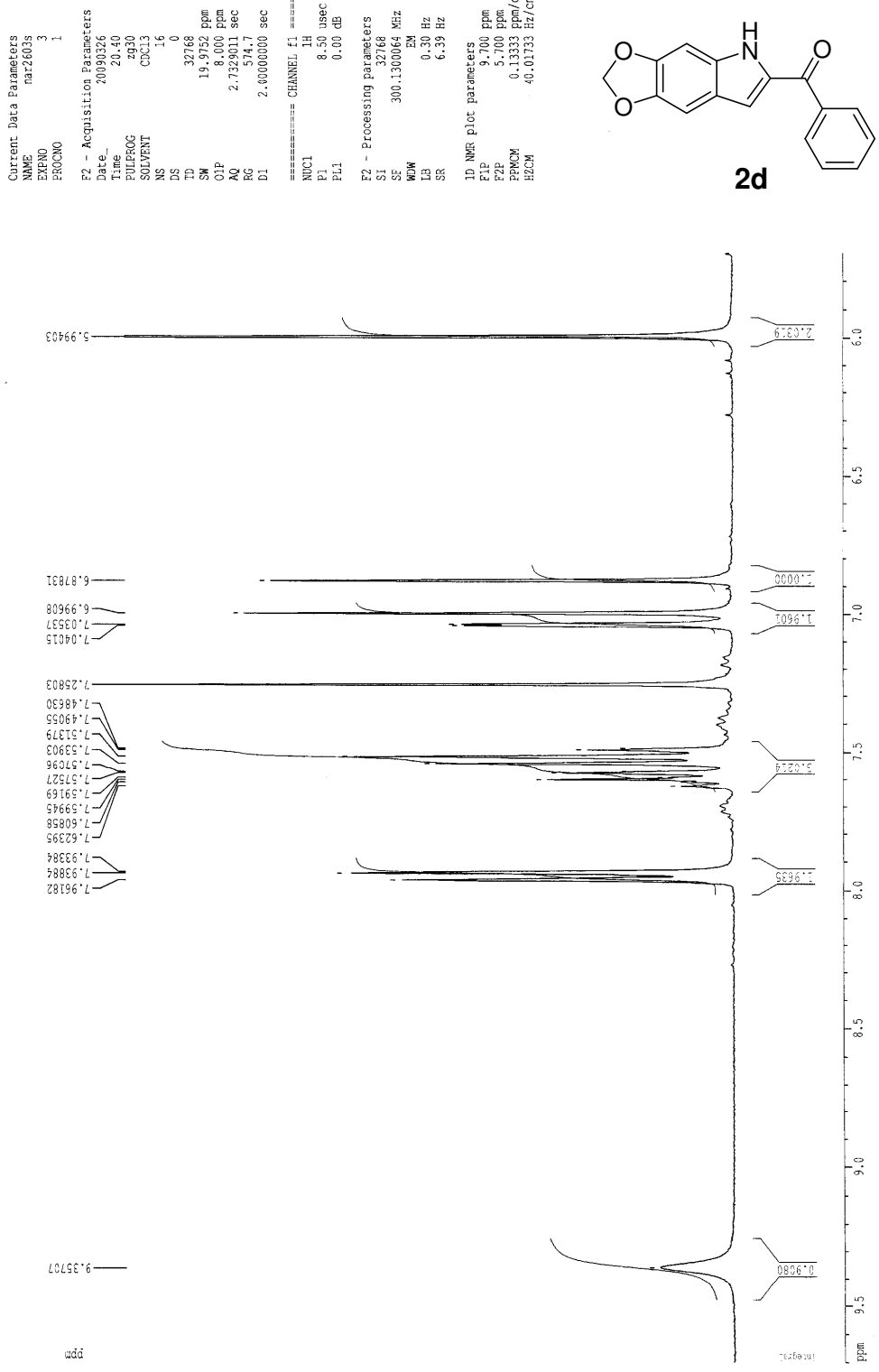
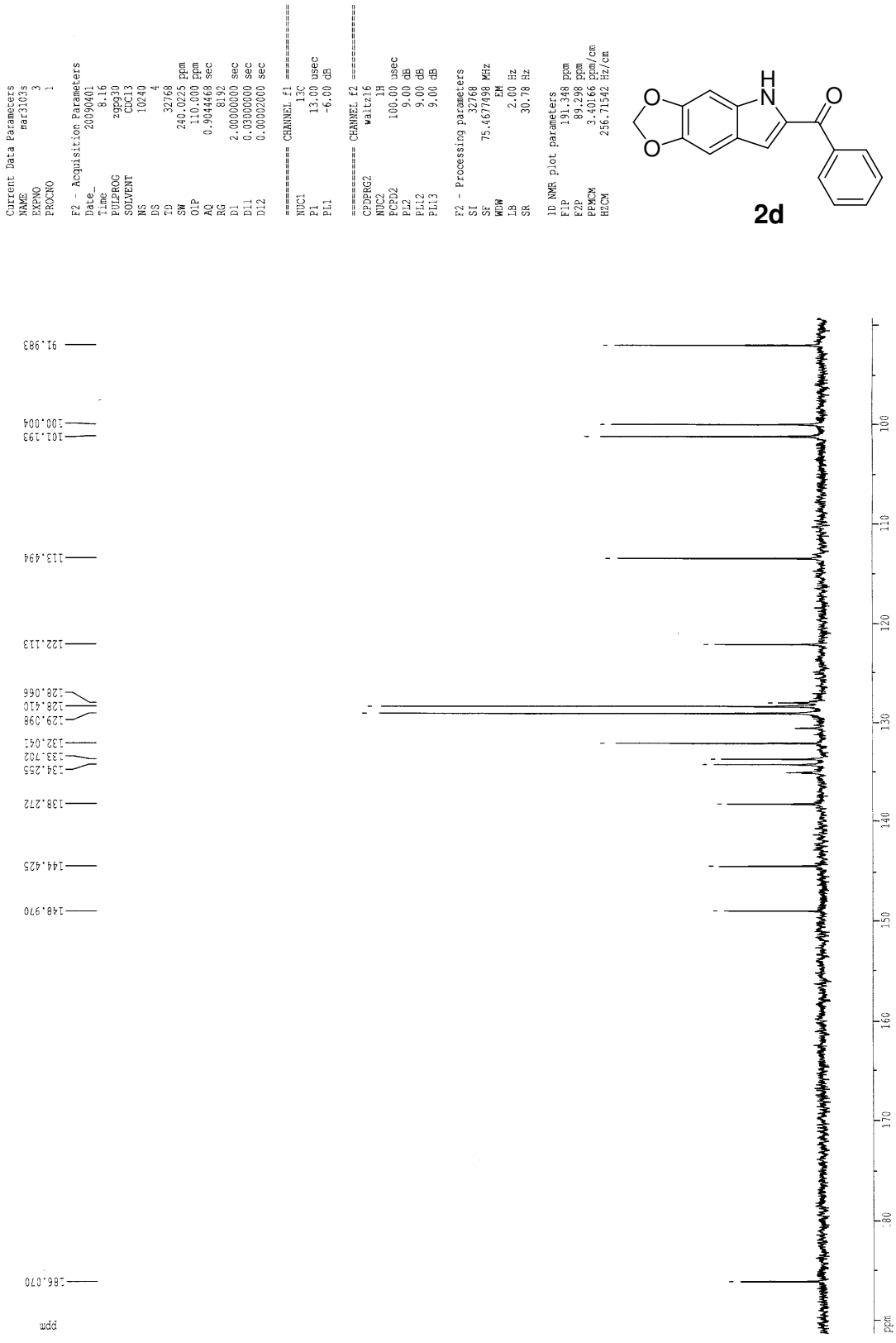
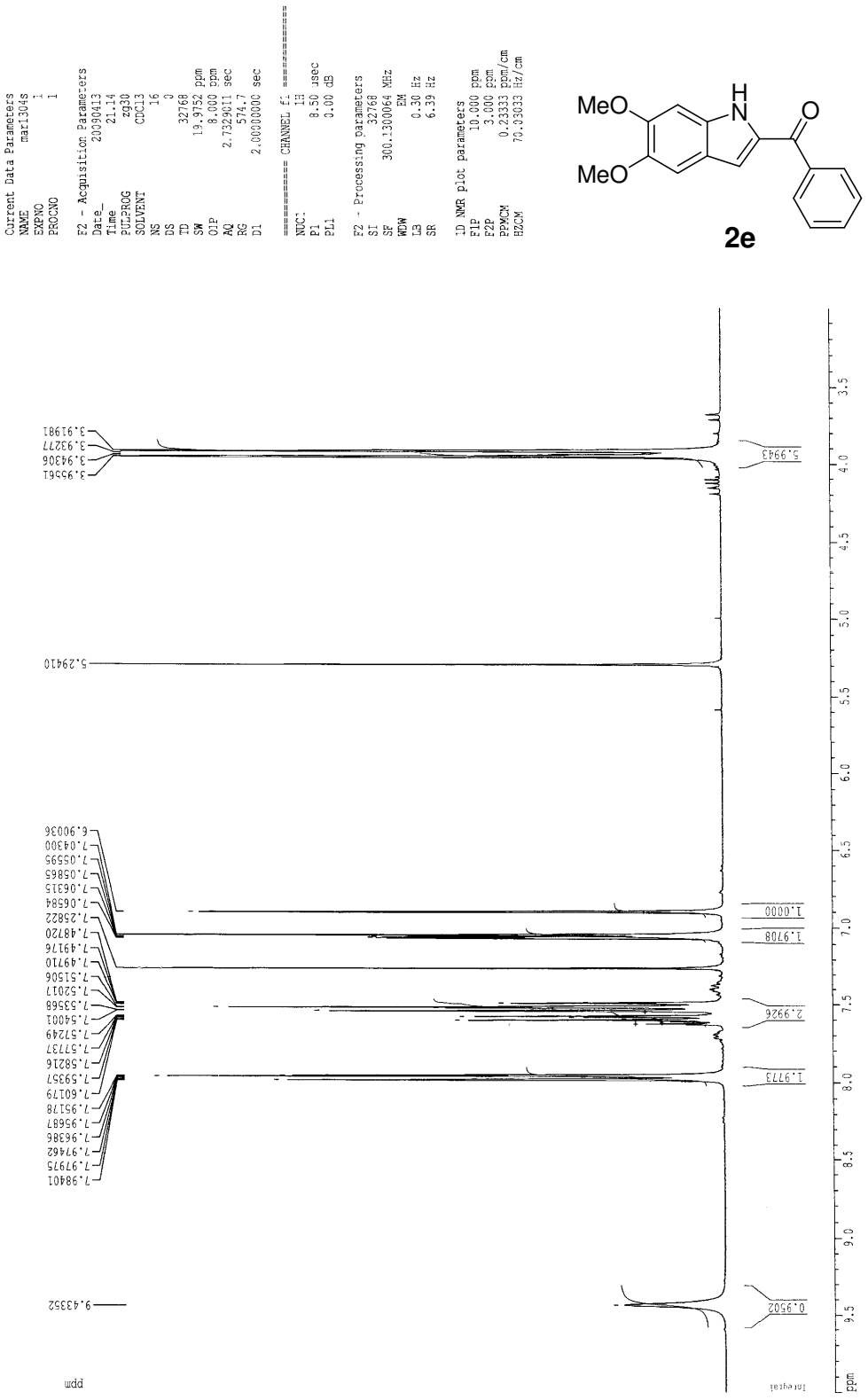
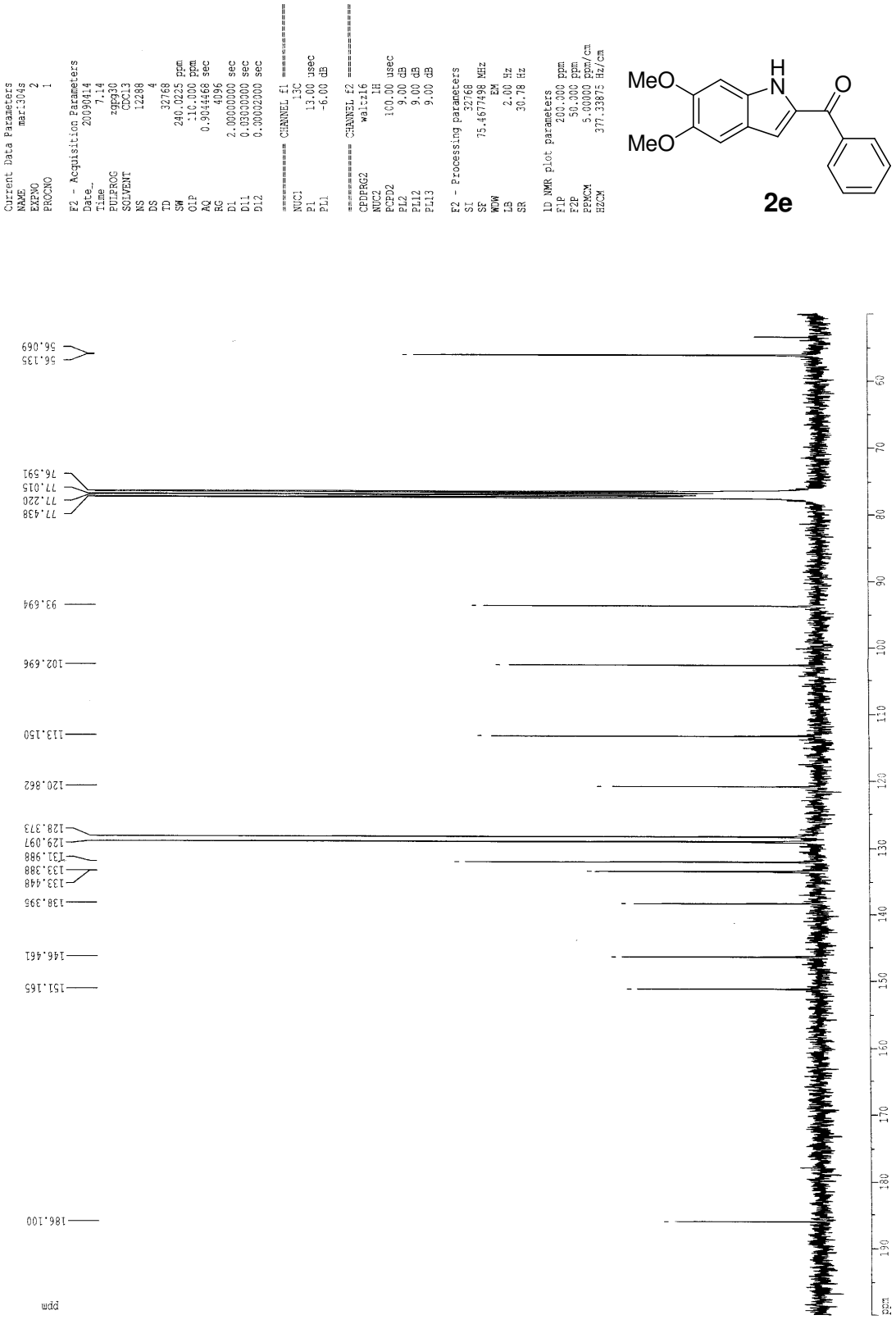
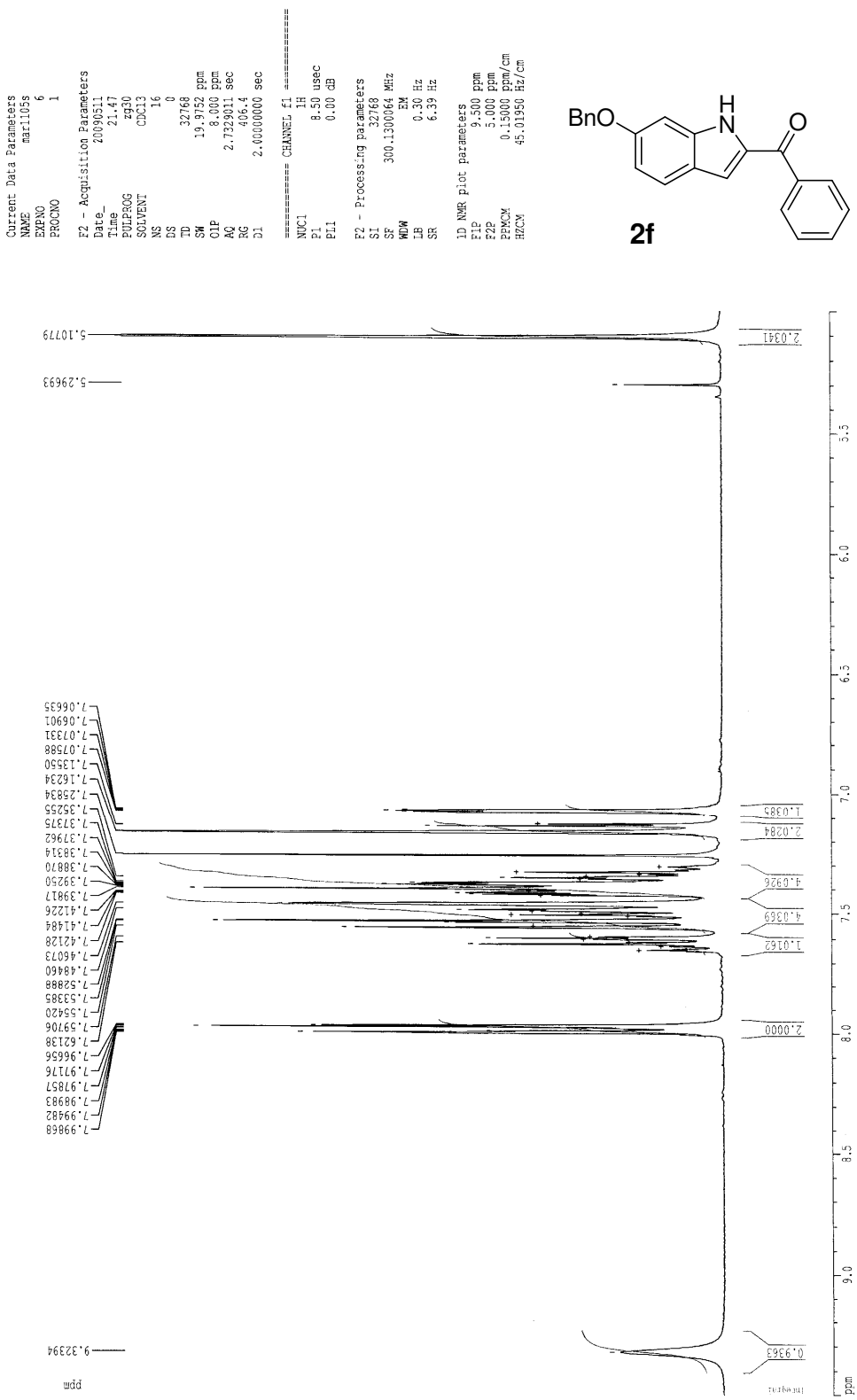
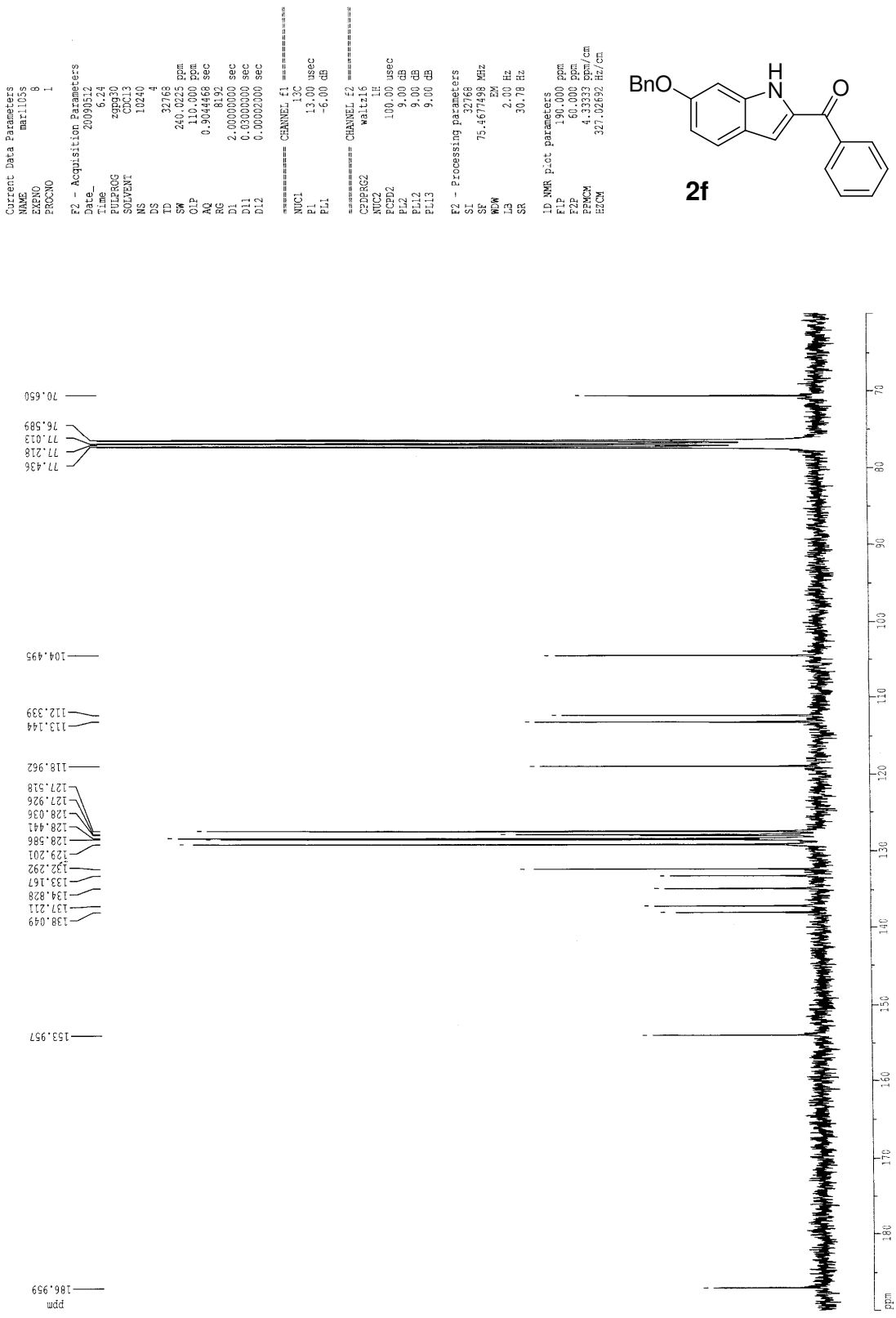

Current Data Parameters
NAME mar2603s
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090326
Time 20.40
PULPROG zg30
SOLVENT CDCl3
NS 16
DS 0
TD 32768
SW 19.9752 ppm
O1P 8.000 ppm
AQ 2.7329011 sec
RG 574.7
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.50 usec
PL1 0.00 dB
F2 - Processing parameters
SI 32768
SF 300.1300064 MHz
WDW EM
LB 0.30 Hz
SR 6.39 Hz
1D NMR plot parameters
F1P 9.700 ppm
F2P 5.700 ppm
PPMCM 0.13333 ppm/cm
HZCM 40.01733 Hz/cm
2d
5.99403
6.87831
6.99608
7.03537
7.04015
7.25803
7.48630
7.49055
7.51379
7.53903
7.57096
7.57527
7.59169
7.59945
7.60858
7.62395
7.93384
7.93884
7.96182
9.35707
2.039
2.000
1.960
3.023
1.963
0.908
ppm

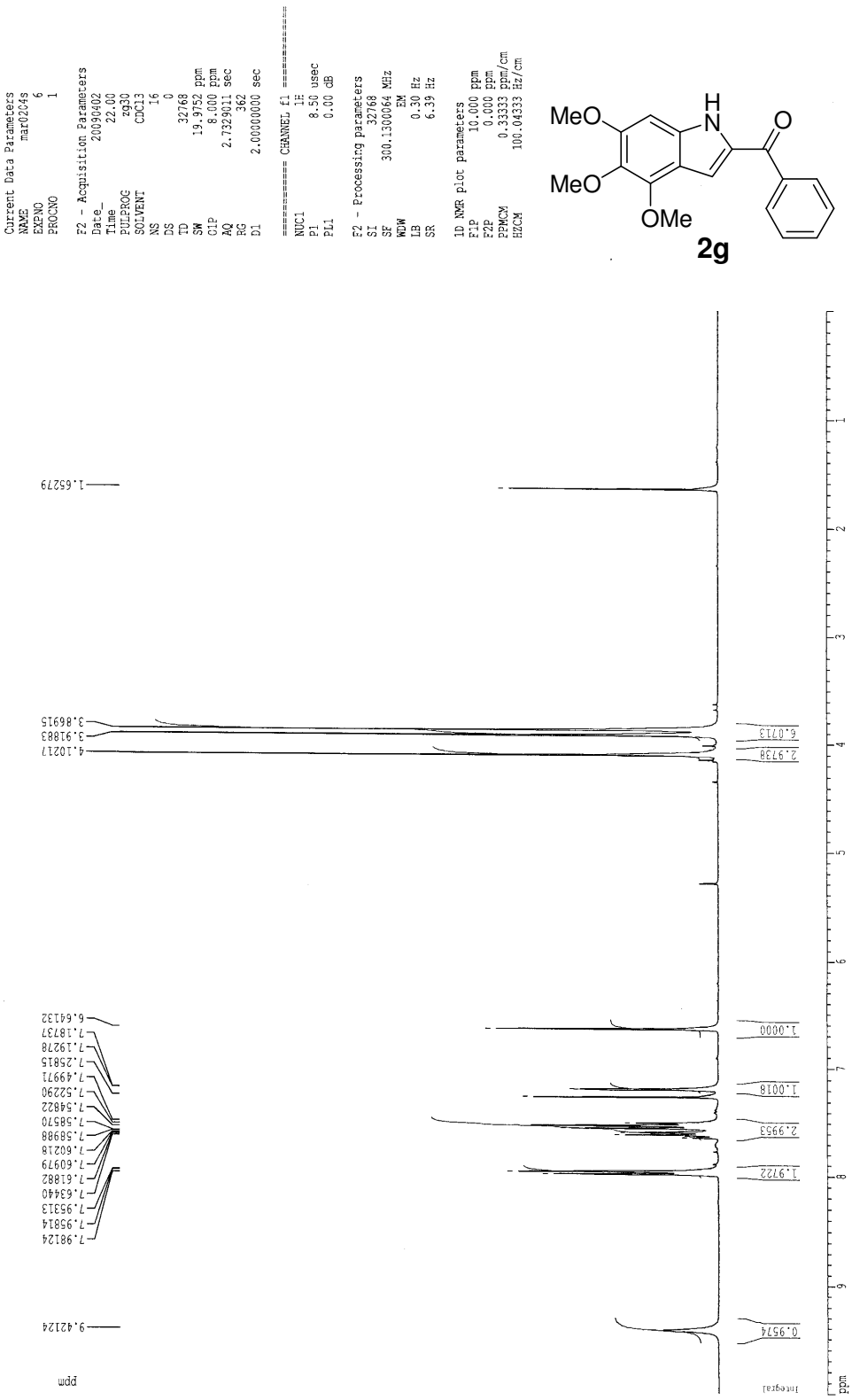

Current Data Parameters
NAME mar3103s
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090401
Time 8.16
PULPROG zgpg30
SOLVENT CDCl3
NS 10240
DS 4
TD 32768
SW 240.0225 ppm
O1P 110.000 ppm
AQ 0.9044468 sec
RG 8192
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 13.00 usec
PL1 -6.00 dB
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 9.00 dB
PL12 9.00 dB
PL13 9.00 dB
F2 - Processing parameters
SI 32768
SF 75.4677498 MHz
WDW EM
LB 2.00 Hz
SR 30.78 Hz
1D NMR plot parameters
F1P 191.348 ppm
F2P 89.298 ppm
PPMCM 3.40166 ppm/cm
HZCM 256.71542 Hz/cm
H
N
O
O
O
2d
91.983
100.004
101.193
113.494
122.113
128.066
128.410
129.098
132.041
133.702
134.255
138.272
144.425
148.970
186.070
ppm
100
110
120
130
140
150
160
170
180
ppm

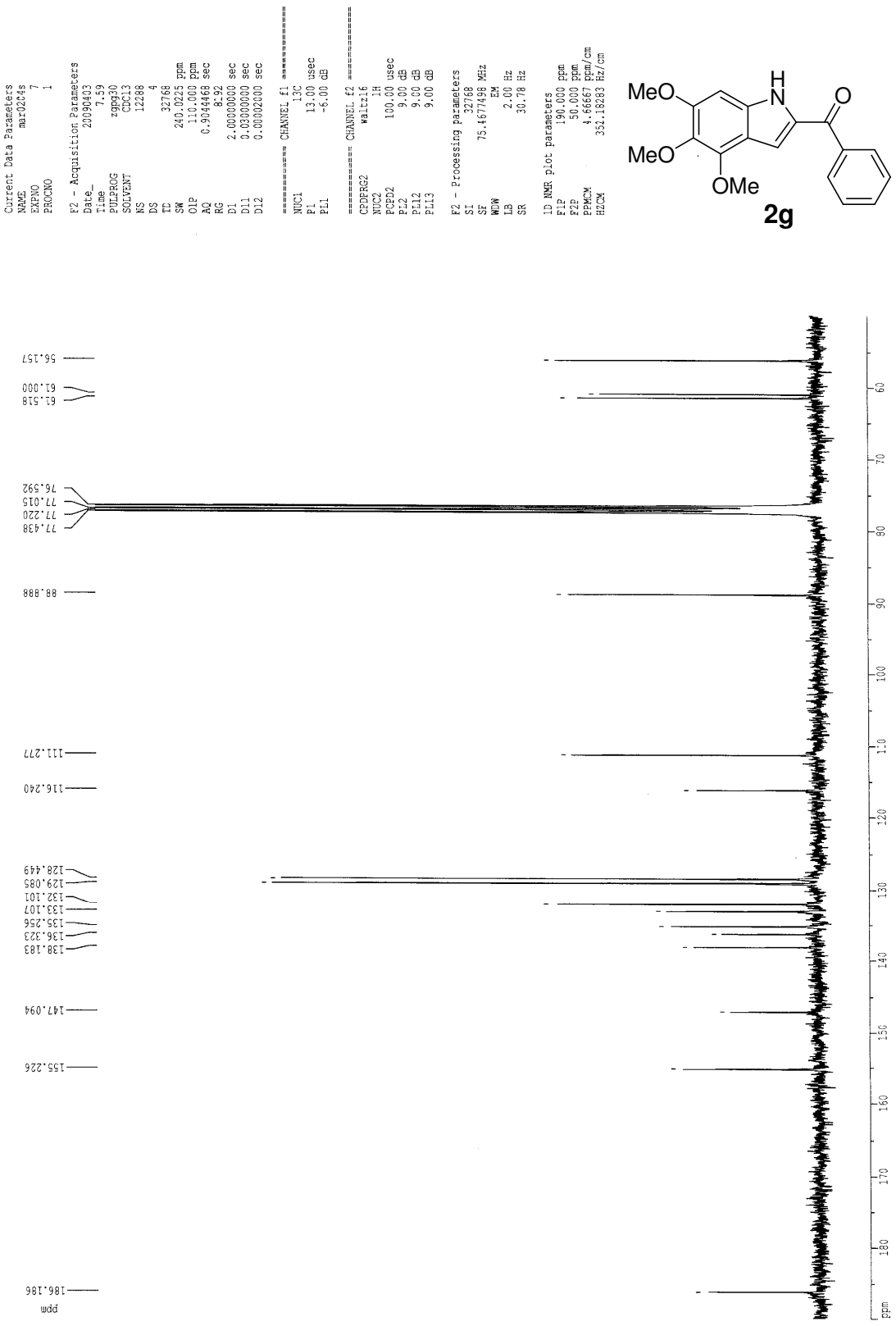

Current Data Parameters
NAME mar1304s
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090413
Time 21.14
PULPROG zg30
SOLVENT CDCl3
NS 16
DS 0
TD 32768
SW 19.9752 ppm
O1P 8.000 ppm
AQ 2.7329011 sec
RG 574.7
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.50 usec
PL1 0.00 dB
F2 - Processing parameters
SI 32768
SF 300.1300064 MHz
WDW EM
LB 0.30 Hz
SR 6.39 Hz
1D NMR plot parameters
F1P 10.000 ppm
F2P 3.000 ppm
PPMCM 0.23333 ppm/cm
HZCM 70.03033 Hz/cm
MeO
MeO
H
N
O
2e
3.91981
3.93277
3.94306
3.95561
5.29410
6.90036
7.04300
7.05595
7.05865
7.06315
7.06584
7.25822
7.48720
7.49176
7.49710
7.51506
7.52017
7.53568
7.54001
7.57249
7.57737
7.58216
7.59357
7.60179
7.95178
7.95687
7.96386
7.97462
7.97975
7.98401
9.43352
5.9943
1.0000
1.9708
2.9926
1.9773
0.9502
Integral
ppm

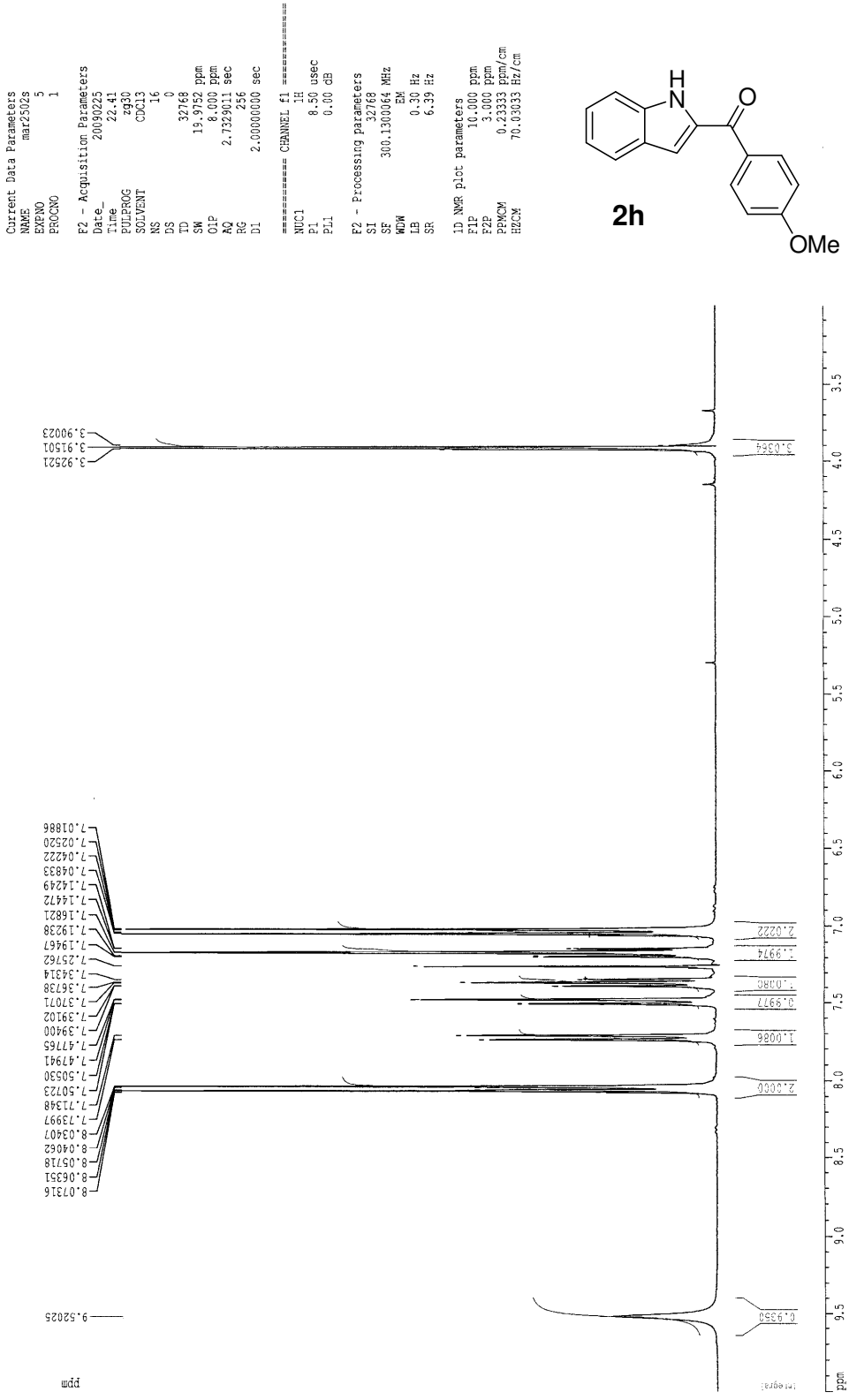

Current Data Parameters
NAME mar304s
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090414
Time_ 7.14
PULPROG zgpg30
SOLVENT CDCl3
NS 12288
DS 4
TD 32768
SW 240.0225 ppm
O1P 110.000 ppm
AQ 0.9044468 sec
RG 4096
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 13.00 usec
PL1 -6.00 dB
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 9.00 dB
PL12 9.00 dB
PL13 9.00 dB
F2 - Processing parameters
SI 32768
SF 75.4677498 MHz
WDW EM
LB 2.00 Hz
SR 30.78 Hz
1D NMR plot parameters
F1P 200.000 ppm
F2P 50.000 ppm
PPMCM 5.00000 ppm/cm
HZCM 377.33875 Hz/cm
MeO
MeO
H
N
O
2e
56.069
56.135
76.591
77.015
77.220
77.438
93.694
102.696
113.150
120.862
128.373
129.097
131.988
133.388
133.448
138.396
146.461
151.165
186.100
ppm

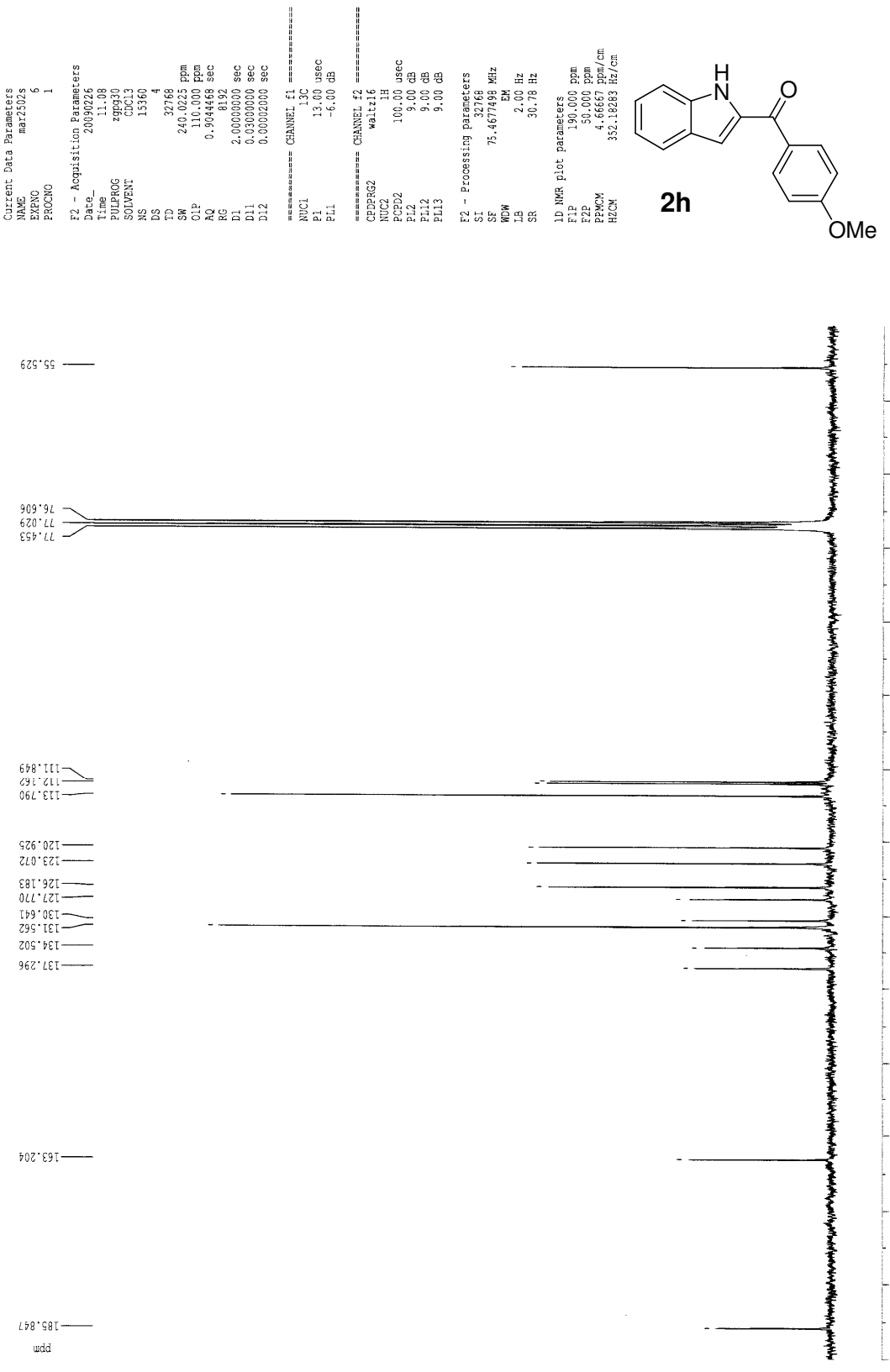

Current Data Parameters
NAME mar1105s
EXPNO 6
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090511
Time 21.47
PULPROG zg30
SOLVENT CDCl3
NS 16
DS 0
TD 32768
SW 19.9752 ppm
O1P 8.000 ppm
AQ 2.7329011 sec
RG 406.4
D1 2.00000000 sec
========== CHANNEL f1 ==========
NUC1 1H
P1 8.50 usec
PL1 0.00 dB
F2 - Processing parameters
SI 32768
SF 300.1300064 MHz
WDW EM
LB 0.30 Hz
SR 6.39 Hz
1D NMR plot parameters
F1P 9.500 ppm
F2P 5.000 ppm
PPMCM 0.15000 ppm/cm
HZCM 45.01950 Hz/cm
BnO
H
N
O
2f
5.10779
5.29693
9.32394
7.99868
7.99482
7.98983
7.97857
7.97176
7.96656
7.62138
7.59706
7.55420
7.53985
7.52888
7.48460
7.46073
7.42128
7.41484
7.41226
7.39817
7.39250
7.38870
7.38314
7.37962
7.37375
7.35255
7.26834
7.16234
7.13550
7.07588
7.07331
7.06901
7.06635
2.0341
1.0385
2.0284
4.0926
4.0369
1.0162
2.0000
0.9363
ppm

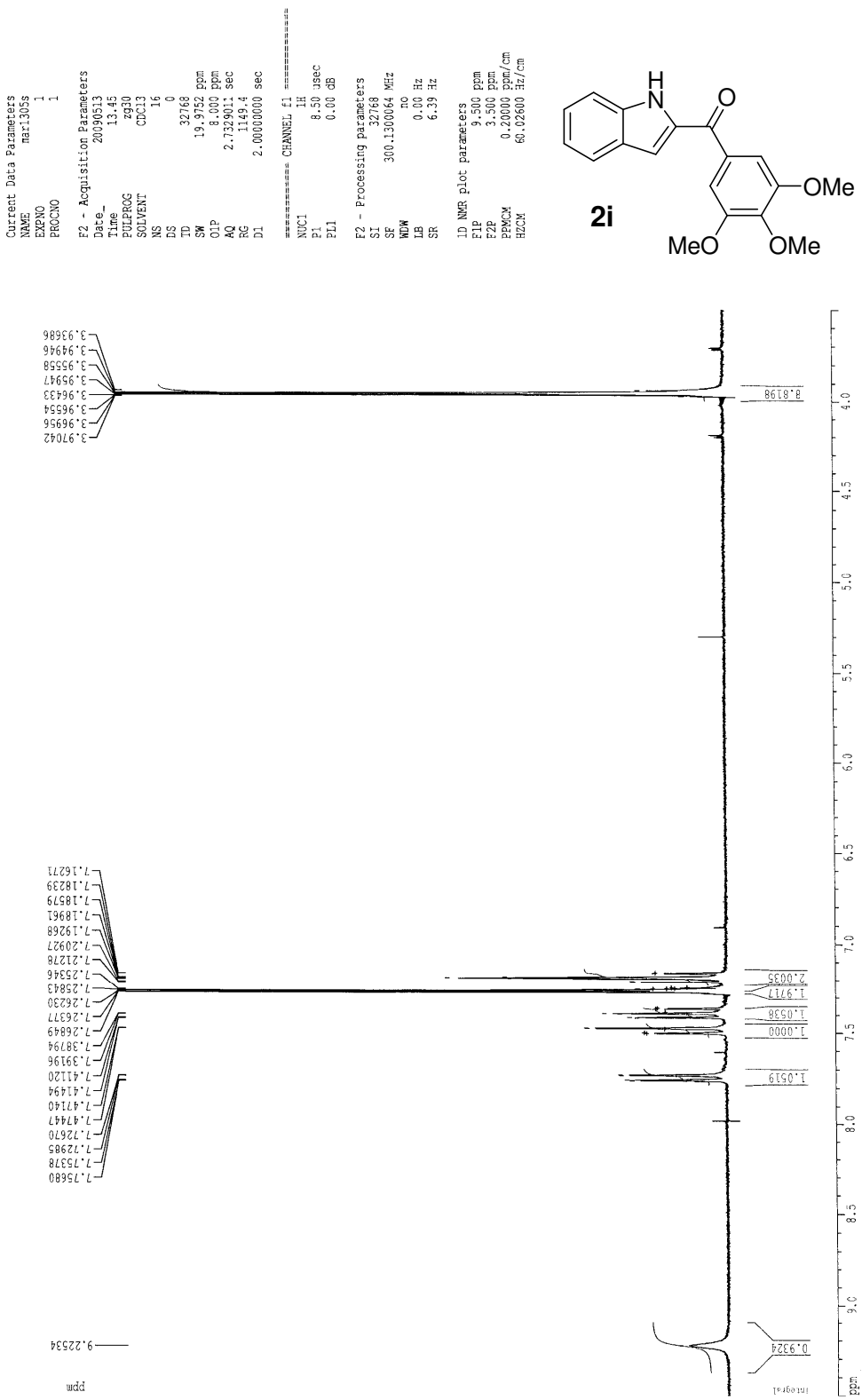

Current Data Parameters
NAME mar110ss
EXPNO 8
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090512
Time 6.24
PULPROG zgpg30
SOLVENT CDCl3
NS 10240
DS 4
TD 32768
SW 240.0225 ppm
O1P 110.000 ppm
AQ 0.9044468 sec
RG 8192
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 13.00 usec
PL1 -6.00 dB
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 9.00 dB
PL12 9.00 dB
PL13 9.00 dB
F2 - Processing parameters
SI 32768
SF 75.4677498 MHz
WDW EM
LB 2.00 Hz
SR 30.78 Hz
1D NMR plot parameters
F1P 190.000 ppm
F2P 60.000 ppm
PPMCM 4.33333 ppm/cm
HZCM 327.02692 Hz/cm
BnO
H
N
O
2f
70.650
76.589
77.013
77.218
77.436
104.495
112.339
113.144
118.962
127.518
127.926
128.036
128.441
128.586
129.201
132.292
133.167
134.828
137.211
138.049
153.957
186.959
ppm

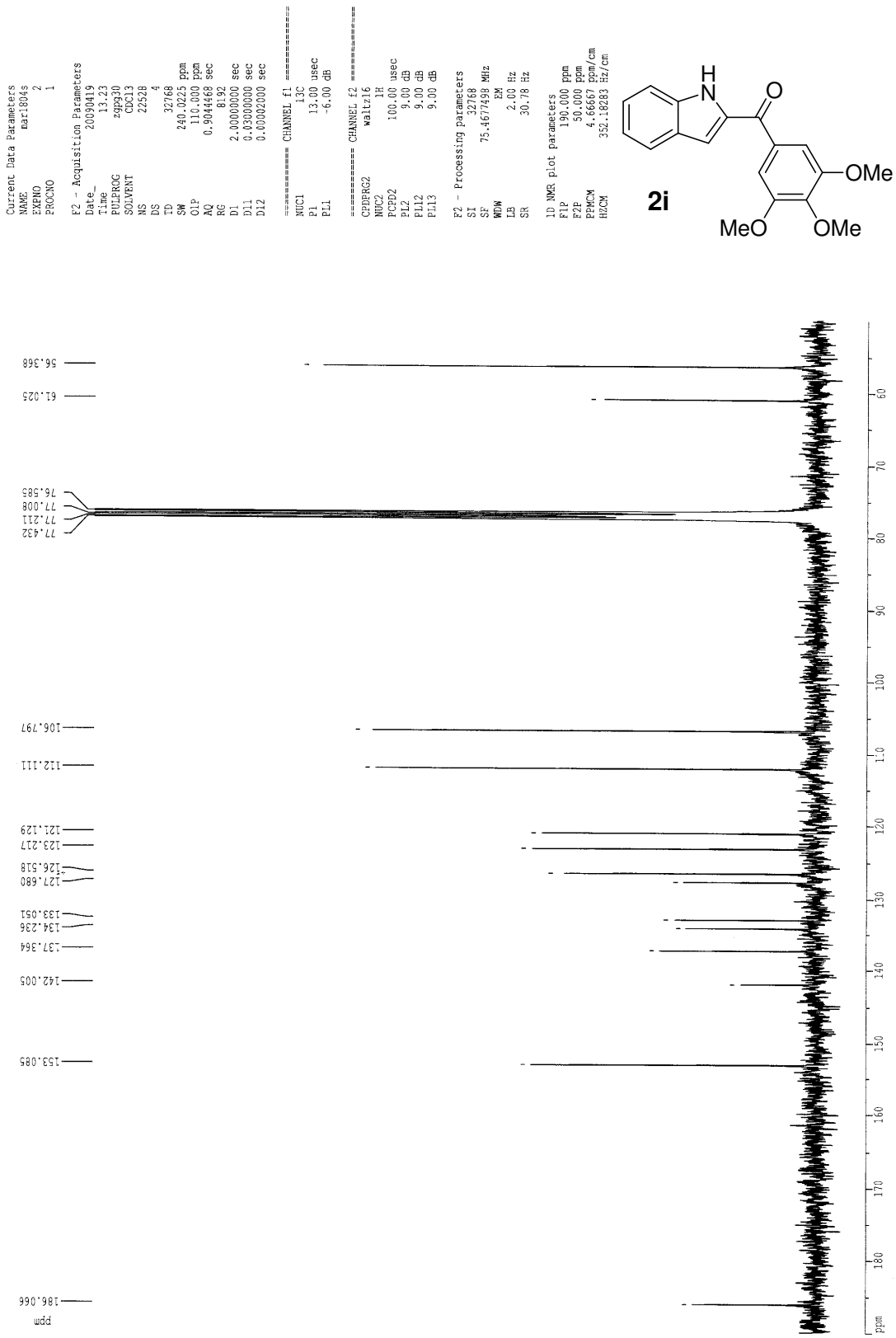

Current Data Parameters
NAME mar0204s
EXPNO 6
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090402
Time 22.00
PULPROG zg30
SOLVENT CDCl3
NS 16
DS 0
TD 32768
SW 19.9752 ppm
O1P 8.000 ppm
AQ 2.7329011 sec
RG 362
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.50 usec
PL1 0.00 dB
F2 - Processing parameters
SI 32768
SF 300.1300064 MHz
WDW EM
LB 0.30 Hz
SR 6.39 Hz
1D NMR plot parameters
F1P 10.000 ppm
F2P 0.000 ppm
PPMCM 0.33333 ppm/cm
HZCM 100.04333 Hz/cm
MeO
MeO
OMe
H
N
O
2g
1.65279
3.86915
3.91883
4.10217
6.64132
7.18737
7.19278
7.25815
7.49971
7.52290
7.54822
7.58570
7.58988
7.60218
7.60979
7.61882
7.63440
7.95313
7.95814
7.98124
9.42124
6.0713
2.9738
1.0000
1.0018
2.9953
1.9722
0.9574
Integral
ppm

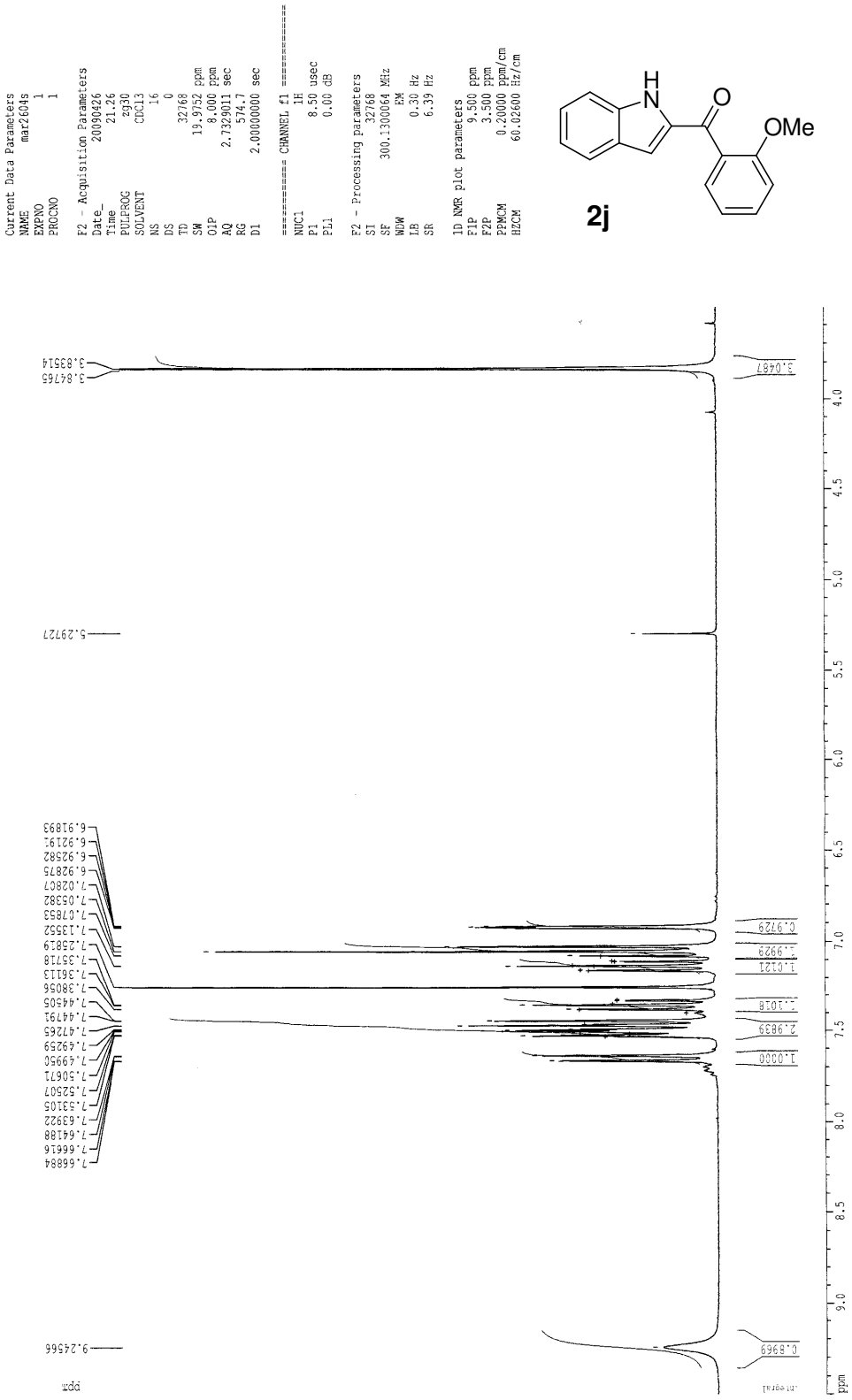

Current Data Parameters
NAME mar0204s
EXPNO 7
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090403
Time 7.59
PULPROG zgpg30
SOLVENT CDCl3
NS 12288
DS 4
TD 32768
SW 240.0225 ppm
O1P 110.000 ppm
AQ 0.9044468 sec
RG 8192
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 13.00 usec
PL1 -6.00 dB
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 9.00 dB
PL12 9.00 dB
PL13 9.00 dB
F2 - Processing parameters
SI 32768
SF 75.4677498 MHz
WDW EM
LB 2.00 Hz
SR 30.78 Hz
1D NMR plot parameters
F1P 190.000 ppm
F2P 50.000 ppm
PPMCM 4.66667 ppm/cm
HZCM 352.18293 Hz/cm
MeO
MeO
OMe
H
N
O
2g
56.157
61.000
61.518
76.592
77.015
77.220
77.438
88.888
111.277
116.240
128.449
129.085
132.101
133.107
135.256
136.323
138.183
147.094
155.226
186.186
ppm

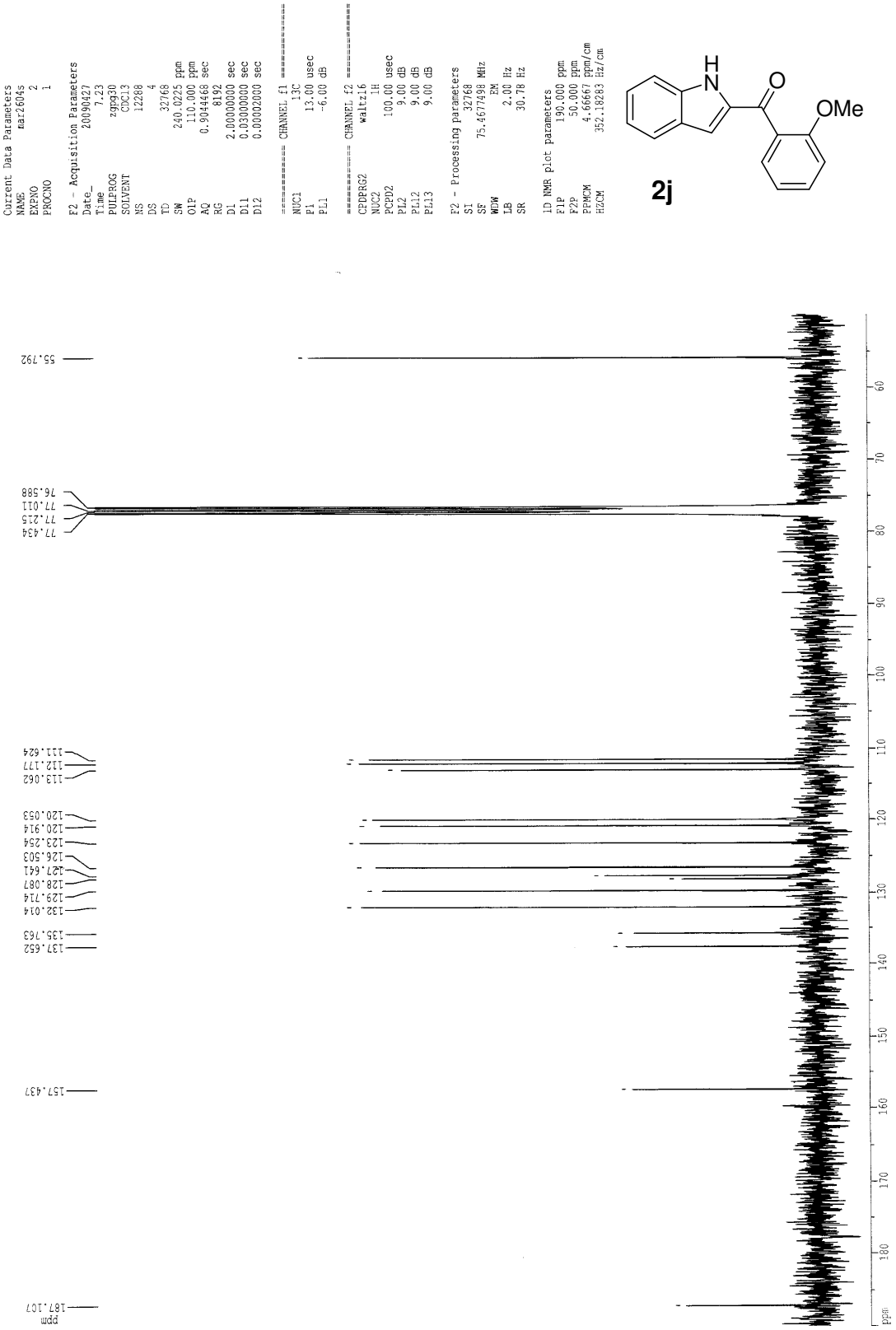

Current Data Parameters
NAME mar2502s
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090225
Time 22.41
PULPROG zg30
SOLVENT CDCl3
NS 16
DS 0
TD 32768
SW 19.9752 ppm
O1P 8.000 ppm
AQ 2.7329011 sec
RG 256
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.50 usec
PL1 0.00 dB
F2 - Processing parameters
SI 32768
SF 300.1300064 MHz
WDW EM
LB 0.30 Hz
SR 6.39 Hz
1D NMR plot parameters
PIP 10.000 ppm
F2P 3.000 ppm
PPMCM 0.23333 ppm/cm
HZCM 70.03033 Hz/cm
2h
OMe
3.92521
3.91501
3.90023
7.01886
7.02520
7.04222
7.04833
7.14249
7.14472
7.16821
7.19238
7.19467
7.25762
7.34314
7.36738
7.37071
7.39102
7.39400
7.47765
7.47941
7.50530
7.50723
7.71348
7.73997
8.03407
8.04062
8.05718
8.06351
8.07316
9.52025
5.0364
2.0222
1.9974
1.0180
0.9977
1.0086
2.0000
0.9350
ppm

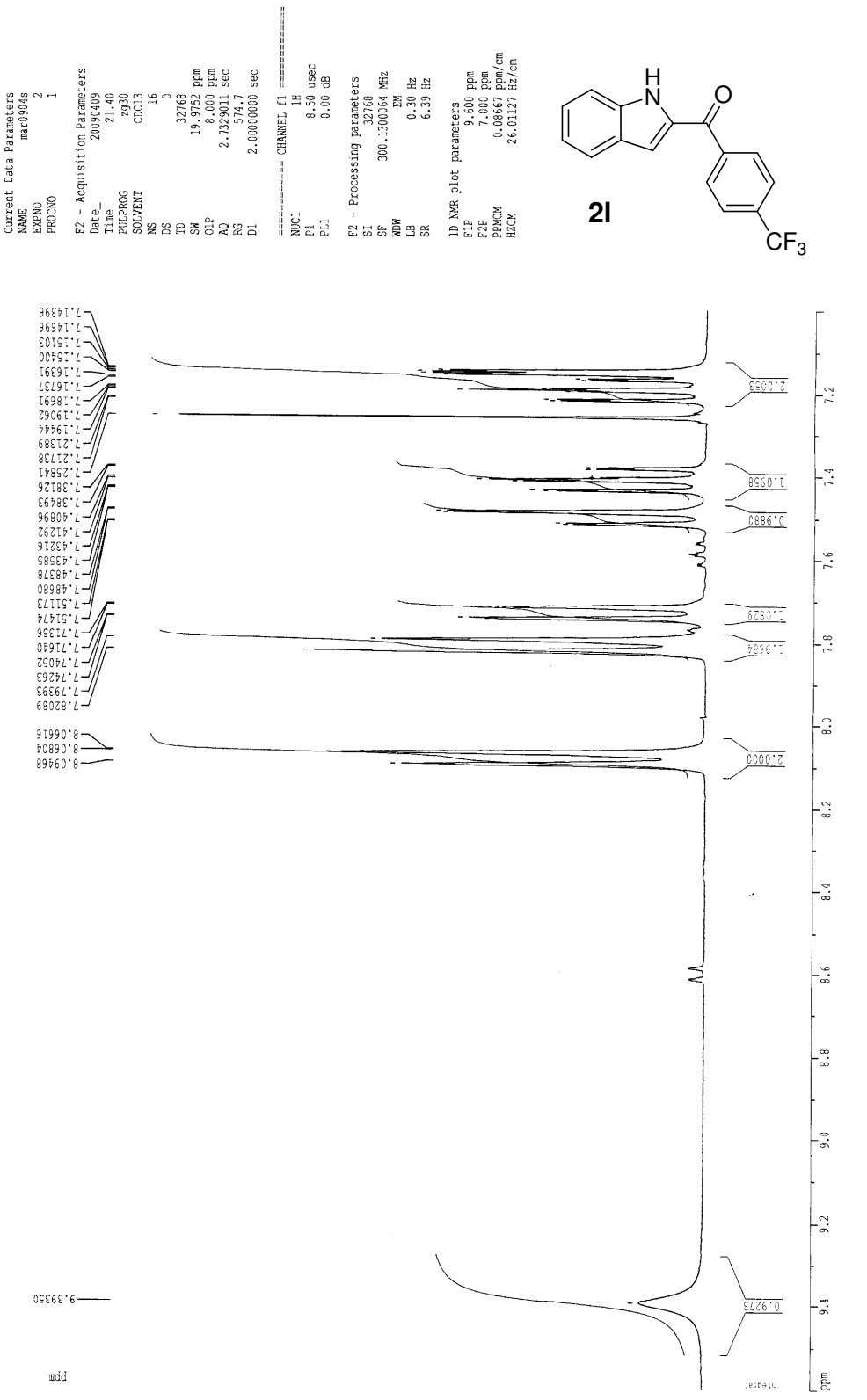

Current Data Parameters
NAME mar2502s
EXPNO 6
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090226
Time 11.08
PULPROG zgpg30
SOLVENT CDCl3
NS 15360
DS 4
TD 32768
SW 240.0225 ppm
O1P 110.000 ppm
AQ 0.9044468 sec
RG 8192
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 13.00 usec
PL1 -6.00 dB
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 9.00 dB
PL12 9.00 dB
PL13 9.00 dB
F2 - Processing parameters
SI 32768
SF 75.4677498 MHz
WDW EM
LB 2.00 Hz
SR 30.78 Hz
1D NMR plot parameters
F1P 190.000 ppm
F2P 50.000 ppm
PPMCM 4.66667 ppm/cm
HZCM 352.18283 Hz/cm
2h
H
N
O
OMe
55.529
76.606
77.029
77.453
111.849
117.167
113.790
120.925
123.072
126.183
127.770
130.641
131.562
134.502
137.296
163.204
185.847
ppm

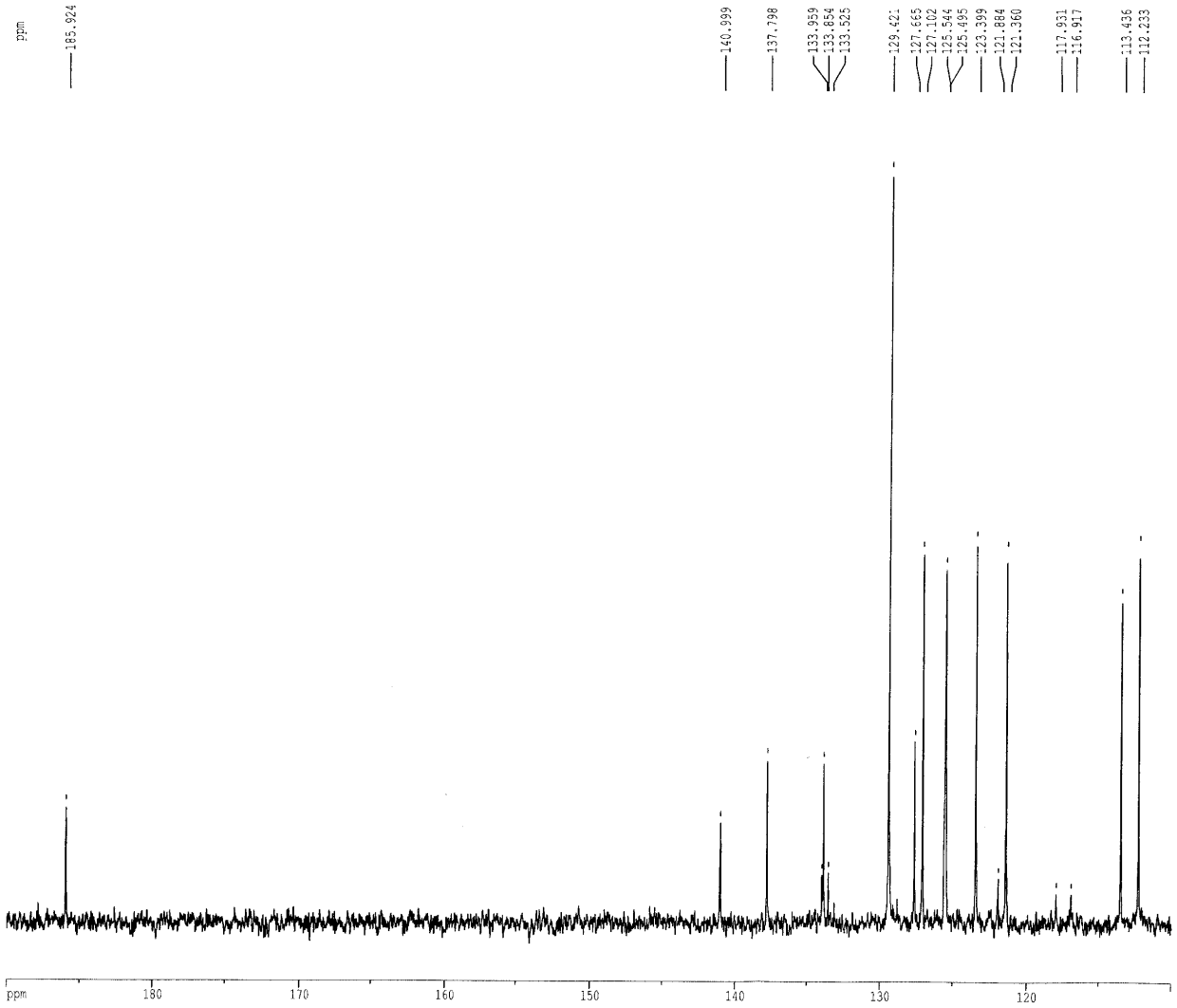

Current Data Parameters
NAME mar1305s
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090513
Time 13.45
PULPROG zg30
SOLVENT CDCl3
NS 16
DS 0
TD 32768
SW 19.9752 ppm
O1P 8.000 ppm
AQ 2.7329011 sec
RG 1149.4
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.50 usec
PL1 0.00 dB
F2 - Processing parameters
SI 32768
SF 300.1300064 MHz
WDW no
LB 0.00 Hz
SR 6.39 Hz
1D NMR plot parameters
F1P 9.500 ppm
F2P 3.500 ppm
PPMCM 0.20000 ppm/cm
HZCM 60.02600 Hz/cm
2i
H
N
O
OMe
MeO
OMe
3.93686
3.94946
3.95558
3.95947
3.96433
3.96554
3.96956
3.97042
8.8198
7.16271
7.18239
7.18579
7.18961
7.19268
7.20927
7.21278
7.25346
7.25843
7.26230
7.26377
7.26849
7.38794
7.39196
7.41120
7.41494
7.47140
7.47447
7.72670
7.72985
7.75378
7.75680
9.22534
2.0035
1.9717
1.0538
1.0000
1.0519
0.9324
integral
ppm
ppm


Current Data Parameters
NAME kar18044s
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090419
Time 13.23
PULPROG zgpg30
SOLVENT CDCl3
NS 22528
DS 4
TD 32768
SW 240.0225 ppm
O1P 110.000 ppm
AQ 0.9044468 sec
RG 8192
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 13.00 usec
PL1 -6.00 dB
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 9.00 dB
PL12 9.00 dB
PL13 9.00 dB
F2 - Processing parameters
SI 32768
SF 75.4677498 MHz
WDW EM
LB 2.00 Hz
SR 30.78 Hz
1D NMR plot parameters
F1P 190.000 ppm
F2P 50.000 ppm
PPMCM 4.66667 ppm/cm
HZCM 352.18283 Hz/cm
2i
H
N
O
OMe
MeO
OMe
56.368
61.025
76.585
77.008
77.211
77.432
106.797
112.111
121.129
123.217
126.518
127.680
133.051
134.236
137.364
142.005
153.085
186.066
ppm

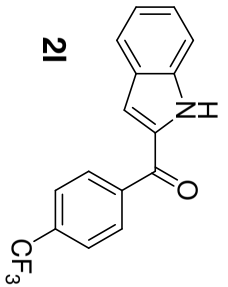

Current Data Parameters
NAME mar2604s
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090426
Time 21.26
PULPROG zg30
SOLVENT CDCl3
NS 16
DS 0
TD 32768
SW 19.9752 ppm
O1P 8.000 ppm
AQ 2.7329011 sec
RG 574.7
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.50 usec
PL1 0.00 dB
F2 - Processing parameters
SI 32768
SF 300.1300064 MHz
WDW EM
LB 0.30 Hz
SR 6.39 Hz
1D NMR plot parameters
F1P 9.500 ppm
F2P 3.500 ppm
PPMCM 0.20000 ppm/cm
HZCM 60.02600 Hz/cm
H
N
O
OMe
2j
3.83514
3.84765
5.04687
5.29727
6.91893
6.92191
6.92582
6.92875
7.02807
7.05382
7.07853
7.13562
7.25829
7.35718
7.36113
7.38056
7.44505
7.44791
7.47265
7.49259
7.49950
7.50671
7.52507
7.53105
7.63922
7.64188
7.66616
7.66884
0.9729
1.9929
1.0121
2.1018
2.9839
1.0000
9.24566
0.8969
ppm
integral

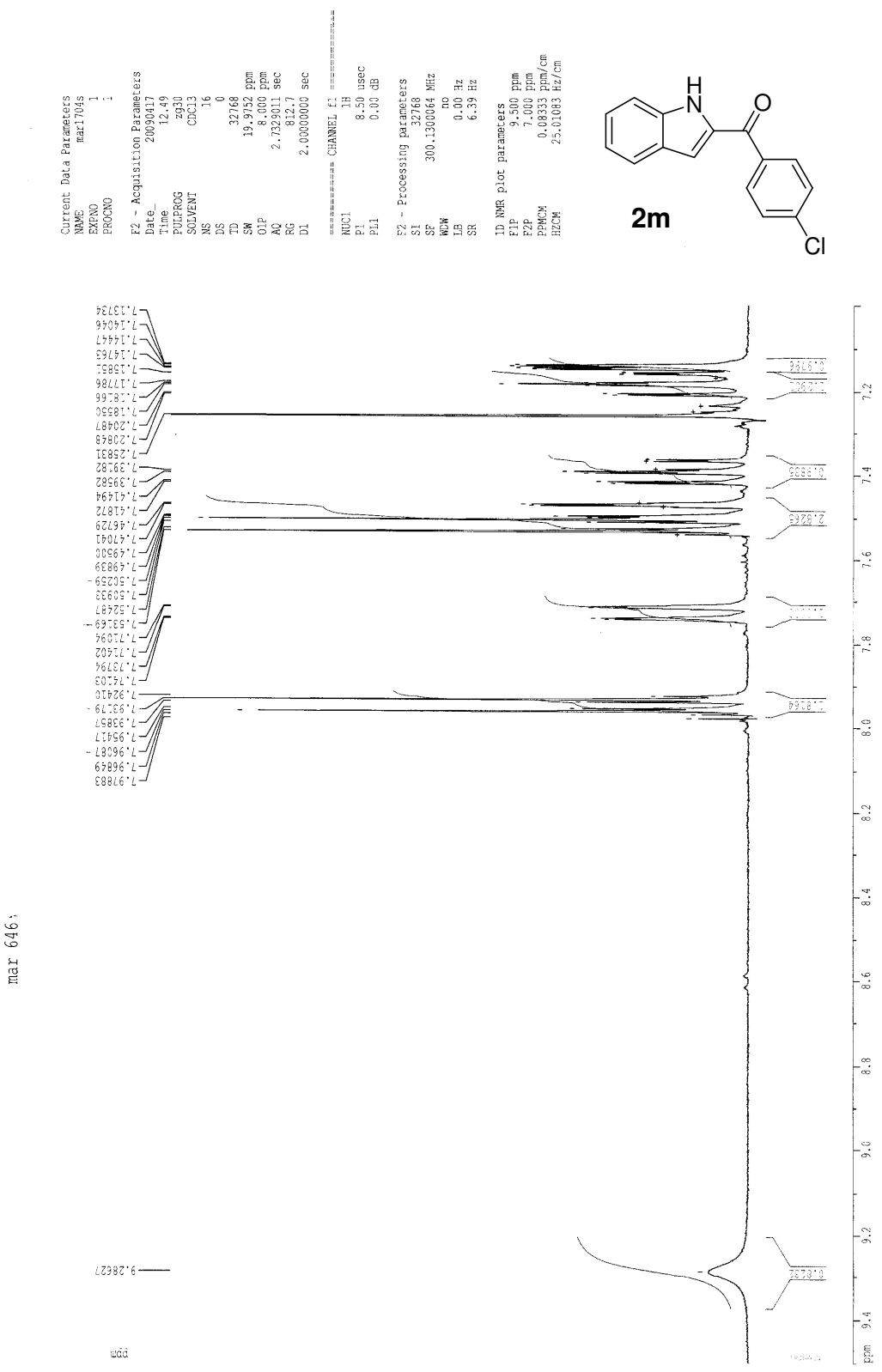

Current Data Parameters
NAME maz2604s
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090427
Time 7.23
PULPROG zgpg30
SOLVENT CDCl3
NS 12288
DS 4
TD 32768
SW 240.0225 ppm
O1P 110.000 ppm
AQ 0.9044468 sec
RG 8192
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 13.00 usec
PL1 -6.00 dB
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 9.00 dB
PL12 9.00 dB
PL13 9.00 dB
F2 - Processing parameters
SI 32768
SF 75.4677498 MHz
WDW EM
LB 2.00 Hz
SR 30.78 Hz
1D NMR plot parameters
F1P 190.000 ppm
F2P 50.000 ppm
PPMCM 4.66667 ppm/cm
HZCM 352.18283 Hz/cm
H
N
O
OMe
2j
55.792
76.588
77.011
77.215
77.434
111.624
112.177
113.062
120.053
120.914
123.264
126.503
127.641
128.087
129.714
132.014
135.763
137.652
157.437
187.107
ppm
60
70
80
90
100
110
120
130
140
150
160
170
180
ppm

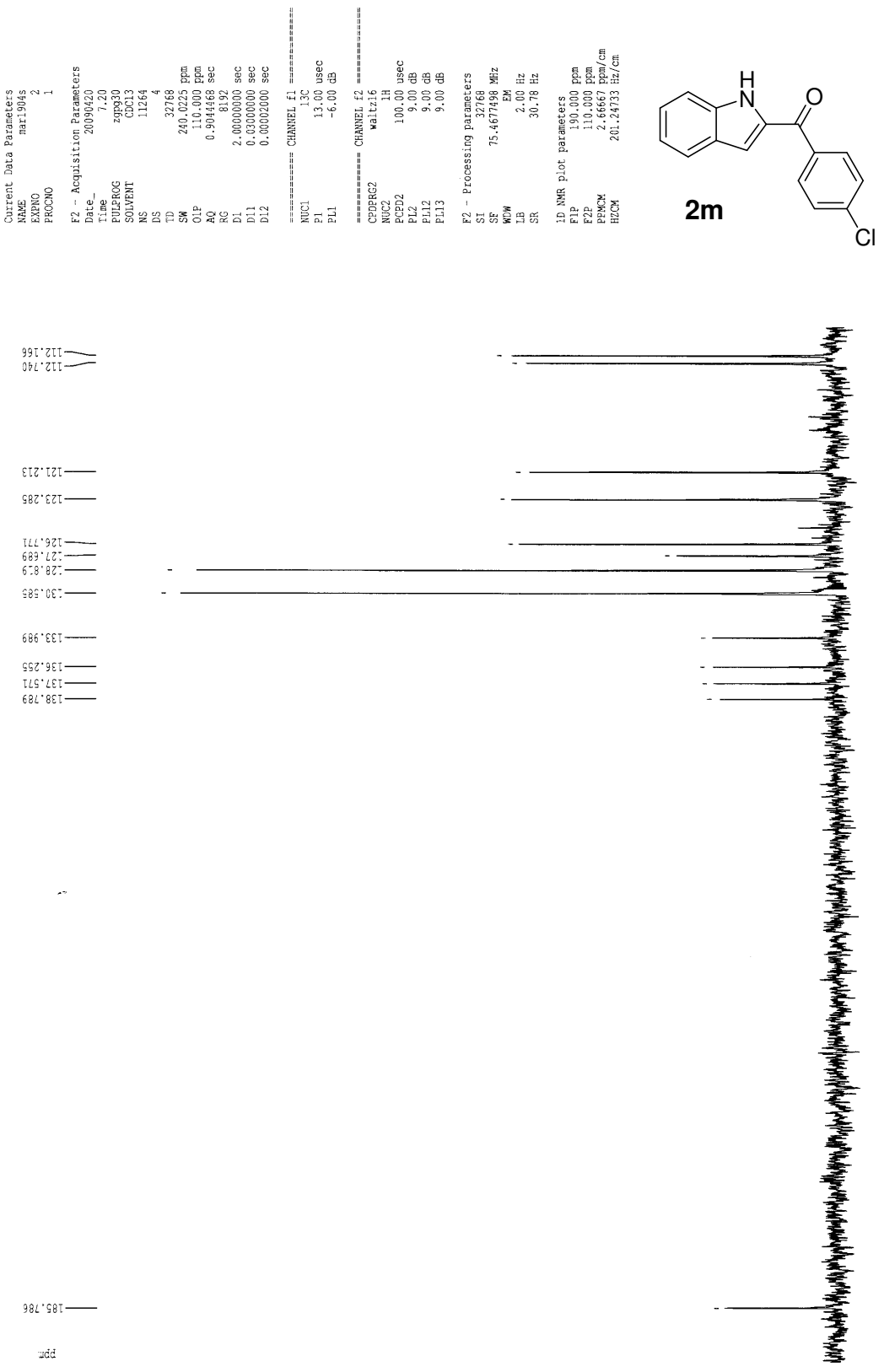

Current Data Parameters
NAME mar0904s
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090409
Time_ 21.40
PULPROG zg30
SOLVENT CDCl3
NS 16
DS 0
TD 32768
SW 19.9752 ppm
O1P 8.000 ppm
AQ 2.7329011 sec
RG 574.7
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.50 usec
PL1 0.00 dB
F2 - Processing parameters
SI 32768
SF 300.1300064 MHz
WDW EM
LB 0.30 Hz
SR 6.39 Hz
1D NMR plot parameters
F1P 9.600 ppm
F2P 7.000 ppm
PPMCM 0.08667 ppm/cm
HZCM 26.01127 Hz/cm
2I
H
N
O
CF3
7.14396
7.14696
7.15103
7.15400
7.16391
7.16737
7.18691
7.19062
7.19444
7.21389
7.21738
7.25641
7.38126
7.38493
7.40896
7.41292
7.43216
7.43585
7.48378
7.48680
7.51173
7.51474
7.71356
7.71640
7.74052
7.74263
7.79393
7.82089
8.06616
8.06804
8.09468
9.39350
2.0033
1.0958
0.9883
1.0829
1.9684
2.0000
0.9213
ppm

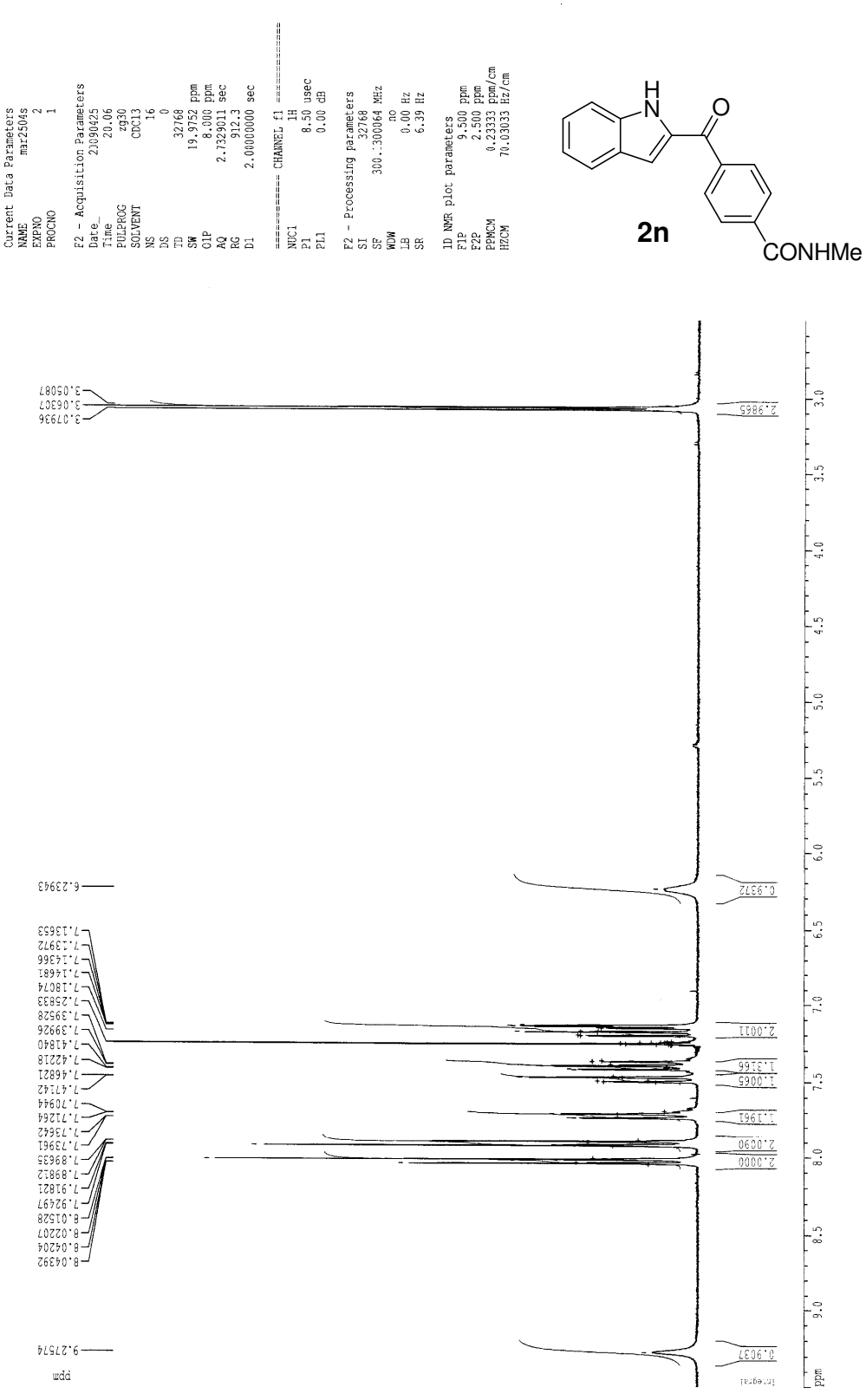


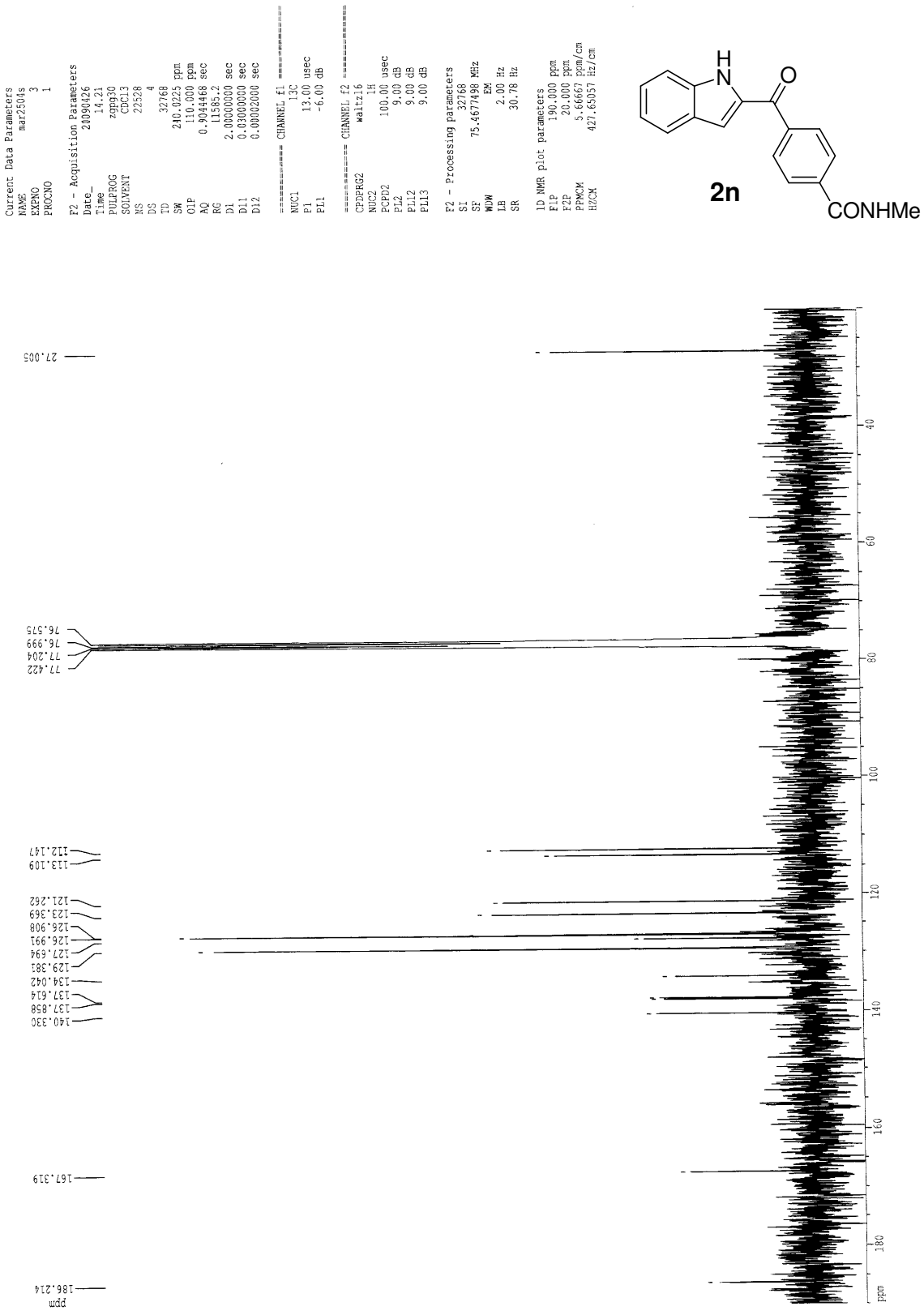

```
Current Data Parameters
NAME                  mar0904s
EXPNO                        3
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20090410
Time                      5.59
PULPROG                zgpg30
SOLVENT                ------
NS
DS
TD
SW                             ppm
O1P                            ppm
AQ                             sec
RG
D1                             sec
D11                            sec
D12                            sec

============ CHANNEL f1 =============
NUC1                        13C
P1                        13.00 usec
PL1                       -6.00 dB

============ CHANNEL f2 =============
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                    100.00 usec
PL2                        9.00 dB
PL12                       9.00 dB
PL13                       9.00 dB

F2 - Processing parameters
SI                          32768
SF                  75.4677498 MHz
WDW                            EM
LB                         2.00 Hz
SR                        30.78 Hz

1D NMR plot parameters
F1P                     190.000 ppm
F2P                     110.000 ppm
PPMCM                   2.66667 ppm/cm
HZCM                  201.24733 Hz/cm
```

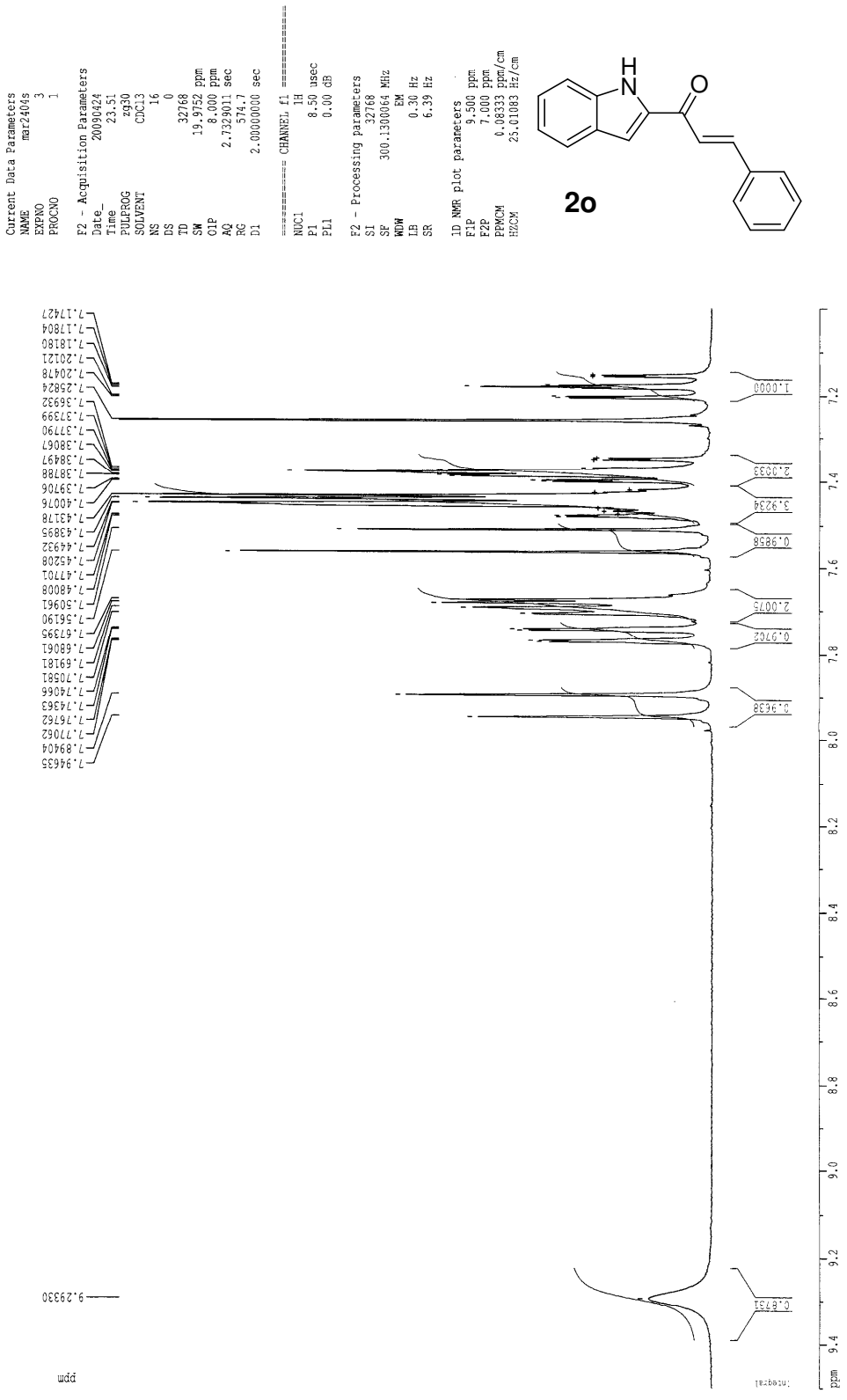


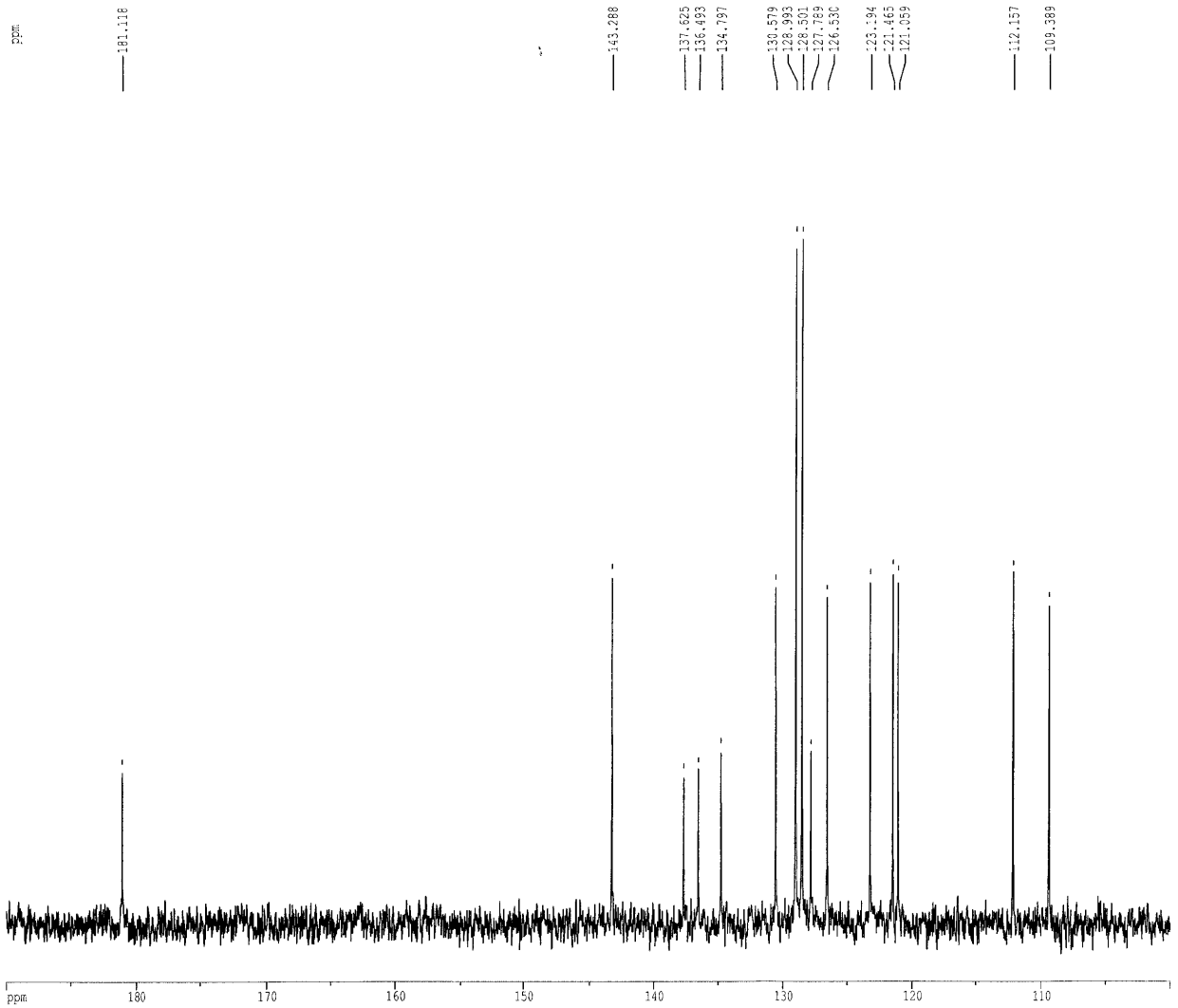

Current Data Parameters
NAME mar1704s
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090417
Time 12.49
PULPROG zg30
SOLVENT CDCl3
NS 16
DS 0
TD 32768
SW 19.9752 ppm
O1P 8.000 ppm
AQ 2.7329011 sec
RG 812.7
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.50 usec
PL1 0.00 dB
F2 - Processing parameters
SI 32768
SF 300.1300064 MHz
WDW no
LB 0.00 Hz
SR 6.39 Hz
1D NMR plot parameters
F1P 9.500 ppm
F2P 7.000 ppm
PPMCM 0.08333 ppm/cm
HZCM 25.01083 Hz/cm
2m
Cl
mar 646
ppm


Current Data Parameters
NAME mar1904s
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090420
Time 7.20
PULPROG zgpg30
SOLVENT CDCl3
NS 11264
DS 4
TD 32768
SW 240.0225 ppm
O1P 110.000 ppm
AQ 0.9044468 sec
RG 8192
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 13.00 usec
PL1 -6.00 dB
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 9.00 dB
PL12 9.00 dB
PL13 9.00 dB
F2 - Processing parameters
SI 32768
SF 75.4677498 MHz
WDW EM
LB 2.00 Hz
SR 30.78 Hz
1D NMR plot parameters
F1P 190.000 ppm
110.000 ppm
F2P 2.66667 ppm/cm
PPMCM 201.24733 Hz/cm
HZCM
H
N
O
Cl
2m
112.166
112.740
121.213
123.285
126.771
127.689
128.879
130.585
133.989
136.255
137.571
138.789
185.786
ppm
120
130
140
150
160
170
180

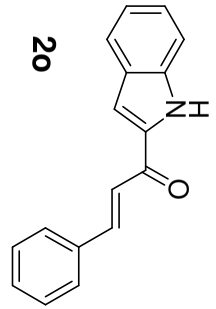

Current Data Parameters
NAME maz2504s
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090425
Time 20.06
PULPROG zg30
SOLVENT CDCl3
NS 16
DS 0
TD 32768
SW 19.9752 ppm
O1P 8.000 ppm
AQ 2.7329011 sec
RG 912.3
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.50 usec
PL1 0.00 dB
F2 - Processing parameters
SI 32768
SF 300.3000064 MHz
WDW no
LB 0.00 Hz
SR 6.39 Hz
1D NMR plot parameters
F1P 9.500 ppm
F2P 2.500 ppm
PPMCM 0.23333 ppm/cm
HZCM 70.03033 Hz/cm
2n
CONHMe
3.079936
3.063071
3.050871
6.239431
7.136531
7.139721
7.143661
7.146811
7.180741
7.258331
7.395281
7.399261
7.418401
7.422181
7.468211
7.471421
7.709441
7.712641
7.736421
7.739611
7.896351
7.898121
7.918211
7.924971
8.015281
8.022071
8.042041
8.043921
9.275741
2.9865
0.9372
2.0011
1.3166
1.0065
1.1961
2.0090
2.0000
0.9037
ppm

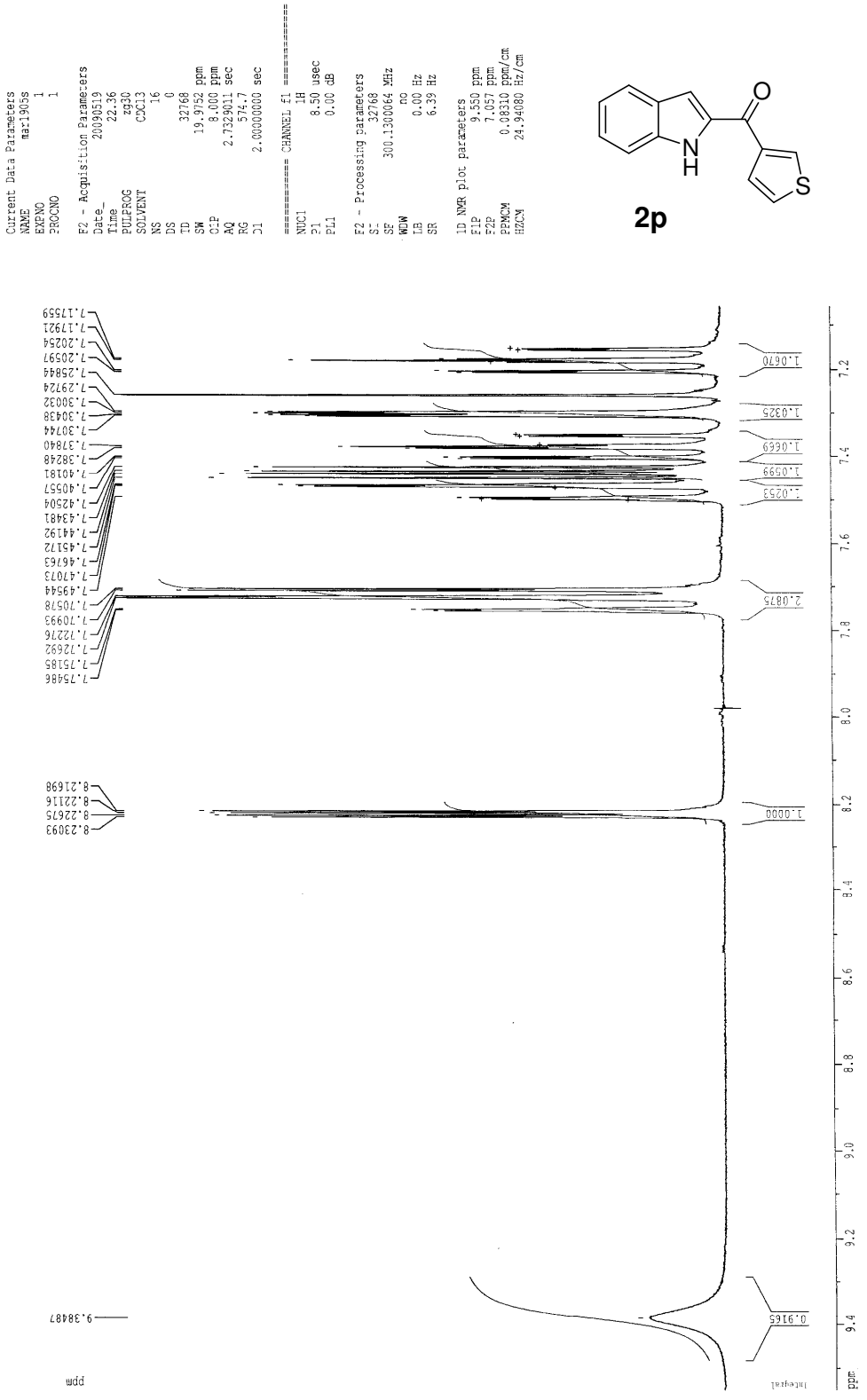

Current Data Parameters
NAME mar2504s
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090426
Time 14.21
PULPROG zgpg30
SOLVENT CDCl3
NS 22528
DS 4
TD 32768
SW 240.0225 ppm
O1P 110.000 ppm
AQ 0.9044468 sec
RG 11585.2
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 13.00 usec
PL1 -6.00 dB
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 9.00 dB
PL12 9.00 dB
PL13 9.00 dB
F2 - Processing parameters
SI 32768
SF 75.4677498 MHz
WDW EM
LB 2.00 Hz
SR 30.78 Hz
1D NMR plot parameters
F1P 190.000 ppm
F2P 20.000 ppm
PPMCM 5.66667 ppm/cm
HZCM 427.65057 Hz/cm
H
N
O
2n
CONHMe
27.005
76.575
76.999
77.204
77.422
112.147
113.109
121.262
123.369
126.908
126.991
127.694
129.381
134.042
137.614
137.858
140.330
167.319
186.214
ppm
40
60
80
100
120
140
160
180
ppm


Current Data Parameters
NAME mar2404s
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090424
Time 23.51
PULPROG zg30
SOLVENT CDCl3
NS 16
DS 0
TD 32768
SW 19.9752 ppm
O1P 8.000 ppm
AQ 2.7329011 sec
RG 574.7
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.50 usec
PL1 0.00 dB
F2 - Processing parameters
SI 32768
SF 300.1300064 MHz
WDW EM
LB 0.30 Hz
SR 6.39 Hz
1D NMR plot parameters
F1P 9.500 ppm
F2P 7.000 ppm
PPMCM 0.08333 ppm/cm
HZCM 25.01083 Hz/cm
2o
7.17427
7.17804
7.18180
7.20121
7.20478
7.25824
7.36932
7.37399
7.37790
7.38067
7.38497
7.38788
7.39706
7.40076
7.43178
7.43895
7.44932
7.45208
7.47701
7.48008
7.50961
7.56190
7.67395
7.68061
7.69181
7.70581
7.74066
7.74363
7.76762
7.77062
7.89404
7.94635
9.29330
1.0000
2.0033
3.9239
0.9858
2.0075
0.9703
3.9638
0.8751
integral
ppm
9.4
9.2
9.0
8.8
8.6
8.4
8.2
8.0
7.8
7.6
7.4
7.2
ppm




```
Current Data Parameters
NAME            mar2404s
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20090425
Time               12.21
PULPROG          zgpg30
SOLVENT            CDCl3
NS                 15360
DS                     4
TD                 32768
SW             240.0225 ppm
O1P              110.000 ppm
AQ             0.9044468 sec
RG                  8192
D1             2.00000000 sec
D11            0.03000000 sec
D12            0.00002000 sec

============ CHANNEL f1 ============
NUC1                13C
P1               13.00 usec
PL1               -6.00 dB

============ CHANNEL f2 ============
CPDPRG2          waltz16
NUC2                1H
PCPD2           100.00 usec
PL2               9.00 dB
PL12              9.00 dB
PL13              9.00 dB

F2 - Processing parameters
SI                 32768
SF          75.4677498 MHz
WDW                   EM
LB                 2.00 Hz
SR                30.78 Hz

1D NMR plot parameters
F1P              190.000 ppm
F2P              100.000 ppm
PPMCM            3.00000 ppm/cm
HZCM           226.40326 Hz/cm
```




Current Data Parameters
NAME mar1905s
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090519
Time 22.36
PULPROG zg30
SOLVENT CDCl3
NS 16
DS 0
TD 32768
SW 19.9752 ppm
O1P 8.000 ppm
AQ 2.7329011 sec
RG 574.7
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.50 usec
PL1 0.00 dB
F2 - Processing parameters
SI 32768
SF 300.1300064 MHz
WDW no
LB 0.00 Hz
SR 6.39 Hz
1D NMR plot parameters
F1P 9.550 ppm
F2P 7.057 ppm
PPMCM 0.08310 ppm/cm
HZCM 24.94080 Hz/cm
O
N
H
S
2p
7.17559
7.17921
7.20254
7.20597
7.25884
7.29724
7.30032
7.30438
7.30744
7.37840
7.38248
7.40181
7.40557
7.42504
7.43481
7.44192
7.45172
7.46763
7.47073
7.49544
7.70578
7.70993
7.72276
7.72692
7.75185
7.75486
8.21698
8.22116
8.22675
8.23093
9.38487
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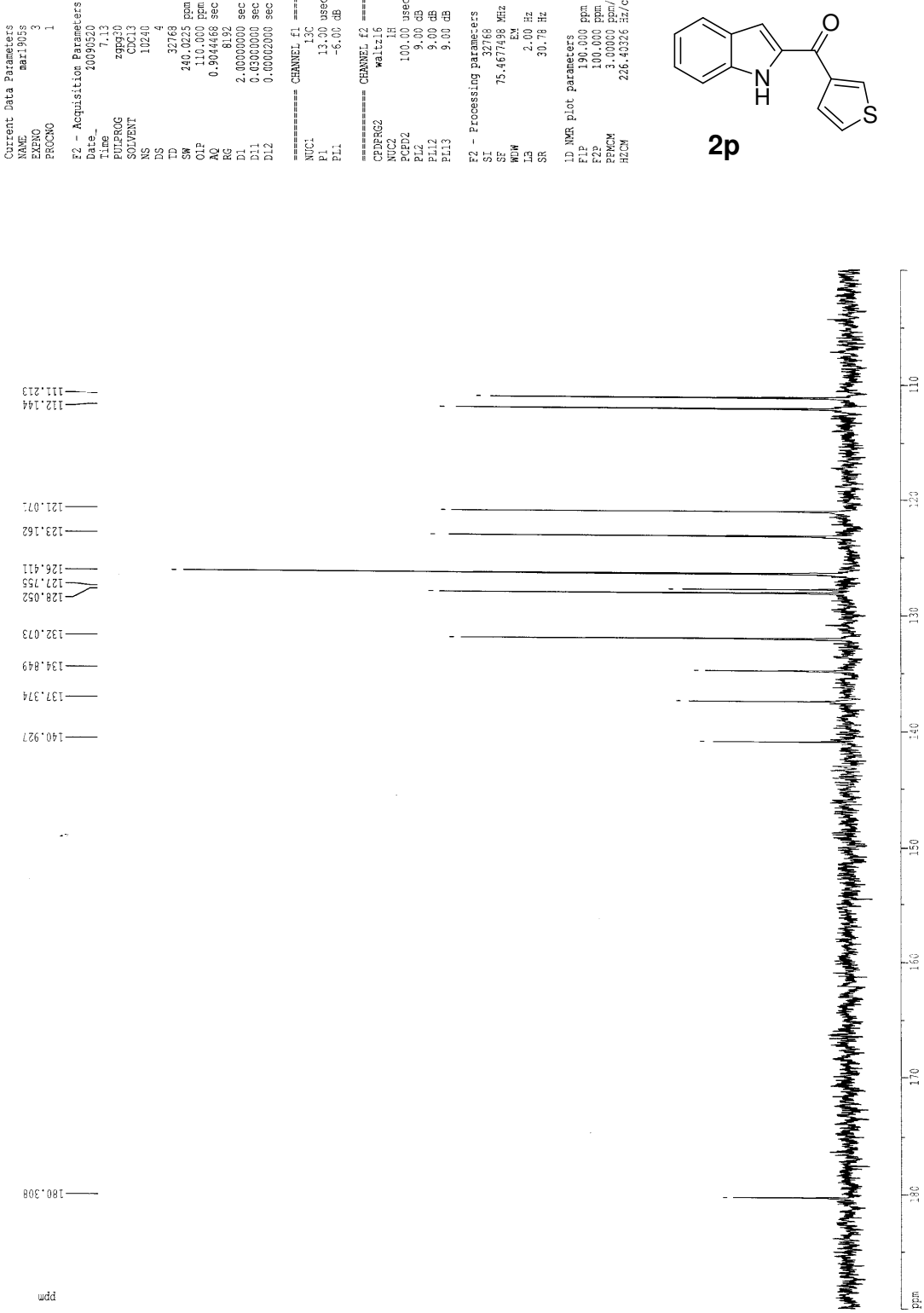
1.0325
1.0669
1.0599
1.0253
2.0875
1.0000
0.9165
Integral
ppm


Current Data Parameters
NAME mar1905s
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090520
Time_ 7.13
PULPROG zgpg30
SOLVENT CDCl3
NS 10240
DS 4
TD 32768
SW 240.0225 ppm
O1P 110.000 ppm
AQ 0.90444468 sec
RG 8192
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 13.00 usec
PL1 -6.00 dB
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 9.00 dB
PL12 9.00 dB
PL13 9.00 dB
F2 - Processing parameters
SI 32768
SF 75.4677498 MHz
WDW EM
LB 2.00 Hz
SR 30.78 Hz
1D NMR plot parameters
F1P 190.000 ppm
F2P 100.000 ppm
PPMCM 3.00000 ppm/cm
HZCM 226.40326 Hz/cm
O
N
H
S
2p
180.308
140.921
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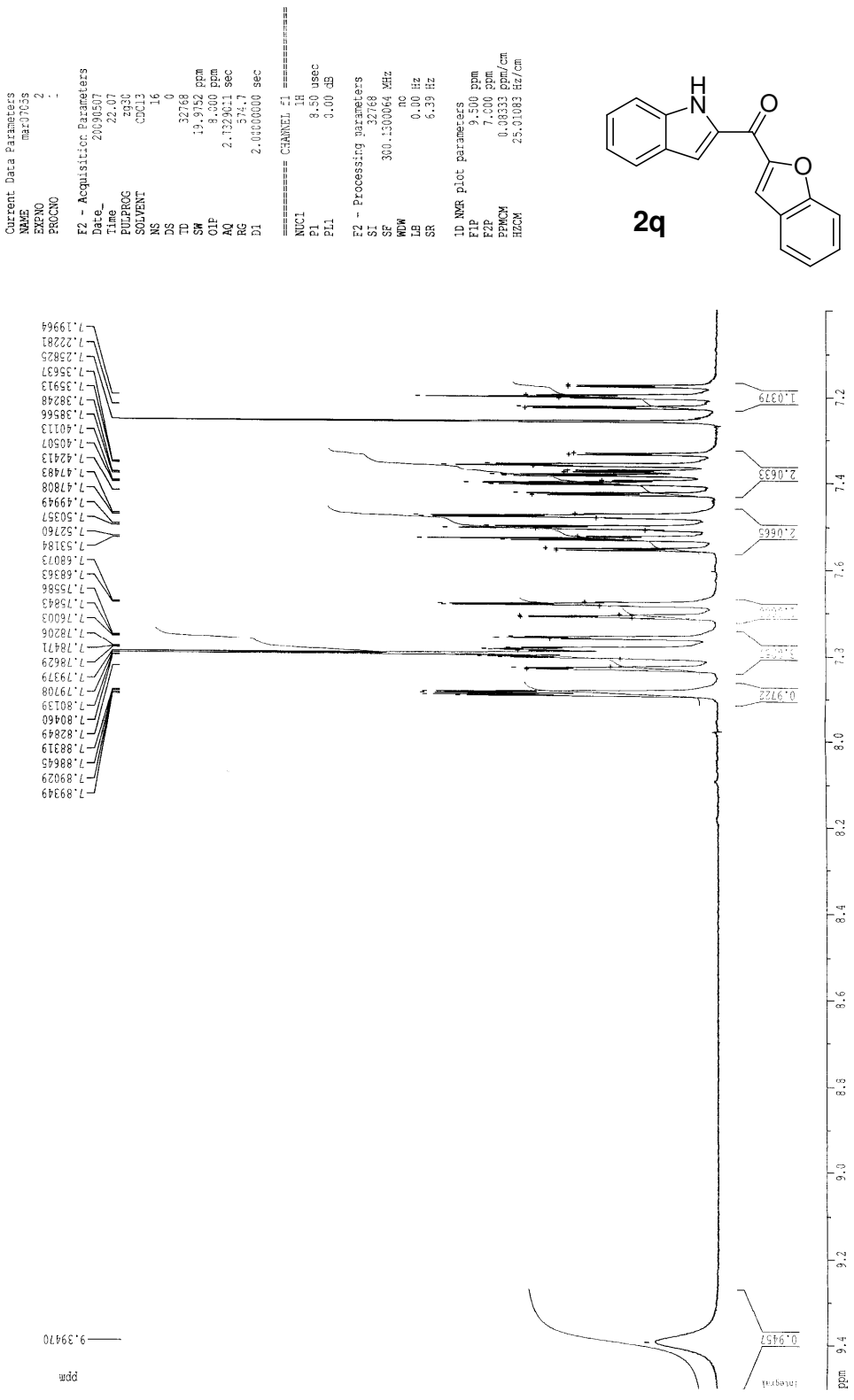
134.849
132.073
128.052
127.755
126.411
123.162
121.072
112.144
111.213
ppm


Current Data Parameters
NAME mar07b3s
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090507
Time 22.07
PULPROG zg30
SOLVENT CDCl3
NS 16
DS 0
TD 32768
SW 19.9752 ppm
O1P 8.000 ppm
AQ 2.1329611 sec
RG 574.7
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.50 usec
PL1 1.00 dB
F2 - Processing parameters
SI 32768
SF 300.1300064 MHz
WDW no
LB 0.00 Hz
SR 6.39 Hz
1D NMR plot parameters
F1P 9.500 ppm
F2P 7.000 ppm
PPMCM 0.08333 ppm/cm
HZCM 25.01083 Hz/cm
2q
7.19964
7.22281
7.25825
7.35637
7.35913
7.38248
7.38566
7.40113
7.40507
7.42413
7.47483
7.47808
7.49949
7.50357
7.52760
7.53184
7.68073
7.68363
7.75586
7.75843
7.76003
7.78206
7.78471
7.78629
7.79379
7.79708
7.80139
7.80460
7.82849
7.88319
7.88645
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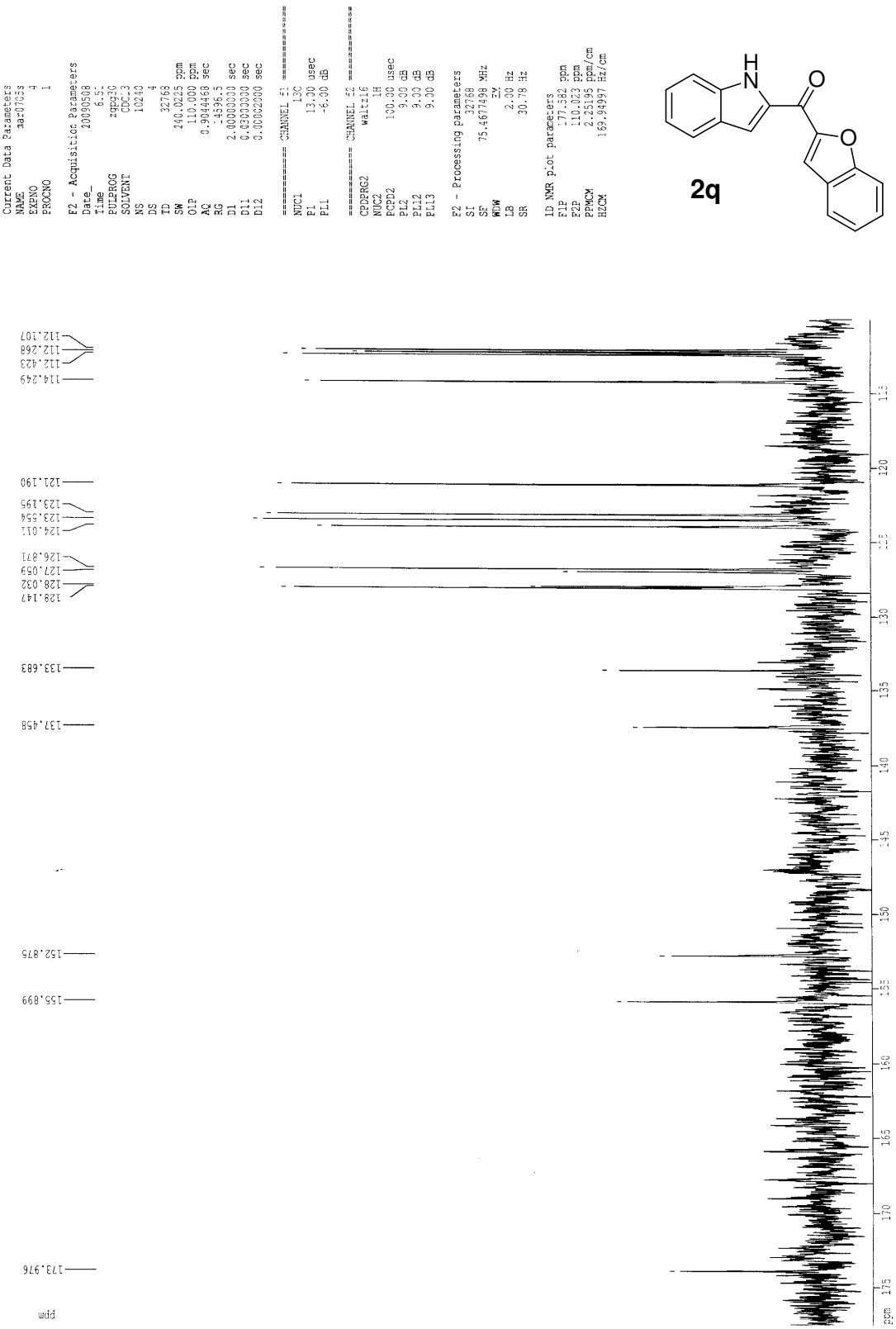
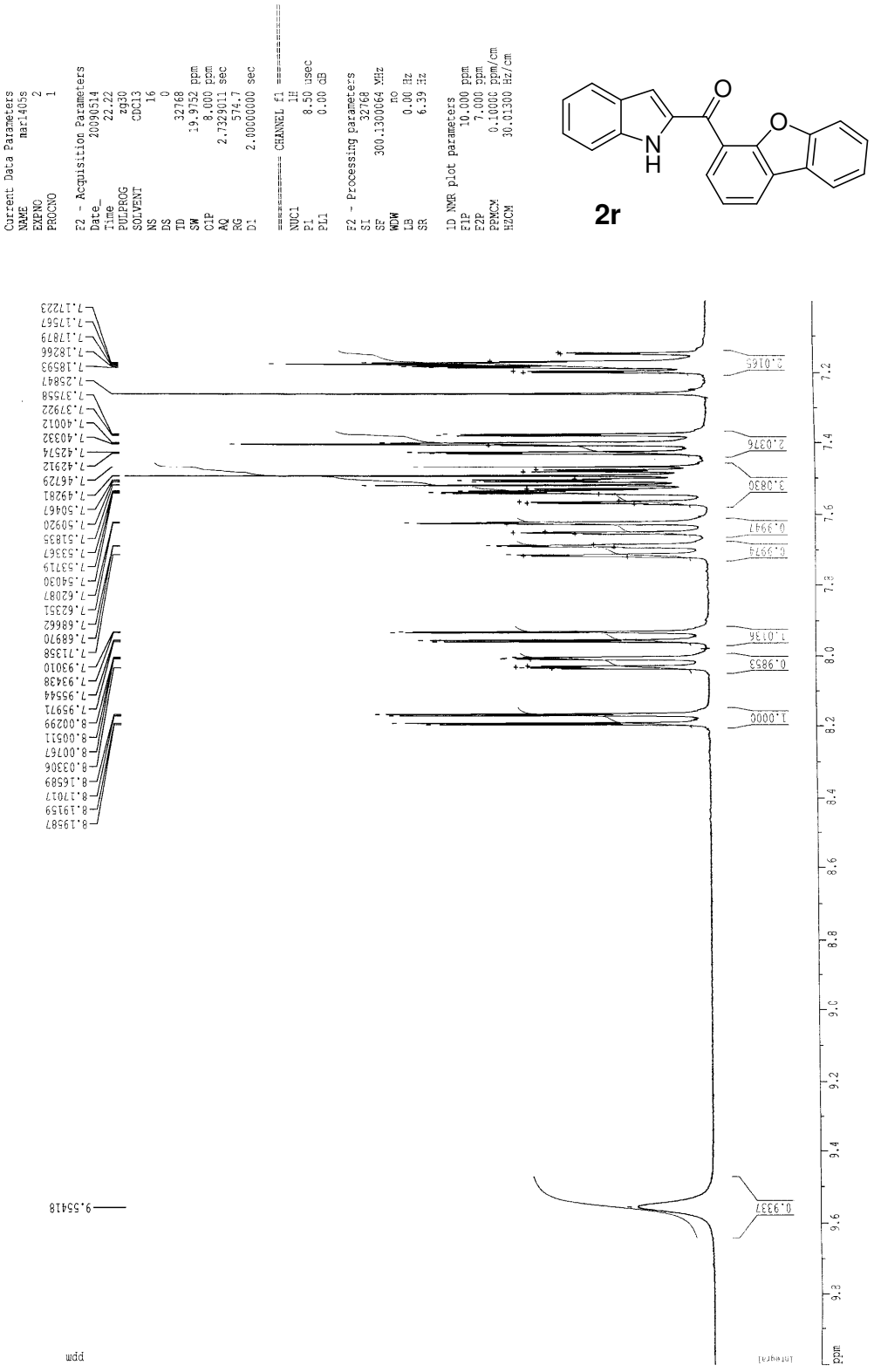
7.89349
9.39470
1.0379
2.0633
2.0665
0.9722
0.9457
ppm
integral


Current Data Parameters
NAME mar0705s
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090508
Time 6.5
PULPROG zgpg30
SOLVENT CDCl3
NS 10240
DS 4
TD 32768
SW 240.0225 ppm
O1P 110.000 ppm
AQ 0.9044468 sec
RG 4536.5
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 13.00 usec
PL1 -6.00 dB
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 9.00 dB
PL12 9.00 dB
PL13 9.00 dB
F2 - Processing parameters
SI 32768
SF 75.4677498 MHz
WDW EM
LB 2.00 Hz
SR 30.78 Hz
1D NMR plot parameters
F1P 177.582 ppm
F2P 110.023 ppm
PPMCM 2.25195 ppm/cm
HZCM 169.94997 Hz/cm
2q
H
N
O
O
112.107
112.268
112.423
114.249
121.190
123.195
123.554
124.011
126.871
127.059
128.032
128.147
133.683
137.458
152.875
155.099
173.976
ppm
110
115
120
125
130
135
140
145
150
155
160
165
170
175


Current Data Parameters
NAME mar1405s
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090514
Time_ 22.22
PULPROG zg30
SOLVENT CDCl3
NS 16
DS 0
TD 32768
SW 19.9752 ppm
O1P 8.000 ppm
AQ 2.7329011 sec
RG 574.7
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.50 usec
PL1 0.00 dB
F2 - Processing parameters
SI 32768
SF 300.1300064 MHz
WDW no
LB 0.00 Hz
SR 6.39 Hz
1D NMR plot parameters
F1P 10.000 ppm
F2P 7.000 ppm
PPMCM 0.10000 ppm/cm
HZCM 30.01300 Hz/cm
2r
O
N
H
O
7.17223
7.17567
7.17879
7.18266
7.18593
7.25847
7.37558
7.37922
7.40012
7.40332
7.42574
7.42912
7.46729
7.49281
7.50467
7.50920
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7.53719
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7.62087
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7.95544
7.95971
8.00299
8.00511
8.00767
8.03306
8.16589
8.17017
8.19159
8.19587
9.55418
2.0165
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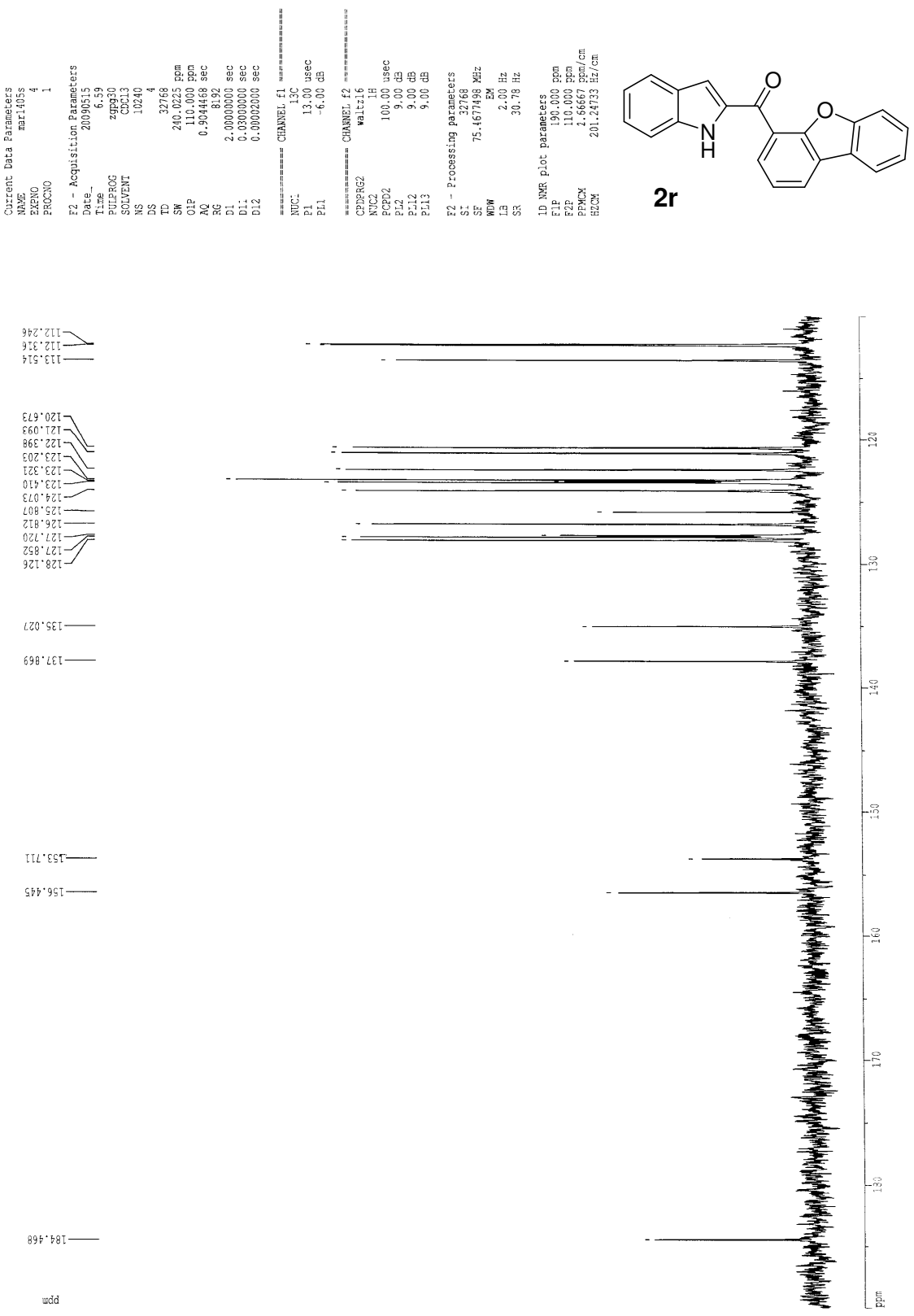
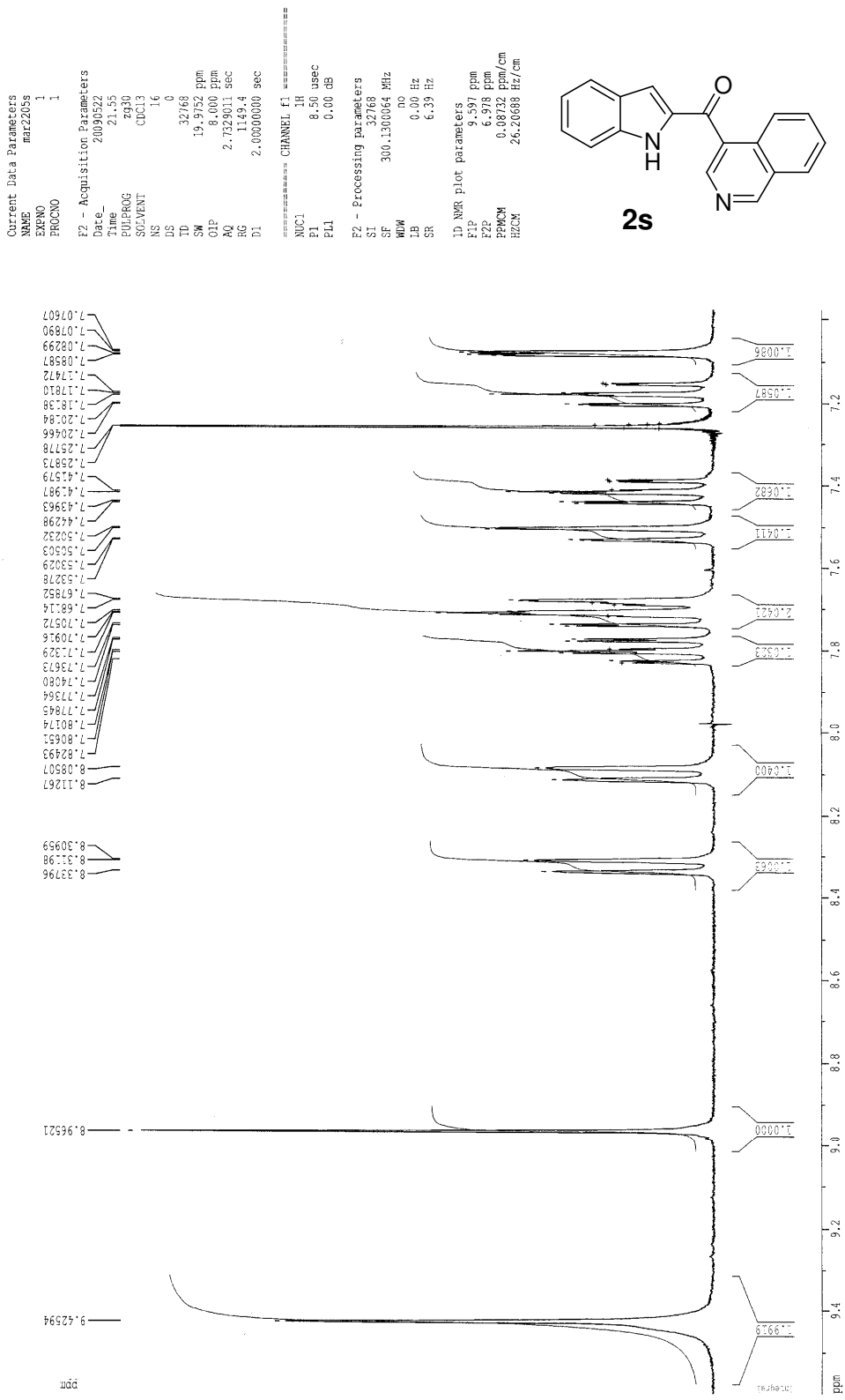
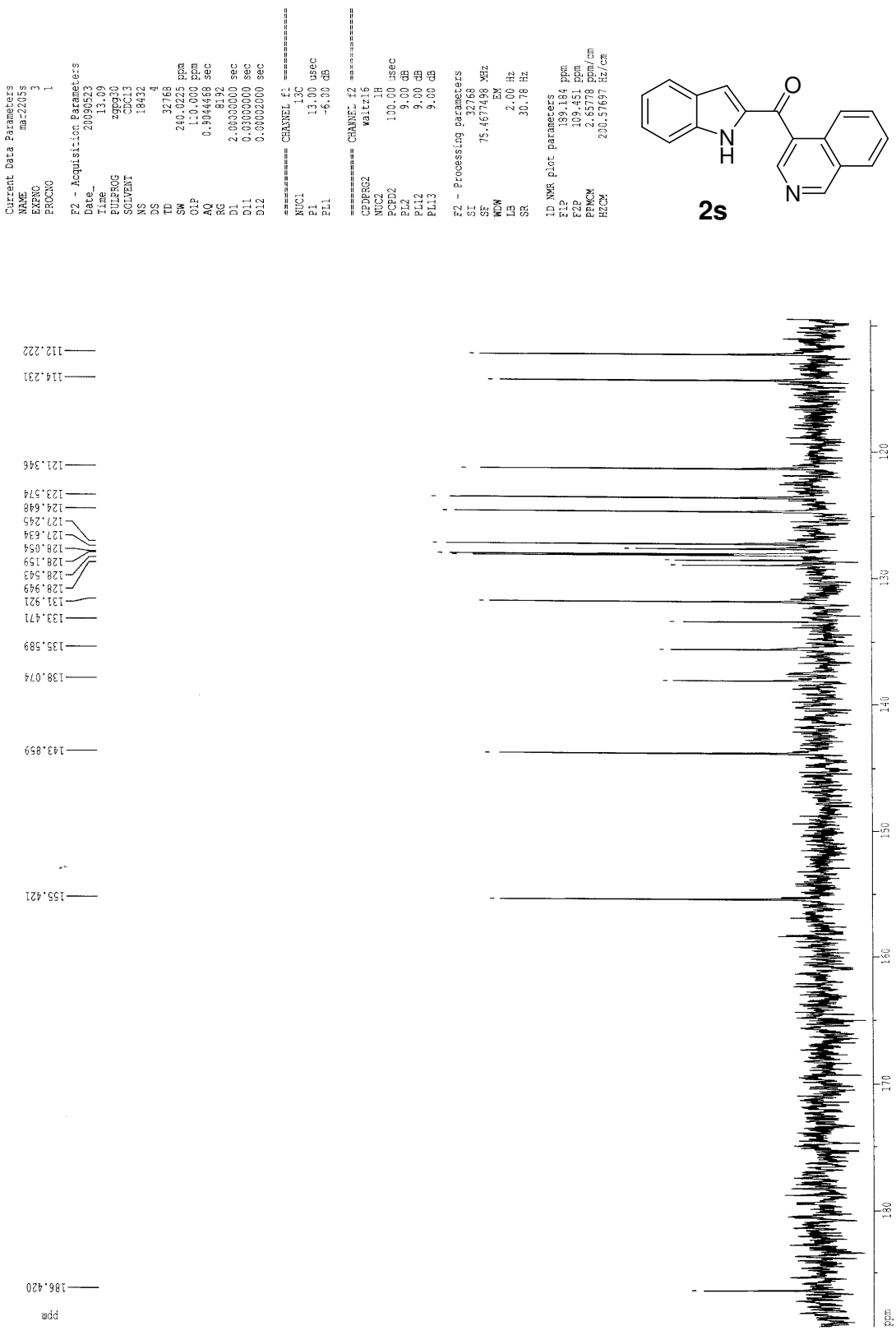
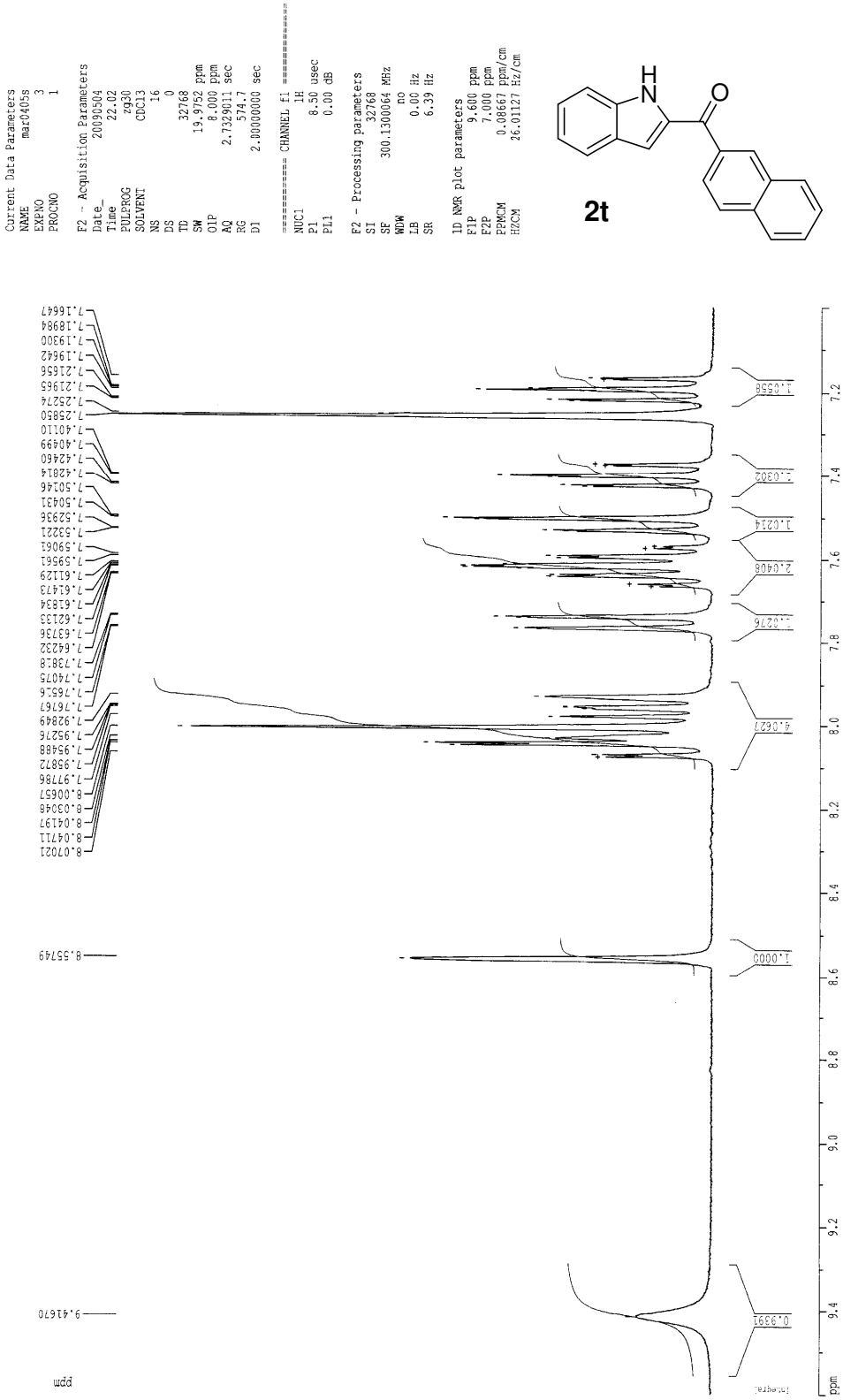
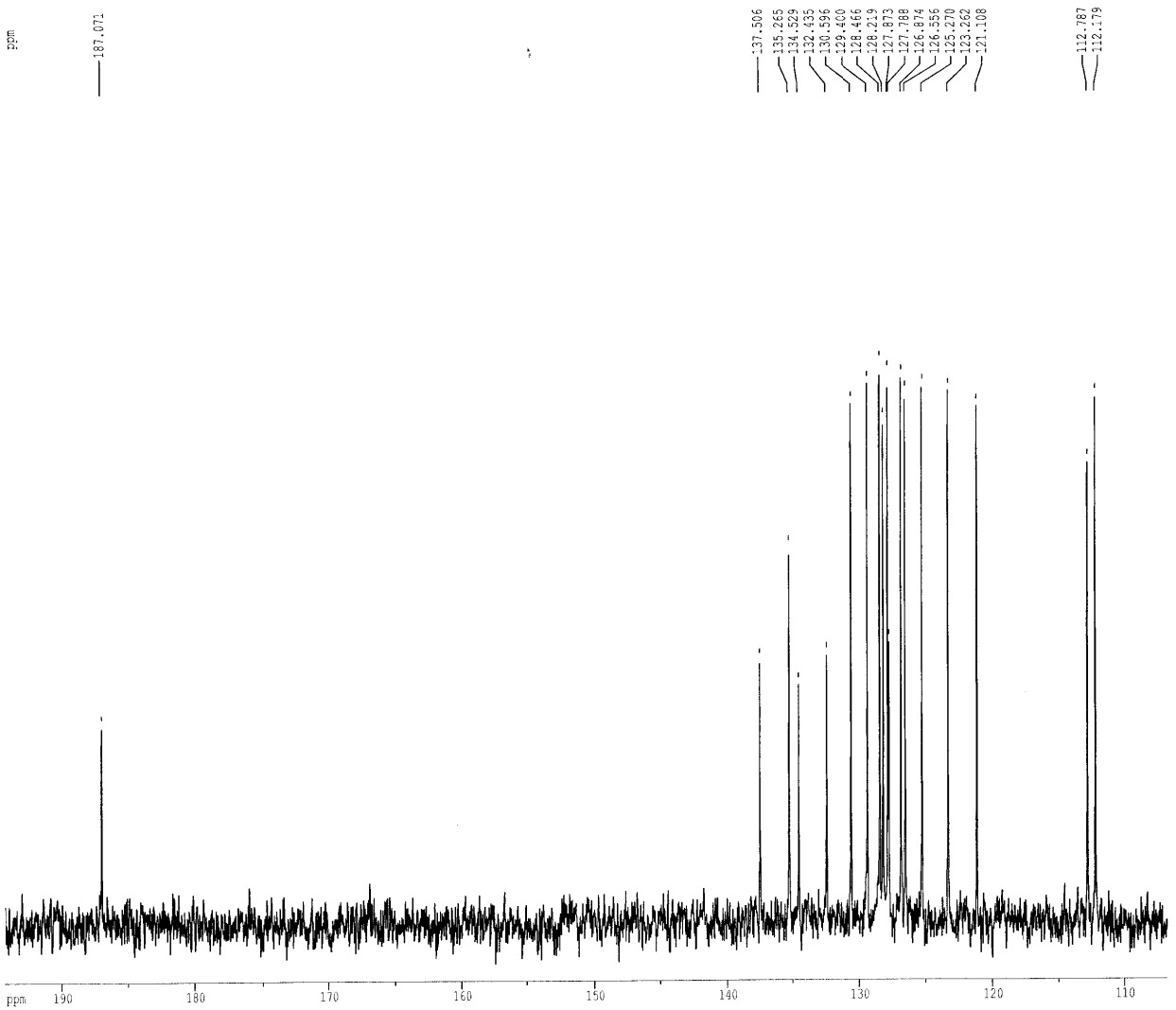
3.0830
0.9947
0.9979
1.0136
0.9853
1.0000
0.9337
ppm
ppm


Current Data Parameters
NAME mar1405s
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090515
Time_ 6.59
PULPROG zgpg30
SOLVENT CDCl3
NS 10240
DS 4
TD 32768
SW 240.0225 ppm
O1P 110.000 ppm
AQ 0.9044468 sec
RG 8192
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 13.00 usec
PL1 -6.00 dB
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 9.00 dB
PL12 9.00 dB
PL13 9.00 dB
F2 - Processing parameters
SI 32768
SF 75.4677498 MHz
WDW EM
LB 2.00 Hz
SR 30.78 Hz
1D NMR plot parameters
F1P 190.000 ppm
F2P 110.000 ppm
PPMCM 2.66667 ppm/cm
HZCM 201.24733 Hz/cm
O
N
H
O
2r
112.246
112.316
113.514
120.673
121.093
122.398
123.203
123.321
123.410
124.073
125.807
126.812
127.720
127.852
128.126
135.027
137.869
153.711
156.445
184.468
ppm
120
130
140
150
160
170
180
ppm


Current Data Parameters
NAME mar220ss
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090522
Time 21.55
PULPROG zg30
SOLVENT CDCl3
NS 16
DS 0
TD 32768
SW 19.9752 ppm
O1P 8.000 ppm
AQ 2.7329011 sec
RG 1149.4
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.50 usec
PL1 0.00 dB
F2 - Processing parameters
SI 32768
SF 300.1300064 MHz
WDW no
LB 0.00 Hz
SR 6.39 Hz
1D NMR plot parameters
F1P 9.597 ppm
F2P 6.978 ppm
PPMCM 0.08732 ppm/cm
HZCM 26.20608 Hz/cm
O
N
H
N
2s
7.07607
7.07890
7.08299
7.08587
7.11472
7.11810
7.18138
7.20184
7.20466
7.25778
7.25873
7.41579
7.41987
7.43963
7.44298
7.50232
7.50503
7.53029
7.53278
7.67852
7.68114
7.70572
7.70946
7.71329
7.73673
7.74080
7.77364
7.77846
7.80174
7.80651
7.82493
8.08507
8.11267
8.30959
8.31198
8.33796
8.96521
9.42594
1.0086
1.0587
1.0682
1.0611
0.9621
1.0323
1.0900
1.0063
1.0000
0.9929
ppm


Current Data Parameters
NAME ma-2205s
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090523
Time_ 13.09
PULPROG zgpg30
SOLVENT CDCl3
NS 18432
DS 4
TD 32768
SW 240.0225 ppm
O1P 110.000 ppm
AQ 0.9044468 sec
RG 8192
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 13.00 usec
PL1 -6.00 dB
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 9.00 dB
PL12 9.00 dB
PL13 9.00 dB
F2 - Processing parameters
SI 32768
SF 75.4677498 MHz
WDW EM
LB 2.00 Hz
SR 30.78 Hz
1D NMR plot parameters
F1P 189.184 ppm
F2P 109.451 ppm
PPMCM 2.65778 ppm/cm
HZCM 200.57697 Hz/cm
O
N
H
N
2s
112.222
114.231
121.346
123.574
124.648
127.245
127.634
128.054
128.159
128.543
128.949
131.921
133.471
135.589
138.074
143.859
155.421
186.420
ppm
120
130
140
150
160
170
180
ppm


Current Data Parameters
NAME mar0405s
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090504
Time 22.02
PULPROG zg30
SOLVENT CDCl3
NS 16
DS 0
TD 32768
SW 19.9752 ppm
O1P 8.000 ppm
AQ 2.7329011 sec
RG 574.7
D1 2.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.50 usec
PL1 0.00 dB
F2 - Processing parameters
SI 32768
SF 300.1300064 MHz
WDW no
LB 0.00 Hz
SR 6.39 Hz
1D NMR plot parameters
F1P 9.600 ppm
F2P 7.000 ppm
PPMCM 0.08667 ppm/cm
HZCM 26.01127 Hz/cm
H
N
O
2t
7.16647
7.18984
7.19300
7.19642
7.21656
7.21965
7.25274
7.25850
7.40110
7.40499
7.42460
7.42814
7.50146
7.50431
7.52936
7.53221
7.59061
7.59561
7.61129
7.61473
7.61834
7.62133
7.63736
7.64232
7.73818
7.74075
7.76516
7.76767
7.92849
7.95276
7.95488
7.95872
7.97786
8.00657
8.03048
8.04197
8.04711
8.07021
8.55749
9.41670
1.0558
1.0302
1.0214
2.0408
1.0276
4.0627
1.0000
0.9391
ppm



Current Data Parameters
NAME mar0405s
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090505
Time 6.21
PULPROG zgpg30
SOLVENT CDCl3
NS 9500
DS 4
TD 32768
SW 240.0225 ppm
O1P 110.000 ppm
AQ 0.90444468 sec
RG 8192
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 13.00 usec
PL1 -6.00 dB

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 9.00 dB
PL12 9.00 dB
PL13 9.00 dB

F2 - Processing parameters
SI 32768
SF 75.4677498 MHz
WDW EM
LB 2.00 Hz
SR 30.78 Hz

1D NMR plot parameters
F1P 194.256 ppm
F2P 106.861 ppm
PPMCM 2.91317 ppm/cm
HZCM 219.85016 Hz/cm

2t