

# Supporting Information

## Evolution of a Strategy for Total Synthesis of the Marine Fungal

### Alkaloid ( $\pm$ )-Communesin F

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**General Methods.** All non-aqueous reactions were carried out in oven- or flame-dried glassware under an argon atmosphere. All chemicals were purchased from commercial vendors and used as is, unless otherwise specified. Anhydrous tetrahydrofuran (THF) and dichloromethane ( $\text{CH}_2\text{Cl}_2$ ) were obtained from a solvent purification system. Reactions were magnetically stirred and monitored by thin layer chromatography (TLC) with 250  $\mu\text{m}$  precoated silica gel plates. Preparative TLC was performed with 500  $\mu\text{m}$  precoated silica gel plates. Flash column chromatography was performed using silica gel (230-400 mesh). Chemical shifts are reported relative to chloroform ( $\delta$  7.24), acetonitrile ( $\delta$  1.93) , toluene ( $\delta$  7.00) and methylene chloride ( $\delta$  5.32) for  $^1\text{H}$  NMR and chloroform ( $\delta$  77.0), acetonitrile ( $\delta$  1.3), toluene ( $\delta$  20.4) and methylene chloride ( $\delta$  54.0) ) for  $^{13}\text{C}$  NMR.

**Synthesis of TBS Ether 17b.** To a solution of alcohol **16** (507 mg, 1.82 mmol) and imidazole (247 mg, 3.63 mmol) in DMF (5 mL) was added TBSCl (329 mg, 2.18 mmol) in a single portion. The reaction mixture was stirred at rt for 3 h and then diluted with EtOAc (150 mL). The solution was washed with water (3×30 mL) and brine (30 mL), dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated *in vacuo*. The residue was purified by flash column chromatography (1:10 EtOAc:hexanes) to give the TBS ether **17b** (714 mg, 100%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.73 (d, *J* = 7.6 Hz, 1H), 7.56 (d, *J* = 7.2 Hz, 1H), 7.48 (t, *J* = 7.8 Hz, 1H), 4.68 (s, 2H), 0.98 (s, 9H), 0.17 (s, 6H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 154.8, 146.9, 130.5, 129.3, 123.5, 87.4, 70.7, 26.4, 18.8, -4.9; LRMS-ES (*m/z*): [M - NO<sub>2</sub> + H]<sup>+</sup> calcd for C<sub>13</sub>H<sub>21</sub>IOSi, 348.0; found, 348.1.

**Synthesis of Aniline 18b.** To a solution of the nitrobenzene **17b** (714 mg, 1.82 mmol) in EtOH (7 mL) and glacial acetic acid (7 mL) was added iron powder (507 mg, 9.08 mmol). The mixture was heated at 60 °C for 4 h and then cooled to rt. The mixture was diluted with water (150 mL) and carefully neutralized with solid Na<sub>2</sub>CO<sub>3</sub>. The resulting solution was extracted with EtOAc (150 mL). The organic layer was dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated *in vacuo*. The residue was purified by flash column chromatography (1:5 EtOAc:hexanes) to give the aniline **18b** (2.36 g, 95%) as a colorless oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.16 (t, *J* = 7.7 Hz, 1H), 6.93 (d, *J* = 7.5 Hz, 1H), 6.69 (d, *J* = 7.8 Hz, 1H), 4.63 (s, 2H), 4.18 (bs, 2H), 1.00 (s, 9H), 0.17 (s, 6H). HRMS-ES (*m/z*): [M + H]<sup>+</sup> calcd for C<sub>13</sub>H<sub>23</sub>NOSi, 364.0594; found, 364.0612.

**Synthesis of Amide 23b.** A mixture of the acid **21** (3.72 g, 11.0 mmol) and SOCl<sub>2</sub> (16 mL) was refluxed for 3 h. Excess SOCl<sub>2</sub> was removed under reduced pressure and the residue was diluted with CH<sub>2</sub>Cl<sub>2</sub> (20 mL) to give a stock solution of acid chloride (0.55 M). To a stirred solution of the aniline **18b** (2.54 g, 7.0 mmol) and (*i*-Pr)<sub>2</sub>NEt (4.9 mL, 28.1 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (50 mL) was added the above solution of the acid chloride (16.5 mL, 0.55 M in CH<sub>2</sub>Cl<sub>2</sub>, 9.1 mmol)

dropwise at 0 °C. The reaction mixture was warmed to rt and stirred overnight. The mixture was diluted with CH<sub>2</sub>Cl<sub>2</sub> (100 mL) and saturated aqueous NaHCO<sub>3</sub> (50 mL). The organic layer was dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated *in vacuo*. The residue was purified by flash column chromatography (1:1:2 EtOAc:CH<sub>2</sub>Cl<sub>2</sub>:hexanes) to give amide **23b** (3.24 g, 68%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.00 (d, *J* = 8.1 Hz, 1H), 7.79 (d, *J* = 6.9 Hz, 1H), 7.71 (s, 1H), 7.55 (t, *J* = 7.5 Hz, 1H), 7.43-7.33 (m, 6H), 7.29-7.20 (m, 3H), 4.53 (s, 2H), 3.71 (d, *J* = 6.2 Hz, 2H), 3.27, 3.07 (ABq, *J* = 16.5 Hz, 2H), 2.93-2.70 (m, 4H), .095 (s, 9H), 0.11 (s, 6H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 167.0, 148.3, 144.0, 138.2, 138.1, 137.9, 135.6, 134.3, 131.5, 130.4, 129.4, 129.3, 129.0, 128.8, 127.7, 125.2, 124.1, 121.4, 92.4, 70.5, 61.9, 57.3, 49.2, 26.9, 26.3, 18.8, -4.9; HRMS-ES (*m/z*): [M + H]<sup>+</sup> calcd for C<sub>32</sub>H<sub>39</sub>N<sub>3</sub>O<sub>4</sub>SiI, 684.1755; found, 684.1754.

**Synthesis of Ethyl Carbamate 24b.** To a stirred solution of amide **23b** (503 mg, 0.735 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (10 mL) was added ClCO<sub>2</sub>Et (0.084 mL, 0.879 mmol) dropwise at 0 °C and then the temperature was gradually raised to rt. The mixture was stirred at rt overnight and diluted with saturated aqueous NaHCO<sub>3</sub> (30 mL) and CH<sub>2</sub>Cl<sub>2</sub> (80 mL). The organic layer was dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated *in vacuo*. The residue was purified by flash column chromatography (1:2:2 EtOAc:CH<sub>2</sub>Cl<sub>2</sub>:hexanes) to give the carbamate protected amide **24b** (464 mg, 95%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.07 (d, *J* = 8.1 Hz, 1H), 7.76 (dd, *J* = 2.9, 6.5 Hz, 1H), 7.63-7.59 (m, 2H), 7.47 (t, *J* = 7.7 Hz, 1H), 7.39 (d, *J* = 7.1 Hz, 1H), 7.25-7.20 (m, 2H), 4.52 (d, 2H), 4.24-3.72 (m, 6H), 2.79 (m, 1H), 2.65 (m, 1H), 1.30 (br m, 3H), 0.94 (s, 9H), 0.10 (s, 6H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 166.4, 155.6, 148.4, 144.1, 137.7, 137.0, 134.5, 134.4, 131.5, 130.8, 129.8, 129.0, 125.3, 124.4, 121.7, 92.8, 70.5, 62.1, 47.8, 40.3, 26.9, 26.4, 18.8, 15.1; HRMS-ES (*m/z*): [M + H]<sup>+</sup> calcd for C<sub>28</sub>H<sub>37</sub>N<sub>3</sub>O<sub>6</sub>SiI, 666.1496; found, 666.1489.

**Synthesis of *N*-Methyl Amide 25b.** To a stirred suspension of NaH (183 mg, 60% dispersion in mineral oil, 4.57 mmol) in THF (50 mL) was added a solution of amide **24b** (2.76 g, 4.15 mmol) in THF (10 mL) at 0 °C. The mixture was stirred at 0 °C for 15 min and at rt for 30 min. To the solution was added MeI (0.31 mL, 4.98 mmol) at 0 °C and the mixture was stirred at rt overnight. The reaction mixture was diluted with saturated aqueous NaHCO<sub>3</sub> (50 mL) and extracted with EtOAc (150 mL). The organic layer was dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated *in vacuo*. The residue was purified by flash column chromatography (1:1 EtOAc:hexanes) to give the *N*-methyl amide **25b** (2.46 g, 87%). <sup>1</sup>H NMR (300 MHz, toluene-*d*<sub>8</sub>, 90 °C) δ 7.84 (d, *J* = 8.1 Hz, 1H), 7.46 (d, *J* = 7.5 Hz, 1H), 7.37 (d, *J* = 7.6 Hz, 1H), 7.20-7.09 (m, 2H), 7.00 (t, 7.8 Hz, 1H), 6.65 (br s, 1H), 4.72 (s, 2H), 4.26-4.17 (m, 4H), 3.64 (br s, 2H), 2.98 (s, 3H), 2.44 (br m, 2H), 1.19 (m, 3H), 1.06 (s, 9H), 0.18 (s, 6H); <sup>13</sup>C NMR (75 MHz, toluene-*d*<sub>8</sub>, 90 °C) δ 168.5, 155.3, 149.4, 146.1, 146.0, 134.3, 133.0, 131.9, 129.3, 128.9, 127.6, 127.1, 124.9, 101.1, 70.7, 61.4, 47.9, 40.5, 37.6, 27.5, 26.2, 18.7, 14.9, -5.10, -5.13; HRMS-ES (*m/z*): [M + H]<sup>+</sup> calcd for C<sub>29</sub>H<sub>39</sub>N<sub>3</sub>O<sub>6</sub>SiI, 680.1653; found, 680.1649.

**Synthesis of Tetracyclic Enamide 26b.** To a solution of *N*-methyl amide **25b** (199 mg, 0.293 mmol) in DMA (4.0 mL) were added Pd(OAc)<sub>2</sub> (6.6 mg, 0.029 mmol), PPh<sub>3</sub> (23 mg, 0.088 mmol), *n*-Bu<sub>4</sub>NBr (189 mg, 0.586 mmol) and K<sub>2</sub>CO<sub>3</sub> (81 mg, 0.586 mmol). The mixture was stirred at 150 °C for 30 min. The catalyst was removed by filtration and washed with EtOAc (200 mL). The filtrate was washed with water (4×30 mL) and brine (30 mL), dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated *in vacuo*. The residue was purified by flash column chromatography (1:2 EtOAc:hexanes) to give the Heck product **26b** (142 mg, 88%). <sup>1</sup>H NMR (300 MHz, toluene-*d*<sub>8</sub>, 90 °C) δ 7.79 (s, 1H), 7.19-7.06 (m, 3H), 6.91 (d, *J* = 7.9 Hz, 1H), 6.76 (t, *J* = 7.6 Hz, 1H), 6.61 (t, *J* = 7.7 Hz, 1H), 6.37 (d, *J* = 7.6 Hz, 1H), 4.98, 4.71 (ABq, *J* = 12.5 Hz, 2H), 4.32 (ddd, *J* =

3.8, 12.9, 12.9 Hz, 1H), 4.22-4.06 (m, 3H), 2.95 (s, 3H), 2.64 (ddd,  $J = 4.9, 13.9, 13.9$  Hz, 1H), 1.79 (ddd,  $J = 3.5, 3.5, 14.1$  Hz, 1H), 1.17 (t,  $J = 7.1$  Hz, 3H), 1.06 (s, 9H), 0.26 (s, 3H), 0.21 (s, 3H);  $^{13}\text{C}$  NMR (75 MHz, toluene- $d_8$ , 90 °C)  $\delta$  177.9, 153.3, 150.8, 144.1, 139.0, 137.7, 132.5, 131.4, 130.5, 129.0, 128.2, 127.1, 124.2, 123.7, 109.1, 107.1, 62.5, 62.0, 50.2, 38.5, 31.3, 26.33, 26.28, 18.7, 14.5, -5.0, -5.1; HRMS-ES ( $m/z$ ):  $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{29}\text{H}_{38}\text{N}_3\text{O}_6\text{Si}$ , 552.2530; found, 552.2500.

**Synthesis of *N*-Boc Aniline 29b.** To a solution of Heck product **26b** (1.29 g, 2.33 mmol) in THF (60 mL) was added 10% Pd/C (500 mg). The mixture was stirred at rt under a  $\text{H}_2$  atmosphere (1 atm) for 9 h. The catalyst was removed by filtration through Celite and the filtrate was concentrated *in vacuo*. The residue was purified by flash column chromatography (1:1 EtOAc:hexanes) to give aniline (1.16 g, 95%). To a stirred solution of the aniline (1.16 g, 2.22 mmol) in THF (50 mL) and  $\text{H}_2\text{O}$  (25 mL) were added  $\text{K}_2\text{CO}_3$  (4.61 g, 33.36 mmol) and  $(\text{Boc})_2\text{O}$  (4.85 g, 22.22 mmol). The reaction mixture was stirred at 60 °C for 20 h and diluted with EtOAc (200 mL). The organic layer was washed with brine (20 mL), dried over  $\text{Na}_2\text{SO}_4$  and concentrated *in vacuo*. The residue was purified by flash column chromatography (1:3 EtOAc:hexanes) to give the *N*-Boc aniline **29b** (1.22 g, 89%).  $^1\text{H}$  NMR (300 MHz, toluene- $d_8$ , 90 °C)  $\delta$  8.06 (dd,  $J = 1.1, 8.3$  Hz, 1H), 7.52 (br s, 1H), 7.35 (s, 1H), 7.17 (d,  $J = 7.9$  Hz, 1H), 6.98 (t,  $J = 7.8$  Hz, 1H), 6.87 (m, 1H), 6.66 (dd,  $J = 1.6, 7.8$  Hz, 1H), 6.48 (ddd,  $J = 1.2, 7.7, 7.7$  Hz, 1H), 6.14 (d,  $J = 7.7$  Hz, 1H), 4.87 (s, 2H), 4.16-3.87 (m, 4H), 2.61 (s, 3H), 2.27 (ddd,  $J = 4.5, 9.7, 15.7$  Hz, 1H), 1.84 (ddd,  $J = 4.0, 4.0, 14.2$  Hz, 1H), 1.49 (s, 9H), 1.01 (t,  $J = 7.1$  Hz, 3H), 0.95 (s, 9H), 0.12 (s, 3H), 0.10 (s, 3H);  $^{13}\text{C}$  NMR (75 MHz, toluene- $d_8$ , 90 °C)  $\delta$  179.2, 153.7, 153.4, 144.3, 138.9, 138.4, 131.7, 131.0, 128.6, 128.2, 123.4, 122.2, 122.1, 111.6, 107.5, 79.5,

62.3, 61.8, 51.3, 39.1, 32.2, 28.7, 26.3 26.1, 18.7, 14.6, -5.0, -5.1; HRMS-ES ( $m/z$ ):  $[M + H]^+$  calcd for  $C_{34}H_{48}N_3O_6Si$ , 622.3312; found, 622.3312.

**Synthesis of Pentacyclic Aminal 30b.** To a stirred solution of *N*-Boc aniline **29b** (124 mg, 0.199 mmol) in THF (10 mL) was added  $AlH_3 \cdot Me_2NEt$  (0.60 mL, 0.5 M in toluene, 0.300 mmol) dropwise at 0 °C. After 1 h the mixture was quenched with saturated aqueous  $Na_2SO_4$  (0.6 mL) and then stirred at rt overnight. The mixture was diluted with EtOAc (50 mL), dried over  $Na_2SO_4$  and concentrated *in vacuo*. The residue was purified by flash column chromatography (1:5 EtOAc:hexanes) to give aminal **30b** (101 mg, 83%) and unreacted *N*-Boc aniline **29b** (10 mg, 8%).  $^1H$  NMR (300 MHz, toluene- $d_8$ , 90 °C)  $\delta$  7.27 (s, 1H), 7.12 (dd,  $J = 1.3, 7.8$  Hz, 1H), 7.00 (dd,  $J = 1.7, 7.4$  Hz, 1H), 6.81 (t,  $J = 7.7$  Hz, 1H), 6.77 (ddd,  $J = 1.7, 7.6, 7.6$  Hz, 1H), 6.72-6.68 (m, 2H), 6.02 (d,  $J = 7.7$  Hz, 1H), 5.88 (s, 1H), 4.84, 4.56 (ABq,  $J = 12.7$  Hz, 2H), 4.13-4.01 (m, 3H), 3.22 (ddd,  $J = 3.4, 11.1, 13.1$  Hz, 1H), 2.92 (s, 3H), 2.24 (ddd,  $J = 3.8, 10.7, 14.5$  Hz, 1H), 2.01 (ddd,  $J = 3.4, 5.0, 14.3$  Hz, 1H), 1.34 (s, 9H), 1.10 (t,  $J = 7.1$  Hz, 3H), 0.96 (s, 9H), 0.12 (s, 3H), 0.10 (s, 3H);  $^{13}C$  NMR (75 MHz, toluene- $d_8$ , 90 °C)  $\delta$  154.7, 153.6, 151.5, 139.5, 137.3, 134.2, 129.4, 128.9, 126.8, 126.6, 125.6, 124.9, 124.3 118.2, 117.3, 104.8, 86.1, 80.9, 62.2, 61.5, 52.1, 40.7, 35.0, 30.9, 28.4, 26.4, 18.8, 14.7, -5.0; HRMS-ES ( $m/z$ ):  $[M + H]^+$  calcd for  $C_{34}H_{48}N_3O_5Si$ , 606.3363; found, 606.3351.

**Allylation of Enamide 30b.** To a stirred solution of enamide **30b** (81 mg, 0.134 mmol) in THF (5.0 mL) was added *n*-BuLi (0.26 mL, 1.6 M in hexanes, 0.416 mmol) dropwise at -78 °C. After stirring the mixture for 10 min at the same temperature, allyl iodide (0.043 ml, 0.470 mmol) was added. The mixture was warmed gradually to rt. After 15 min at rt, the mixture was diluted with saturated aqueous  $NaHCO_3$  (10 mL) and EtOAc (30 mL). The organic layer was dried over  $Na_2SO_4$  and concentrated *in vacuo*. The residue was purified by flash column

chromatography (1:10 to 1:3 EtOAc:hexanes) to give the *C*-allyl product **32** (61 mg, 80%) and *N*-allyl enamine **33** (7 mg, 9%).

**C-Allyl Product 32:**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.75 (s, 1H), 7.32 (d,  $J = 8.3$  Hz, 1H), 7.06-6.94 (m, 4H), 6.74 (d,  $J = 7.8$  Hz, 1H), 6.27 (d,  $J = 7.7$  Hz, 1H), 5.64 (m, 1H), 5.51 (s, 1H), 5.0 (d,  $J = 10.0$  Hz, 1H), 4.94 (d,  $J = 16.9$  Hz, 1H), 4.34 (d,  $J = 13.8$  Hz, 1H), 4.09-4.00 (m, 2H), 3.55 (m, 1H), 2.99 (s, 3H), 2.70 (dd,  $J = 8.5, 13.1$  Hz, 1H), 2.62 (dd,  $J = 6.5, 13.1$  Hz, 1H), 2.38 (ddd,  $J = 4.7, 9.8, 9.8$  Hz, 1H), 1.87 (dd,  $J = 5.8, 13.7$  Hz, 1H), 1.46 (s, 9H), 0.93 (s, 9H), 0.09 (s, 3H), 0.03 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  165.5, 154.9, 150.9, 140.1, 137.8, 133.0, 132.7, 128.4, 128.3, 127.8, 126.6, 124.0, 122.8, 119.5, 117.1, 106.2, 84.1, 81.8, 63.7, 54.8, 47.7, 43.1, 41.9, 32.1, 29.0, 28.6, 26.4, 18.8, -4.77, -4.82; HRMS-ES ( $m/z$ ):  $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{34}\text{H}_{48}\text{N}_3\text{O}_3\text{Si}$ , 574.3465; found, 574.3455.

**N-Allyl Product 33.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.11 (br s, 1H), 7.05-6.87 (m, 4H), 6.65 (d,  $J = 7.7$  Hz, 1H), 6.47 (s, 1H), 6.14 (d,  $J = 7.7$  Hz, 1H), 5.91 (m, 1H), 5.67 (br s, 1H), 5.28 (dd,  $J = 1.2, 17.1$  Hz, 1H), 5.22 (d,  $J = 10.1$  Hz, 1H), 4.84, 4.57 (ABq,  $J = 13.8$  Hz, 2H), 3.60 (d,  $J = 6.1$  Hz, 2H), 3.15 (ddd,  $J = 3.9, 3.9, 11.2$  Hz, 1H), 2.93 (s, 3H), 2.90 (m, 1H), 2.25 (m, 2H), 1.50 (s, 9H), 0.97 (s, 9H), 0.12 (s, 6H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  154.9, 151.1, 138.5, 137.1, 135.5, 135.3, 135.1, 130.1, 128.2, 126.5, 125.2, 123.4, 118.3, 116.2, 108.1, 104.1, 87.6, 81.3, 60.9, 58.9, 50.5, 46.2, 34.9, 31.1, 28.8, 26.5, 19.0, -4.8; LRMS-ES ( $m/z$ ):  $[\text{M} + \text{H}]^+$  calcd for  $\text{C}_{34}\text{H}_{48}\text{N}_3\text{O}_3\text{Si}$ , 574.3; found, 574.3.

**Formation of *N,O*-Acetal 34 and Aldehyde 35.** To a stirred solution of imine **32** (28.9 mg, 0.050 mmol) in EtOH (3.0 mL) was added diethyl pyrocarbonate (8.9  $\mu\text{L}$ , 0.060 mmol) at rt. After 10 min the solvent was removed under reduced pressure. The crude residue was used for the next step without further purification due to the instability of the compound on column



chromatography. For analytical purposes the two diastereomers of *N,O*-acetal **34** and aldehyde **35** were isolated by preparative TLC (1:5 EtOAc:hexanes). More polar major diastereomer of **34**:  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.11–6.94 (m, 6H), 6.35 (d,  $J = 7.4$  Hz, 1H), 5.61 (s, 1H), 5.44 (d,  $J = 7.0$  Hz, 1H), 5.48–5.31 (m, 1H), 4.94 (m, 1H), 4.83 (d,  $J = 19.1$  Hz, 1H), 4.71 (d,  $J = 12.4$  Hz, 0.5H), 4.56 (d,  $J = 13.5$  Hz, 0.5H), 4.25 (q,  $J = 7.2$  Hz, 2H), 3.95 (d,  $J = 15.8$  Hz, 0.5H), 3.83 (d,  $J = 13.0$  Hz, 0.5H), 3.71 (d,  $J = 12.9$  Hz, 0.5H) 3.61–3.36 (m, 3.5H), 3.05 (s, 3H), 2.76 (m, 1H), 2.40–2.14 (m, 2H), 1.66 (m, 1H), 1.45 (s, 9H), 0.86 (s, 9H), 0.07 (d,  $J = 3.8$  Hz, 3H), -0.56 (d,  $J = 19.2$  Hz, 3H); HRMS-ES  $[\text{M}+\text{H}]^+$  calcd for  $\text{C}_{39}\text{H}_{58}\text{N}_3\text{O}_6\text{Si}$ , 692.4095; found, 692.4081. Less polar minor diastereomer of **34**:  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.05–6.93 (m, 6H), 6.30 (dd,  $J = 3.2, 5.8$  Hz, 1H), 5.82 (d,  $J = 4.9$  Hz, 1H), 5.53 (d,  $J = 8.7$  Hz, 1H), 5.37–5.24 (m, 1H), 4.95–4.85 (m, 2H), 4.69 (d,  $J = 21.3$  Hz, 0.5H), 4.54 (d,  $J = 13.7$  Hz, 0.5H), 4.28–3.95 (m, 4H), 3.36–3.21 (m, 1H) 3.01 (s, 3H), 2.80–2.70 (m, 2H), 2.54–2.35 (m, 2H), 2.27 (ddd,  $J = 4.9, 13.1, 13.1$  Hz, 1H), 1.73 (t,  $J = 11.7$  Hz, 1H), 1.45 (s, 9H), 1.32 (ddd,  $J = 6.9, 6.9, 13.9$  Hz, 3H), 0.98 (t,  $J = 6.9$  Hz, 3H), 0.87 (s, 9H), 0.11 (d,  $J = 4.7$  Hz, 3H), 0.06 (d,  $J = 3.5$  Hz, 3H); HRMS-ES  $[\text{M}+\text{H}]^+$  calcd for  $\text{C}_{39}\text{H}_{58}\text{N}_3\text{O}_6\text{Si}$ , 692.4095; found, 692.4077.

Aldehyde **35**:  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  10.37 (s, 1H), 7.05–6.92 (m, 4H), 6.76 (br s, 1H), 6.56 (d,  $J = 7.6$  Hz, 1H), 6.49 (d,  $J = 7.8$  Hz, 1H), 5.63 (s, 1H), 5.27 (br s, 1H), 4.96–4.90 (m, 2H), 4.76–4.59 (m, 1H), 4.39–4.18 (m, 3H), 3.49 (m, 1H), 3.08 (m, 1H), 3.02 (s, 3H), 2.69 (m, 1H), 2.43 (m, 1H), 2.32 (ddd,  $J = 5.0, 13.5, 13.5$  Hz, 1H), 1.82 (m, 1H), 1.45 (s, 9H), 1.33 (t,  $J = 7.1$  Hz, 3H);  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  196.5, 156.7, 154.8, 152.0, 138.8, 136.9, 135.2, 133.9, 130.3, 129.0, 127.5, 126.7, 125.1, 123.4, 119.4, 118.2, 110.8, 84.3, 82.0, 62.3, 59.6, 45.4, 41.5, 39.8, 35.4, 32.4, 31.9, 28.6, 15.1; HRMS-ES  $[\text{M}+\text{H}]^+$  calcd for  $\text{C}_{31}\text{H}_{38}\text{N}_3\text{O}_5$ , 532.2811; found, 532.2788.

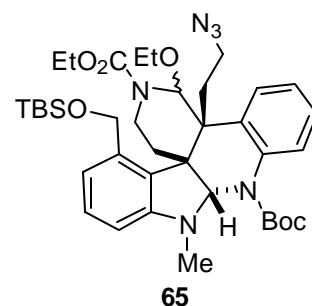
**Synthesis of Hydroxy *N,O*-Acetal **36**.** To a solution of the above crude *N,O*-acetal **34** in dioxane (2.0 mL) and H<sub>2</sub>O (1.0 mL) was added NMO (59 mg, 0.504 mmol) at 0 °C, followed by OsO<sub>4</sub> (0.01 mL, 4 wt% in H<sub>2</sub>O). The mixture was gradually warmed to rt. The mixture was stirred at rt for 6 h and then diluted with saturated aqueous NaHCO<sub>3</sub> (10 mL) and EtOAc (20 mL). The organic layer was dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated *in vacuo*. The crude residue was dissolved in THF (2.0 mL) and H<sub>2</sub>O (1.0 mL), and NaIO<sub>4</sub> (108 mg, 0.505 mmol) was added. The mixture was stirred at rt for 2 h and then diluted with saturated aqueous NaHCO<sub>3</sub> (10 mL) and EtOAc (20 mL). The organic layer was dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated *in vacuo*. The crude residue was dissolved in EtOH (2.0 mL) and NaBH<sub>4</sub> (9.5 mg, 0.251 mmol) was added at 0 °C. After 10 min the mixture was quenched with saturated aqueous NH<sub>4</sub>Cl (0.5 mL) and then diluted with saturated aqueous NaHCO<sub>3</sub> (10 mL) and EtOAc (20 mL). The organic layer was dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated *in vacuo*. The residue was purified by flash column chromatography (1:1:0.01 EtOAc:hexanes:NEt<sub>3</sub>) to give hydroxy *N,O*-acetal **36** (15.7 mg, 45% for 4 steps) as a mixture of diastereomers (2:1) and rearranged diol **37** (8.0 mg, 30%).

***N,O*-Acetal **36**:** <sup>1</sup>H NMR (300 MHz, benzene-*d*<sub>6</sub>, 65 °C, 2:1 mixture of diastereomers) δ 7.32 (d, *J* = 7.8 Hz, 1H, *major*), 7.22 (d, *J* = 8.0 Hz, 1H, *minor*), 7.01-6.71 (m, 5H, *major and minor*), 6.22 (d, *J* = 7.7 Hz, 1H, *major*), 6.18 (d, *J* = 6.8 Hz, 1H, *minor*), 5.99 (br s, 1H, *minor*), 5.80 (s, 1H, *major*), 5.60 (s, 1H, *minor*), 5.53 (s, 1H, *major*), 5.06 (br s, 1H, *major and minor*), 4.27-3.91 (m, 4H, *major and minor*), 3.58-3.35 (m, 4H, *major and minor*), 3.04 (s, 3H, *major*), 3.01 (s, 3H, *minor*), 2.82 (m, 1H, *minor*), 2.63 (m, 1H, *minor*), 2.29-2.19 (m, 2H, *major*), 2.01-1.88 (m, 2H, *major and minor*), 1.56-1.49 (m, 1H, *major and minor*), 1.35 (s, 9H, *major and minor*), 1.15 (t, *J* = 7.1 Hz, 3H, *major*), 1.11 (t, *J* = 7.2 Hz, 3H, *minor*), 1.02 (s, 9H, *minor*), 0.98-0.94 (m, 3H, *major and minor*), 0.96 (s, 9H, *major*), 0.26 (s, 3H, *minor*), 0.25 (s, 3H, *minor*),

0.08 (s, 3H, *major*), -0.37 (br s, 3H, *major*); LRMS-ES  $[M+H]^+$  calcd for  $C_{38}H_{58}N_3O_7Si$ , 696.4; found, 696.7.

**Diol 37:**  $^1H$  NMR (300 MHz, toluene- $d_8$ , 90 °C)  $\delta$  6.90-6.86 (m, 1H), 6.84-6.81 (m, 1H), 6.77 (t,  $J = 7.7$  Hz, 1H), 6.70-6.65 (m, 2H), 6.35 (d,  $J = 7.7$  Hz, 1H), 6.13 (d,  $J = 7.5$  Hz, 1H), 5.50 (s, 1H), 4.84 (d,  $J = 14.2$  Hz, 1H), 4.32, 4.21 (ABq,  $J = 12.0$  Hz, 2H), 4.20-4.05 (m, 4H), 3.45 (ddd,  $J = 6.1, 6.1, 12.2$  Hz, 1H), 3.31 (ddd,  $J = 5.9, 5.9, 11.7$  Hz, 1H), 3.15 (ddd,  $J = 6.8, 6.8, 16.8$  Hz, 1H), 2.95 (s, 3H), 2.17-2.10 (m, 1H), 2.00-1.84 (m, 2H), 1.47 (ddd,  $J = 2.6, 2.6, 13.5$  Hz, 1H), 1.32 (s, 9H), 1.14 (t,  $J = 7.1$  Hz, 3H); HRMS-ES  $[M+H]^+$  calcd for  $C_{30}H_{40}N_3O_6$ , 538.2917; found, 538.2910.

**Synthesis of Azido *N,O*-Acetal 65.** To a solution of hydroxy *N,O*-acetal **36** (15.7 mg, 0.023 mmol),  $PPh_3$  (48 mg, 0.183 mmol) and DPPA (0.04 mL, 0.186 mmol) in THF (1.0 mL) was added DEAD (28  $\mu$ L, 0.178 mmol) at rt. After 30 min the solvent was removed under reduced pressure and the residue was purified by preparative TLC



(1:1:0.01 EtOAc:hexanes: $NEt_3$ ) to give the azido *N,O*-acetal **65** (9.8 mg, 60%) as a mixture of diastereomers (1:0.6).  $^1H$  NMR (300 MHz, benzene- $d_6$ , 65 °C, 1:0.6 mixture of diastereomers)  $\delta$  7.30 (d,  $J = 7.7$  Hz, 1H, *major*), 7.22 (dd,  $J = 1.2, 8.0$  Hz, 1H, *minor*), 7.06-6.90 (m, 2H, *major and minor*), 6.82-6.68 (m, 3H, *major and minor*), 6.20 (dd,  $J = 1.0, 7.7$  Hz, 1H, *major*), 6.16 (dd,  $J = 1.1, 7.7$  Hz, 1H, *minor*), 5.99 (br s, 1H, *minor*), 5.76 (s, 1H, *major*), 5.53 (s, 1H, *minor*), 5.45 (s, 1H, *major*), 4.76 (br s, 1H, *major and minor*), 4.28-3.89 (m, 4H, *major and minor*), 3.67-3.35 (m, 3H, *major and minor*), 3.02 (s, 3H, *major*), 2.99 (s, 3H, *minor*), 2.91 (m, 1H, *minor*), 2.75-2.59 (m, 2H, *major*, 1H, *minor*), 2.32-2.18 (m, 2H, *major*), 1.99-1.78 (m, 2H, *major and minor*), 1.48-1.41 (m, 1H, *major and minor*), 1.38 (s, 9H, *major and minor*), 1.20-1.11 (m, 3H, *major*

and minor), 1.01 (s, 9H, minor), 1.00-0.75 (m, 12H, major, 3H, minor), 0.25 (s, 3H, minor), 0.24 (s, 3H, minor), 0.09 (s, 3H, major), -0.33 (br s, 3H, major); LRMS-ES  $[M+Na]^+$  calcd for  $C_{38}H_{56}N_6NaO_6Si$ , 743.4; found, 743.6.

**Preparation of *N*-Boc Amino *N,O*-Acetal **38**.** To a solution of azido *N,O*-acetal **65** (4.6 mg, 6.4  $\mu$ mol) in EtOAc (2.0 mL) were added 10% Pd/C (3.0 mg) and  $Boc_2O$  (7.0 mg, 32.0  $\mu$ mol). The mixture was stirred at rt under a  $H_2$  atmosphere (1 atm) for 1 h. The catalyst was removed by filtration through Celite and the filtrate was concentrated *in vacuo*. The residue was purified by preparative TLC (1:3:0.01 EtOAc:hexanes: $NEt_3$ ) to give two separable diastereomers of *N*-Boc amino *N,O*-acetal **38** (2.5 mg / 1.1 mg, 49% / 22%; total yield 71%). More polar major diastereomer of **38**:  $^1H$  NMR (300 MHz, benzene- $d_6$ , 65  $^\circ C$ )  $\delta$  7.31 (d,  $J = 8.1$  Hz, 1H), 7.02-6.93 (m, 2H), 6.84-6.72 (m, 3H), 6.22 (d,  $J = 7.7$  Hz, 1H), 5.76 (s, 1H), 5.46 (s, 1H), 4.83 (br s, 1H), 4.28-3.89 (m, 5H), 3.71-3.43 (m, 3H), 3.21 (m, 1H), 3.05 (s, 3H), 3.01 (m, 1H), 2.07 (m, 1H), 1.94 (m, 1H), 1.49 (m, 1H), 1.41 (s, 18H), 1.17-1.12 (m, 6H), 0.96 (s, 9H), 0.07 (s, 3H), -0.37 (s, 3H); LRMS-ES  $[M+Na]^+$  calcd for  $C_{43}H_{66}N_4NaO_8Si$ , 817.5; found, 817.7. Less polar minor diastereomer of **38**:  $^1H$  NMR (300 MHz, benzene- $d_6$ , 65  $^\circ C$ )  $\delta$  7.22 (d,  $J = 8.0$  Hz, 1H), 7.00-6.91 (m, 2H), 6.77-6.65 (m, 3H), 6.17 (dd,  $J = 1.0, 7.7$  Hz, 1H), 5.97 (br s, 1H), 5.53 (s, 1H), 4.83 (br m, 1H), 4.42-3.95 (m, 5H), 3.43 (t,  $J = 13.6$  Hz, 1H), 3.18 (m, 1H), 3.02 (s, 3H), 2.94-2.79 (m, 2H), 2.66-2.57 (m, 1H), 2.08 (m, 1H), 1.93 (m, 1H), 1.50 (m, 1H), 1.41 (s, 18H), 1.19 (m, 3H), 1.02 (s, 9H), 0.95 (t,  $J = 7.0$  Hz, 3H), 0.26 (s, 3H), 0.25 (s, 3H); LRMS-ES  $[M+Na]^+$  calcd for  $C_{43}H_{66}N_4NaO_8Si$ , 817.5; found, 817.7.

**Synthesis of Amide **23c**.** A mixture of the acid **21** (3.72 g, 11.0 mmol) and  $SOCl_2$  (16 mL) was refluxed for 3 h. Excess  $SOCl_2$  was removed under reduced pressure and the residue was diluted with  $CH_2Cl_2$  (20 mL) to give a stock solution of acid chloride (0.55 M). To a stirred

solution of aniline **18c** (435 mg, 1.46 mmol) and (*i*-Pr)<sub>2</sub>NEt (1.0 mL, 5.74 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (25 mL) was added the above solution of acid chloride (3.45 mL, 0.55 M in CH<sub>2</sub>Cl<sub>2</sub>, 1.90 mmol) dropwise at 0 °C. The reaction mixture was warmed to rt and stirred overnight. The mixture was diluted with CH<sub>2</sub>Cl<sub>2</sub> (150 mL) and saturated aqueous NaHCO<sub>3</sub> (30 mL). The organic layer was dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated *in vacuo*. The residue was purified by flash column chromatography (1:4 EtOAc:hexanes to 1:2:2 EtOAc:CH<sub>2</sub>Cl<sub>2</sub>:hexanes) to give the amide **23c** (717 mg, 79%, 91% based on recovered aniline **18c**) and unreacted aniline **18c** (54 mg, 12%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.01 (dd, *J* = 1.1, 8.2 Hz, 1H), 7.91 (dd, *J* = 1.3, 8.2 Hz, 1H), 7.78 (s, 1H), 7.56 (ddd, *J* = 1.2, 7.5, 7.5 Hz, 1H), 7.45-7.26 (m, 8H), 7.10 (t, *J* = 8.1 Hz, 1H), 3.74, 3.69 (ABq, *J* = 13.5 Hz, 2H), 3.28, 3.08 (ABq, *J* = 16.4 Hz, 2H), 2.92 (m, 1H), 2.81-2.78 (m 2H), 2.68 (m, 1H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 166.8, 148.3, 140.8, 139.3, 138.1 135.5, 134.3, 131.3, 130.6, 130.2, 129.7, 129.4, 128.9, 127.8, 125.2, 120.6, 98.4, 61.9, 57.5, 49.2, 27.0; HRMS-ES (*m/z*): [M + H]<sup>+</sup> calcd for C<sub>25</sub>H<sub>22</sub>N<sub>3</sub>O<sub>3</sub>IBr, 617.9889; found, 617.9904.

**Synthesis of Ethyl Carbamate 24c.** To a stirred solution of amide **23c** (700 mg, 1.13 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (10 mL) was added ClCO<sub>2</sub>Et (0.12 mL, 1.26 mmol) dropwise at 0 °C and after 30 min the temperature was gradually raised to rt. The mixture was stirred at rt for 9 h and diluted with saturated aqueous NaHCO<sub>3</sub> (10 mL) and CH<sub>2</sub>Cl<sub>2</sub> (20 mL). The organic layer was dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated *in vacuo*. The residue was purified by flash column chromatography (1:3:3 EtOAc:CH<sub>2</sub>Cl<sub>2</sub>:hexanes) to give the carbamate protected amide **24c** (666 mg, 98%). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ 8.04 (dd, *J* = 0.9, 8.2 Hz, 1H), 7.80 (dd, *J* = 1.4, 8.2 Hz, 1H), 7.70 (s, 1H), 7.58 (ddd, *J* = 1.2, 7.5, 7.5 Hz, 1H), 7.45 (t, *J* = 7.8 Hz, 1H), 7.35-7.27 (m, 2H), 7.05 (t, *J* = 8.1 Hz, 1H), 4.21-4.10 (m, 4H), 3.83-3.73 (m, 2H), 2.76, 2.60 (ABq, *J* = 16.8 Hz, 2H), 1.22 (t, *J* = 7.1 Hz, 3H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 166.4, 155.6, 148.3, 140.5, 138.2,

137.7, 134.6, 134.3, 131.4, 130.6, 130.2, 129.9, 129.1, 125.4, 120.6, 98.5, 62.2, 47.9, 40.2, 26.5, 15.1; HRMS-ES ( $m/z$ ):  $[M + NH_4]^+$  calcd for  $C_{21}H_{23}N_4O_5BrI$ , 616.9897; found, 616.9938.

**Synthesis of *N*-Methyl Amide 25c.** To a stirred suspension of NaH (16 mg, 60% dispersion in mineral oil, 0.400 mmol) in THF (10 mL) was added a solution of amide **24c** (220 mg, 0.366 mmol) in THF (2 mL) at 0 °C. The mixture was stirred at 0 °C for 15 min and at rt for 30 min. To the solution was added MeI (0.027 mL, 0.434 mmol) at 0 °C and the reaction mixture was stirred at rt overnight. The reaction mixture was diluted with saturated aqueous  $NaHCO_3$  (30 mL) and extracted with EtOAc (100 mL). The organic layer was dried over  $Na_2SO_4$  and concentrated *in vacuo*. The residue was purified by flash column chromatography (2:1 EtOAc:hexanes) to give the *N*-methyl amide **25c** (213 mg, 95%).  $^1H$  NMR (300 MHz, toluene- $d_8$ , 90 °C)  $\delta$  7.72 (d,  $J = 8.2$  Hz 1H), 7.22 (d,  $J = 7.5$  Hz, 1H), 7.14-7.08 (m, 2H), 6.98 (m, 1H), 6.68 (m, 1H), 6.43 (br s, 1H), 4.13-4.04 (m, 4H), 2.79 (s, 3H), 2.30 (br s, 2H), 1.07 (t,  $J = 7.0$  Hz 3H);  $^{13}C$  NMR (75 MHz, toluene- $d_8$ , 90 °C)  $\delta$  168.2, 155.2, 149.2, 148.2 133.8, 133.1, 132.0, 131.7, 131.4, 130.2, 129.0, 127.3, 124.8, 106.9, 61.4, 47.7, 40.3, 37.3, 27.0, 14.7; HRMS-ES ( $m/z$ ):  $[M + H]^+$  calcd for  $C_{22}H_{22}N_3O_5IBr$ , 613.9788; found, 613.9805.

**Heck Cyclization of Amide 25c.** To a solution of *N*-methyl amide **25c** (213 mg, 0.347 mmol) in DMA (7.0 mL) were added  $Pd(OAc)_2$  (7.8 mg, 0.035 mmol),  $PPh_3$  (27 mg, 0.104 mmol), *n*- $Bu_4NBr$  (224 mg, 0.694 mmol) and  $K_2CO_3$  (96 mg, 0.694 mmol). The mixture was stirred at 100 °C for 1 h. The catalyst was removed by filtration and washed with EtOAc (200 mL). The filtrate was washed with water (3×20 mL) and brine (20 mL), dried over  $Na_2SO_4$  and concentrated *in vacuo*. The residue was purified by flash column chromatography (1:1 to 3:1 EtOAc:hexanes) to give a mixture of Heck product **26c** and pentacyclic Heck product **27** (86 mg,

51%, **26c:27** = 1:0.15, 85% based on recovered starting material) and unreacted amide **25c** (86 mg, 40%). For analytical purposes further purification of **26c** and **27** was achieved by careful flash column chromatography (1:10 EtOAc: CH<sub>2</sub>Cl<sub>2</sub>).

**Heck Product 26c:** <sup>1</sup>H NMR (300 MHz, toluene-*d*<sub>8</sub>, 90 °C) δ 7.71 (s, 1H), 7.09 (dd, *J* = 1.3, 8.0 Hz, 1H), 7.05 (dd, *J* = 1.3, 7.9 Hz, 1H), 6.79 (ddd, *J* = 1.4, 7.5, 7.5 Hz, 1H), 6.75 (dd, *J* = 1.0, 8.2 Hz, 1H), 6.66-6.61 (m, 2H), 6.23 (dd, *J* = 0.9, 7.7 Hz, 1H), 4.24 (ddd, *J* = 3.9, 12.8, 12.8 Hz, 1H), 4.03 (q, *J* = 7.0 Hz, 2H), 4.01 (m, 1H), 2.84 (s, 3H), 2.82-2.74 (m, 1H), 1.46 (ddd, *J* = 3.1, 3.7, 13.7 Hz, 1H), 1.04 (t, *J* = 7.1 Hz, 3H); <sup>13</sup>C NMR (75 MHz, toluene-*d*<sub>8</sub>, 90 °C) δ 176.7, 153.3, 150.8, 146.3, 133.0, 132.6, 132.2, 130.7, 130.1, 129.2, 127.5, 126.9, 123.9, 119.7, 108.5, 107.1, 62.4, 51.1, 38.0, 28.2, 26.3, 14.3; HRMS-ES [M+H]<sup>+</sup> calcd for C<sub>22</sub>H<sub>21</sub>BrN<sub>3</sub>O<sub>5</sub>, 486.0665; found, 486.0695.

**Pentacyclic Heck Adduct 27:** <sup>1</sup>H NMR (300 MHz, toluene-*d*<sub>8</sub>, 90 °C) δ 7.48 (s, 1H), 7.42 (dd, *J* = 1.2, 7.9 Hz, 1H), 7.08 (dd, *J* = 1.3, 9.5 Hz, 1H), 6.99-6.89 m, 2H), 6.64 (t, *J* = 7.9 Hz, 1H), 6.16 (d, *J* = 7.2 Hz, 1H), 4.04-3.89 (m, 2H), 3.79 (ddd, *J* = 5.3, 13.3, 13.3 Hz, 1H), 3.40 (dd, *J* = 5.2, 13.1 Hz, 1H), 2.59 (s, 3H), 1.59 (ddd, *J* = 1.2, 5.3, 12.8 Hz, 1H), 1.36 (ddd, *J* = 5.7, 13.5, 13.5 Hz, 1H), 1.06 (t, *J* = 7.1 Hz, 3H); <sup>13</sup>C NMR (100 MHz, CD<sub>2</sub>Cl<sub>2</sub>) δ 180.6, 153.2, 148.9, 144.3, 133.3, 131.1, 130.1, 129.9, 129.6, 128.1, 126.1, 126.0, 125.0, 117.1, 108.7, 107.9, 63.2, 48.6, 38.4 (d, *J* = 27.4 Hz), 38.0, 26.7, 14.5; HRMS-ES [M+H]<sup>+</sup> calcd for C<sub>22</sub>H<sub>20</sub>N<sub>3</sub>O<sub>5</sub>, 406.1403; found, 406.1423.

**Preparation of Aniline 28.** To a solution of Heck products **26c** and **27** (149 mg, 6:1 mixture) in EtOH (6 mL) and H<sub>2</sub>O (1.5 mL) was added iron powder (86 mg, 1.54 mmol) and concentrated HCl (12 N, 10 μL). The mixture was stirred at 85 °C for 2 h and then cooled to rt. The mixture was filtered and the filtrate was concentrated. The residue obtained was diluted with

EtOAc and H<sub>2</sub>O. The organic layer was separated and the aqueous layer was extracted with EtOAc. The combined organic phases were dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated under reduced pressure. The residue was purified by flash column chromatography (1:5 EtOAc:CH<sub>2</sub>Cl<sub>2</sub>) to give aniline **28** (103 mg, 84%). <sup>1</sup>H NMR (300 MHz, toluene-*d*<sub>8</sub>, 90 °C) δ 7.53 (s, 1H), 6.98-6.96 (m, 1H), 6.84 (d, *J* = 8.2 Hz, 1H), 6.68 (t, *J* = 7.6 Hz, 1H), 6.55 (t, *J* = 7.6 Hz, 1H), 6.32 (t, *J* = 7.4 Hz, 1H), 6.17 (d, *J* = 8.0 Hz, 1H), 6.02 (d, *J* = 7.8 Hz, 1H), 4.23-4.18 (m, 2H), 4.02 (q, *J* = 7.1 Hz, 2H), 3.63 (br s, 2H), 2.90-2.79 (m, 1H), 2.64 (s, 3H), 1.54 (ddd, *J* = 3.3, 3.3, 13.9 Hz, 1H), 0.99 (t, *J* = 7.1 Hz, 3H); <sup>13</sup>C NMR (75 MHz, CD<sub>2</sub>Cl<sub>2</sub>) δ 179.1, 154.0, 146.0, 131.0, 130.7, 130.11, 130.06, 128.3, 127.2, 123.3, 119.7, 117.5, 115.8, 109.6, 107.7, 62.7, 51.8, 37.7, 28.2, 27.0, 14.8; HRMS-ES [M+H]<sup>+</sup> calcd for C<sub>22</sub>H<sub>23</sub>BrN<sub>3</sub>O<sub>3</sub>, 456.0925; found, 456.0929.

**Synthesis of *N*-Boc Aniline 29c.** To a stirred solution of aniline **28** (7.5 mg, 0.016 mmol) in THF (1.0 mL) and H<sub>2</sub>O (0.5 mL) were added K<sub>2</sub>CO<sub>3</sub> (34.0 mg, 0.246 mmol) and (Boc)<sub>2</sub>O (35.8 mg, 0.164 mmol). The reaction mixture was stirred at 60 °C. After 12 h, additional (Boc)<sub>2</sub>O (16.8 mg, 0.077 mmol) was added. After another 12 h, the mixture was diluted with EtOAc (30 mL). The organic layer was washed with saturated aqueous NaHCO<sub>3</sub> (10 mL), dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated *in vacuo*. The residue was purified by flash column chromatography (1:10 EtOAc:CH<sub>2</sub>Cl<sub>2</sub>) to give *N*-Boc aniline **29c** (8.2 mg, 90%). <sup>1</sup>H NMR (300 MHz, toluene-*d*<sub>8</sub>, 90 °C) δ 8.14 (dd, *J* = 1.1, 8.3 Hz, 1H), 7.42 (br s, 2H), 7.06-7.03 (m, 1H), 6.90-6.83 (m, 2H), 6.58-6.49 (m, 2H), 6.00 (d, *J* = 7.8 Hz, 1H), 4.16 (dd, *J* = 3.5, 8.6 Hz, 2H), 4.00 (q, *J* = 7.1 Hz, 2H), 2.78 (ddd, *J* = 8.3, 8.3, 13.8 Hz, 1H), 2.59 (s, 3H), 1.57 (ddd, *J* = 3.6, 3.6, 13.9 Hz, 1H), 1.48 (s, 9H), 0.97 (t, *J* = 7.1 Hz, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>, peaks of major rotamer) δ 178.8, 153.8, 153.6, 145.7, 137.5, 131.7, 130.7, 130.4, 129.4, 128.5, 127.7, 127.3, 122.5, 120.8,



119.7, 108.7, 107.9, 80.3, 62.9, 52.2, 38.1, 28.8, 28.4, 27.0, 15.0; HRMS-ES  $[M+NH_4]^+$  calcd for  $C_{27}H_{35}BrN_4O_5$ , 573.1713; found, 573.1730.

**Synthesis of Pentacyclic Aminal 30c.** To a stirred solution of *N*-Boc aniline **29c** (177 mg, 0.319 mmol) in THF (8 mL) was added  $AlH_3 \cdot Me_2NEt$  (0.77 mL, 0.5 M in toluene, 0.385 mmol) dropwise at 0 °C. After 30 min, the temperature was raised to rt. After 2 h, the mixture was quenched with saturated aqueous  $Na_2SO_4$  (0.3 mL) and stirred at rt for 2 h. The mixture was diluted with EtOAc (20 mL), dried over  $Na_2SO_4$  and concentrated *in vacuo*. The residue was purified by flash column chromatography (1:5 to 1:1 EtOAc:hexanes) to give pentacyclic aminal **30c** (116 mg, 67%).  $^1H$  NMR (300 MHz, toluene- $d_8$ , 90 °C)  $\delta$  7.37 (s, 1H), 7.19 (dd,  $J = 1.6, 7.4$  Hz, 1H), 7.10 (dd,  $J = 1.1, 7.8$  Hz, 1H), 6.79 (ddd,  $J = 1.7, 7.5, 7.5$  Hz, 1H), 6.72 (ddd,  $J = 1.4, 7.3, 7.3$  Hz, 1H), 6.46-6.39 (m, 2H), 5.90-5.86 (m, 2H), 4.11 (m, 1H), 4.04 (q,  $J = 7.1$  Hz, 2H), 3.32 (ddd,  $J = 3.4, 10.0, 13.4$  Hz, 1H), 2.84 (s, 3H), 2.28 (ddd,  $J = 4.1, 10.1, 14.1$  Hz, 1H), 1.92 (ddd,  $J = 3.5, 6.1, 14.0$  Hz, 1H), 1.32 (s, 9H), 1.03 (t,  $J = 7.1$  Hz, 3H);  $^{13}C$  NMR (75 MHz, toluene- $d_8$ , 90 °C)  $\delta$  154.5, 153.5, 153.0, 138.7, 134.2, 130.6, 129.8, 126.4, 126.3, 126.2, 125.8, 125.1, 122.6, 118.9, 114.3, 85.5, 80.8, 62.0, 53.0, 40.5, 33.8, 30.4, 28.2, 14.4; HRMS-ES  $[M+H]^+$  calcd for  $C_{27}H_{31}BrN_3O_4$ , 540.1498; found, 540.1484.

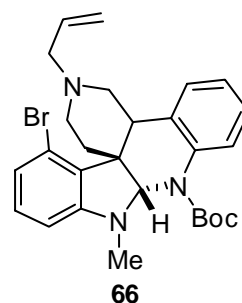
**Preparation of Enamine 40.** Pentacyclic aminal **30c** (38.2 mg, 0.071 mmol) was dissolved in EtOH (2.0 mL) and 1 N aqueous KOH (2.0 mL) and the mixture was refluxed for 2 h. EtOH was removed under reduced pressure. The residue was diluted with EtOAc (20 mL) and saturated aqueous  $NaHCO_3$  (10 mL). The organic layer was dried over  $Na_2SO_4$  and concentrated *in vacuo*. The residue was used for the next step without further purification.  $^1H$  NMR (400 MHz,  $CD_2Cl_2$ )  $\delta$  7.21-7.16 (m, 2H), 7.01-6.96 (m, 2H), 6.77 (t,  $J = 7.9$  Hz, 1H), 6.74 (s, 1H), 6.55 (d,  $J = 7.5$  Hz, 1H), 6.19 (d,  $J = 7.7$  Hz, 1H), 5.80 (s, 1H), 3.89 (br s, 1H), 3.47 (ddd,  $J = 4.6, 4.6, 11.4$

Hz, 1H), 3.24 (ddd,  $J = 3.1, 11.0, 11.0$  Hz, 1H), 2.95 (s, 3H), 2.39 (ddd,  $J = 3.9, 10.3, 13.8$  Hz, 1H), 2.22 (ddd,  $J = 3.2, 4.8, 13.6$  Hz, 1H), 1.51 (s, 9H); LRMS-ES  $[M+H]^+$  calcd for  $C_{24}H_{27}BrN_3O_2$ , 468.1; found, 468.2.

**Allylation of Enamine 40.** The above crude enamine **40** was dissolved in THF (3.0 mL). To the solution was added LDA (0.35 mL, 0.3 M in THF, 0.105 mmol) at  $-78$  °C. After 20 min allyl iodide (0.01 mL, 0.109 mmol) was added, and the mixture was gradually warmed to rt. The mixture was diluted with saturated aqueous  $NaHCO_3$  (10 mL) and EtOAc (25 mL). The organic layer was dried over  $Na_2SO_4$  and concentrated *in vacuo*. The residue was purified by flash column chromatography (1:3 to 1:1 EtOAc:hexanes) to give *C*-allyl product **41** (20.7 mg, 58%) and *N*-allyl enamine **66** (10.5 mg, 29%).

**C-Allyl Product 41:**  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  8.83 (s, 1H), 7.53 (dd,  $J = 3.0, 5.8$  Hz, 1H), 7.02-6.96 (m, 3H), 6.73 (t,  $J = 7.9$  Hz, 1H), 6.57 (d,  $J = 8.0$  Hz, 1H), 6.26 (d,  $J = 7.8$  Hz, 1H), 5.71-5.61 (m, 1H), 5.53 (s, 1H), 5.03-4.93 (m, 2H), 4.04 (dd,  $J = 5.3, 18.7$  Hz, 1H), 3.61 (ddd,  $J = 4.5, 4.5, 12.5$  Hz, 1H), 2.98 (s, 3H), 2.67-2.62 (m, 2H), 2.42 (ddd,  $J = 6.0, 13.3, 13.3$  Hz, 1H), 1.93 (dd,  $J = 5.4, 13.7$  Hz, 1H), 1.46 (s, 9H);  $^{13}C$  NMR (75 MHz,  $CDCl_3$ )  $\delta$  166.1, 154.9, 153.2, 139.6, 133.0, 132.5, 129.7, 127.2, 126.5, 125.0, 124.9, 123.9, 119.5, 118.9, 106.1, 83.8, 81.9, 55.7, 47.4, 43.0, 42.1, 32.1, 29.0, 28.6; HRMS-ES  $[M+H]^+$  calcd for  $C_{27}H_{31}BrN_3O_2$ , 508.1600; found, 508.1601.

**N-Allyl Product 66:**  $^1H$  NMR (400 MHz,  $CD_2Cl_2$ )  $\delta$  7.19-7.15 (m, 2H), 7.00-6.96 (m, 2H), 6.76 (t,  $J = 7.9$  Hz, 1H), 6.55 (s, 1H), 6.54 (d,  $J = 8.7$  Hz, 1H), 6.19 (d,  $J = 7.7$  Hz, 1H), 6.00-5.91 (m, 1H), 5.77 (s, 1H), 5.36-5.22 (m, 2H), 3.69 (d,  $J = 6.0$  Hz, 2H), 3.33 (ddd,  $J = 4.5, 4.5, 11.4$  Hz, 1H), 2.99 (ddd,  $J = 3.1, 11.2, 11.2$  Hz, 1H), 2.95 (s, 3H), 2.43 (ddd,  $J = 3.5, 10.4,$



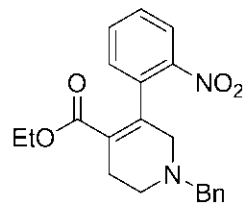
13.9 Hz, 1H), 2.23 (ddd,  $J = 3.2, 4.7, 13.6$  Hz, 1H), 1.51 (s, 9H); LRMS-ES  $[M+H]^+$  calcd for  $C_{27}H_{31}BrN_3O_2$ , 508.2; found, 508.3.

**Formation of  $\alpha$ -Ethoxy Carbamate 42.** To a stirred solution of imine **41** (20.7 mg, 0.041 mmol) in EtOH (1.0 mL) was added diethyl pyrocarbonate (7.2  $\mu$ L, 0.049 mmol) at rt. After 10 min the solvent was removed under reduced pressure. The crude residue was used for the next step without further purification.  $^1H$  NMR (400 MHz,  $CD_2Cl_2$ )  $\delta$  7.44-7.42 (m, 1H), 7.11 (s, 1H), 7.09 (s, 1H), 7.06-7.00 (m, 2H), 6.72 (t,  $J = 7.8$  Hz, 1H), 6.60 (d,  $J = 8.0$  Hz, 1H), 6.23 (d,  $J = 7.3$  Hz, 1H), 5.75 (s, 1H), 5.61-5.51 (m, 1H), 4.80 (d,  $J = 17.0$  Hz, 1H), 4.67 (d,  $J = 10.0$  Hz, 1H), 4.27-4.15 (m, 3H), 3.98-3.92 (m, 2H), 3.73 (ddd,  $J = 5.3, 8.2, 13.5$  Hz, 1H), 3.24 (dd,  $J = 5.5, 15.0$  Hz 1H), 2.95 (s, 3H), 2.65 (dd,  $J = 7.5, 14.9$  Hz, 1H), 2.37 (ddd,  $J = 5.3, 8.6, 13.9$  Hz, 1H), 1.88 (ddd,  $J = 5.2, 6.8, 14.1$  Hz, 1H), 1.51 (s, 9H), 1.33 (t,  $J = 7.1$  Hz, 3H), 1.26 (t,  $J = 7.0$  Hz, 3H); LRMS-ES  $[M+H]^+$  calcd for  $C_{32}H_{41}BrN_3O_5$ , 626.2; found, 626.4.

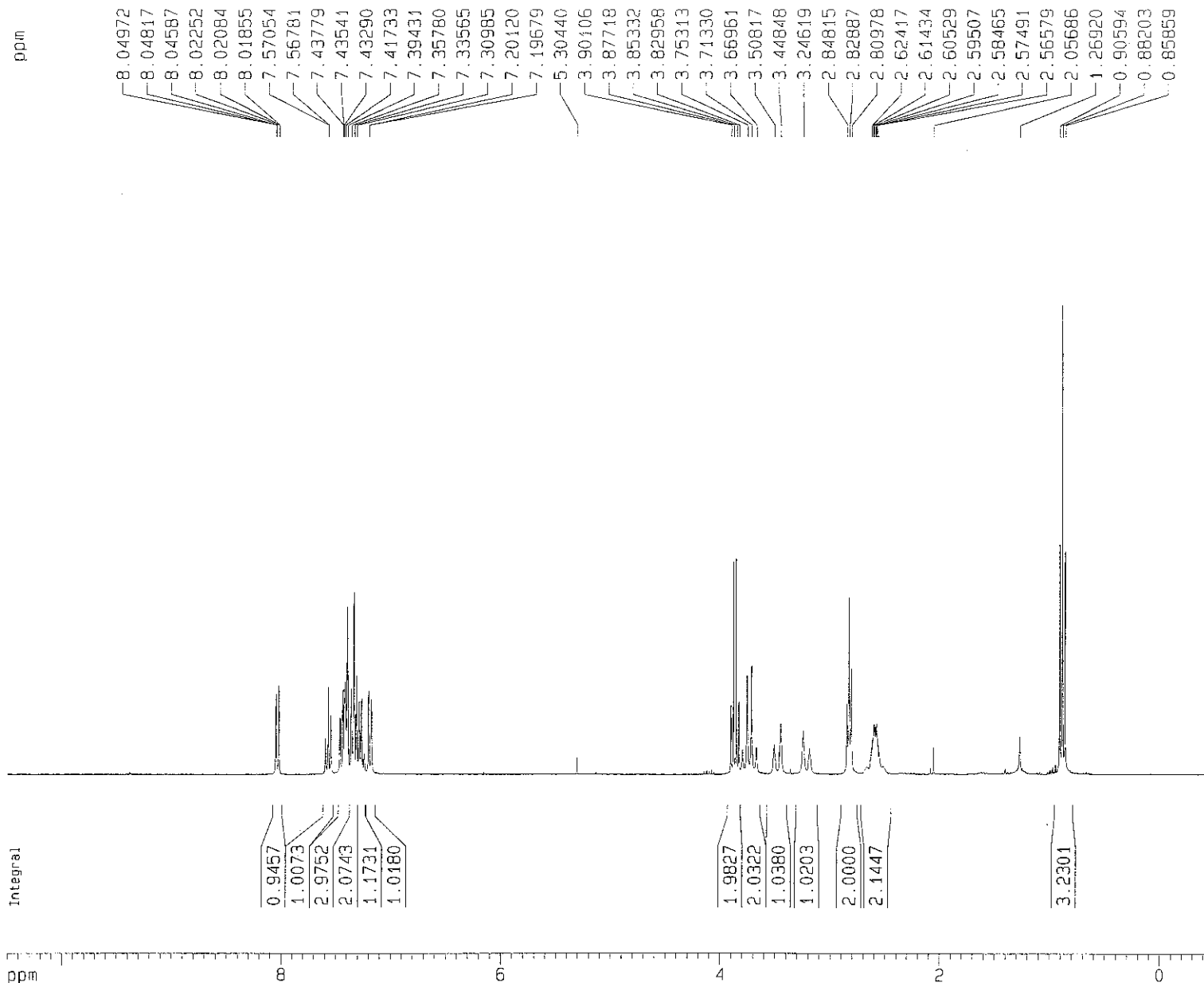
**Synthesis of Hexacyclic Acetal 43.** To a solution of the above crude *N,O*-acetal **42** in dioxane (2.0 mL) and  $H_2O$  (1.0 mL) was added NMO (47.7 mg, 0.407 mmol) at 0  $^\circ C$ , followed by  $OsO_4$  (0.01 mL, 4 wt% in  $H_2O$ ). The mixture was gradually warmed to rt. The mixture was stirred at rt for 3 h and then diluted with saturated aqueous  $Na_2S_2O_3$  (10 mL) and EtOAc (20 mL). The organic layer was dried over  $Na_2SO_4$  and concentrated *in vacuo*. The crude residue was dissolved in THF (2.0 mL) and  $H_2O$  (1.0 mL) and  $NaIO_4$  (87.0 mg, 0.407 mmol) was added. The mixture was stirred at rt for 1 h and then diluted with saturated aqueous  $NaHCO_3$  (10 mL) and EtOAc (20 mL). The organic layer was dried over  $Na_2SO_4$  and concentrated *in vacuo*. The residue was purified by flash column chromatography (1:3 EtOAc:hexanes) to give two separable diastereomers of hexacyclic acetal **43** (7.7 mg and 2.9 mg, 42% for 3 steps). More polar major diastereomer of **43**:  $^1H$  NMR (400 MHz,  $CD_2Cl_2$ )  $\delta$  7.59 (dd,  $J = 3.6, 5.6$  Hz, 1H),

7.05-7.02 (m, 3H), 6.99 (s, 1H), 6.77 (t,  $J = 7.9$  Hz, 1H), 6.58 (d,  $J = 8.0$  Hz, 1H), 6.30 (d,  $J = 7.4$  Hz, 1H), 5.55 (s, 1H), 5.26 (dd,  $J = 1.1, 5.3$  Hz, 1H), 4.25 (q,  $J = 7.1$  Hz, 2H), 3.73 (ddd,  $J = 7.1, 9.7, 14.2$  Hz, 1H), 3.60 (ddd,  $J = 4.4, 6.1, 13.0$  Hz, 1H), 3.48-3.41 (m, 2H), 2.99 (s, 3H), 2.88 (dd,  $J = 5.4, 13.0$  Hz, 1H), 2.46 (ddd,  $J = 4.9, 9.4, 16.9$  Hz, 1H), 2.06 (ddd,  $J = 4.3, 6.1, 14.2$  Hz, 1H), 1.98 (dd,  $J = 1.2, 13.0$  Hz, 1H), 1.51 (s, 9H), 1.33 (t,  $J = 7.1$  Hz, 3H), 1.13 (t,  $J = 7.1$  Hz, 3H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  157.4, 154.8, 153.5, 138.8, 137.3, 131.1, 129.8, 126.9, 126.2, 124.8, 124.3, 124.2, 118.7, 106.2, 101.5, 85.6, 84.4, 82.0, 62.9, 62.2, 57.1, 48.1, 44.4, 39.8, 32.1, 29.5, 28.6, 15.6, 15.3; HRMS-ES  $[\text{M}+\text{H}]^+$  calcd for  $\text{C}_{31}\text{H}_{39}\text{BrN}_3\text{O}_6$ , 628.2022; found, 628.1996.

Less polar minor diastereomer of **43**:  $^1\text{H}$  NMR (300 MHz,  $\text{CD}_2\text{Cl}_2$ )  $\delta$  7.48 (dd,  $J = 3.9, 5.3$  Hz, 1H), 7.04 (s, 1H), 7.03-6.99 (m, 3H), 6.70 (t,  $J = 7.9$  Hz, 1H), 6.56 (d,  $J = 7.3$  Hz, 1H), 6.20 (d,  $J = 7.6$  Hz, 1H), 5.61 (br s, 1H), 5.17 (dd,  $J = 1.8, 6.2$  Hz, 1H), 4.38-4.24 (m, 2H), 3.86-3.71 (m, 2H), 3.60-3.42 (m, 2H), 2.95 (s, 3H), 2.84-2.71 (m, 2H), 2.35-2.31 (m, 1H), 2.04 (ddd,  $J = 5.6, 5.6, 14.1$  Hz, 1H), 1.48 (s, 9H), 1.33 (t,  $J = 7.1$  Hz, 3H), 1.20 (t,  $J = 7.1$  Hz, 3H); LRMS-ES  $[\text{M}+\text{H}]^+$  calcd for  $\text{C}_{31}\text{H}_{39}\text{BrN}_3\text{O}_6$ , 628.2; found, 628.4.



20



## Current Data Parameters

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PROCNO 1

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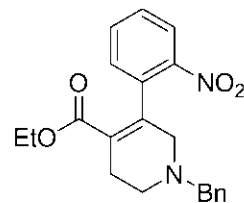
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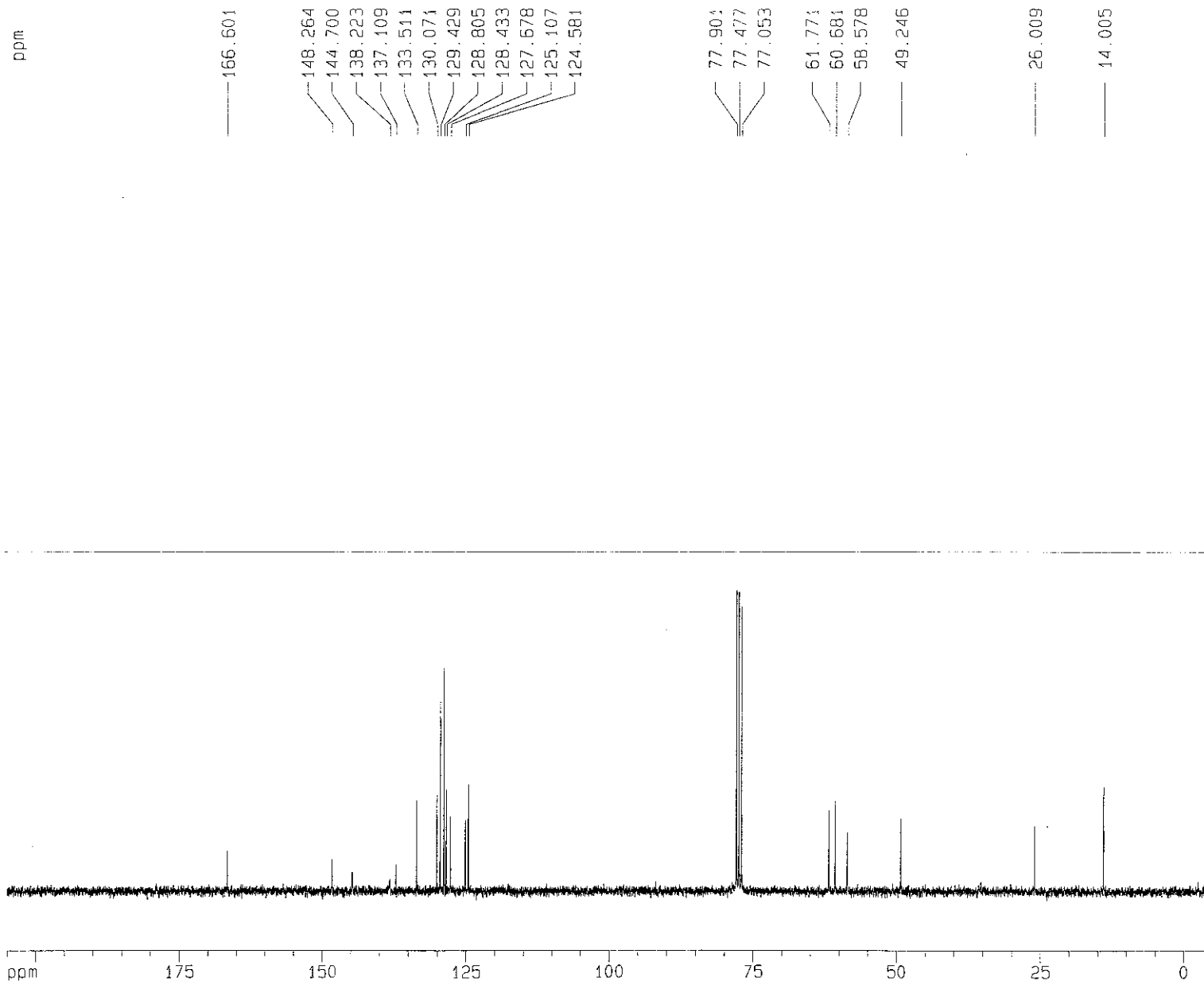
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20



## Current Data Parameters

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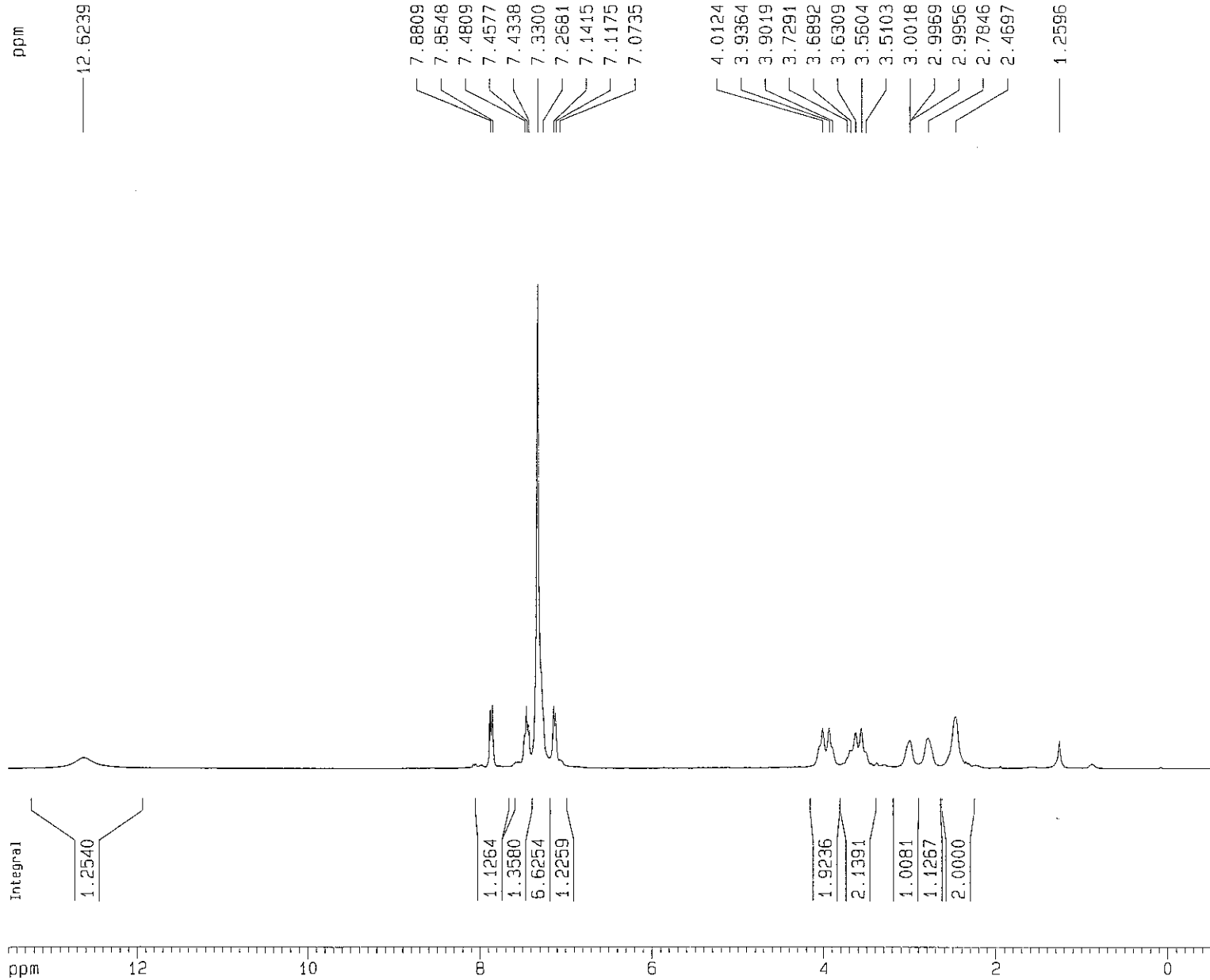
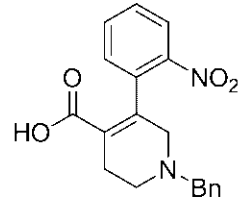
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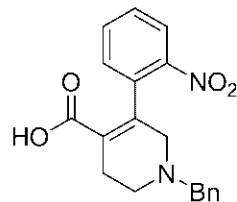
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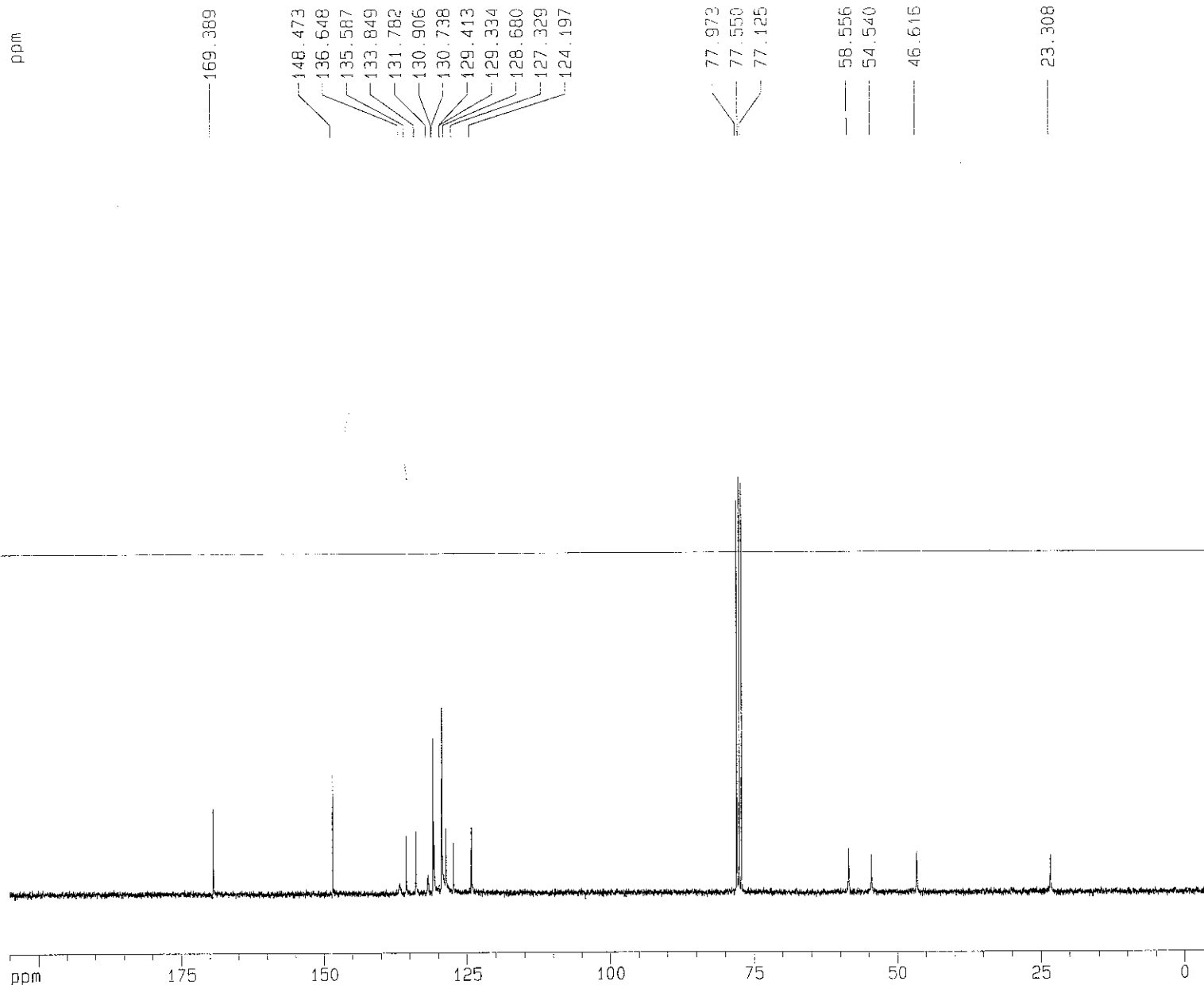
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21



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D11 0.0300000 sec  
D12 0.0000200 sec

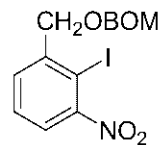
===== CHANNEL f1 =====  
NUC1 13C  
P1 5.40 usec  
PL1 -6.00 dB  
SF01 75.4106357 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 115.00 usec  
PL2 0.00 dB  
PL12 20.00 dB  
PL13 20.00 dB  
SF02 299.6711995 MHz

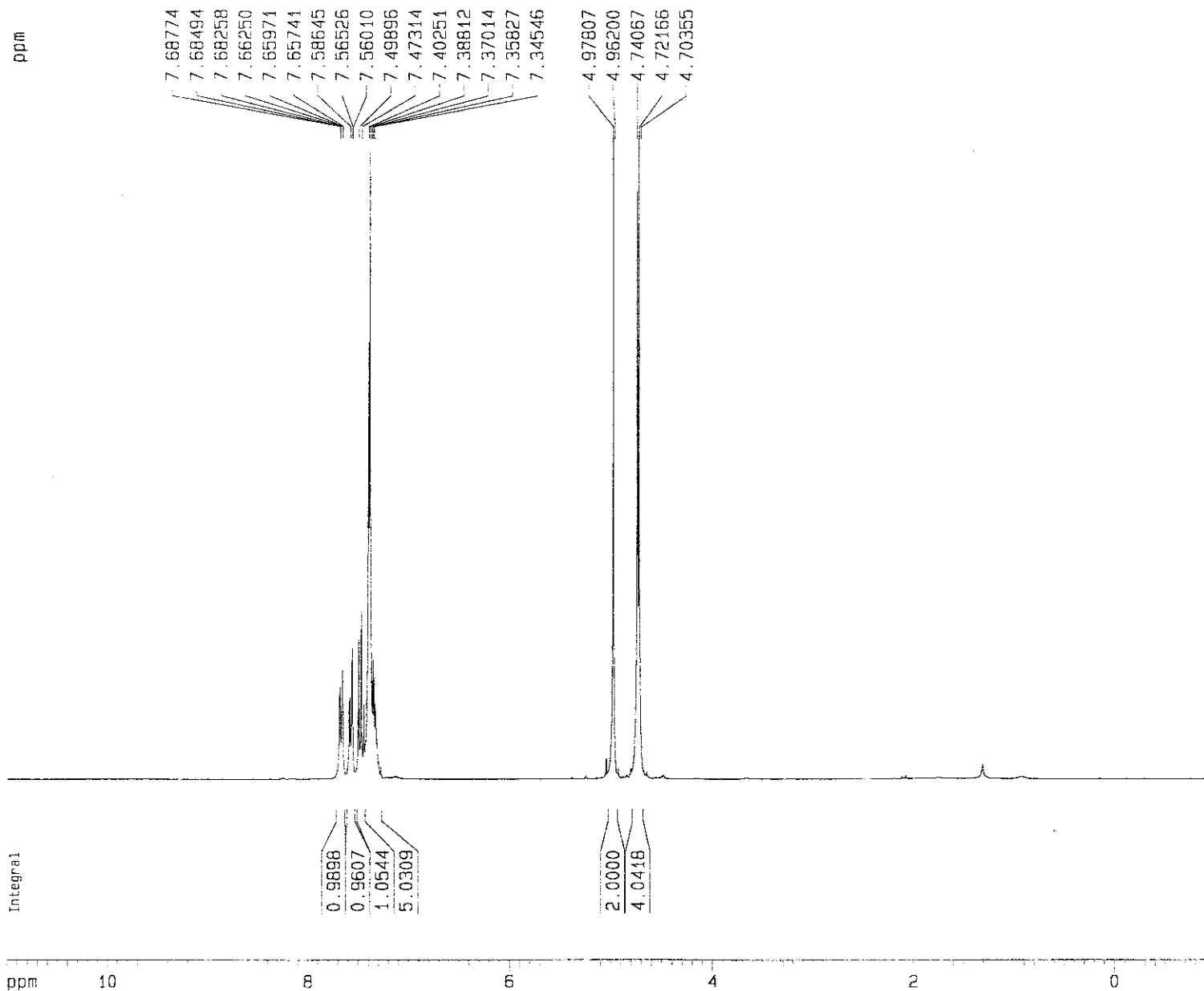
F2 - Processing parameters  
SI 32768  
SF 75.4023410 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

1D NMR plot parameters  
CX 20.00 cm  
F1P 205.000 ppm  
F1 15457.48 Hz  
F2P -5.000 ppm  
F2 -377.01 Hz  
PPMCM 10.50000 ppm/cm  
HZCM 791.72461 Hz/cm





17a



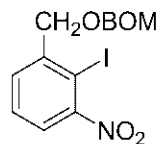
Current Data Parameters  
NAME PL-JUN09-09  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20090609  
Time 11.58  
INSTRUM spect  
PROBHD 5 mm Multinu  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6172.839 Hz  
FIDRES 0.094190 Hz  
AQ 5.3084660 sec  
RG 114  
DW 81.000 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec

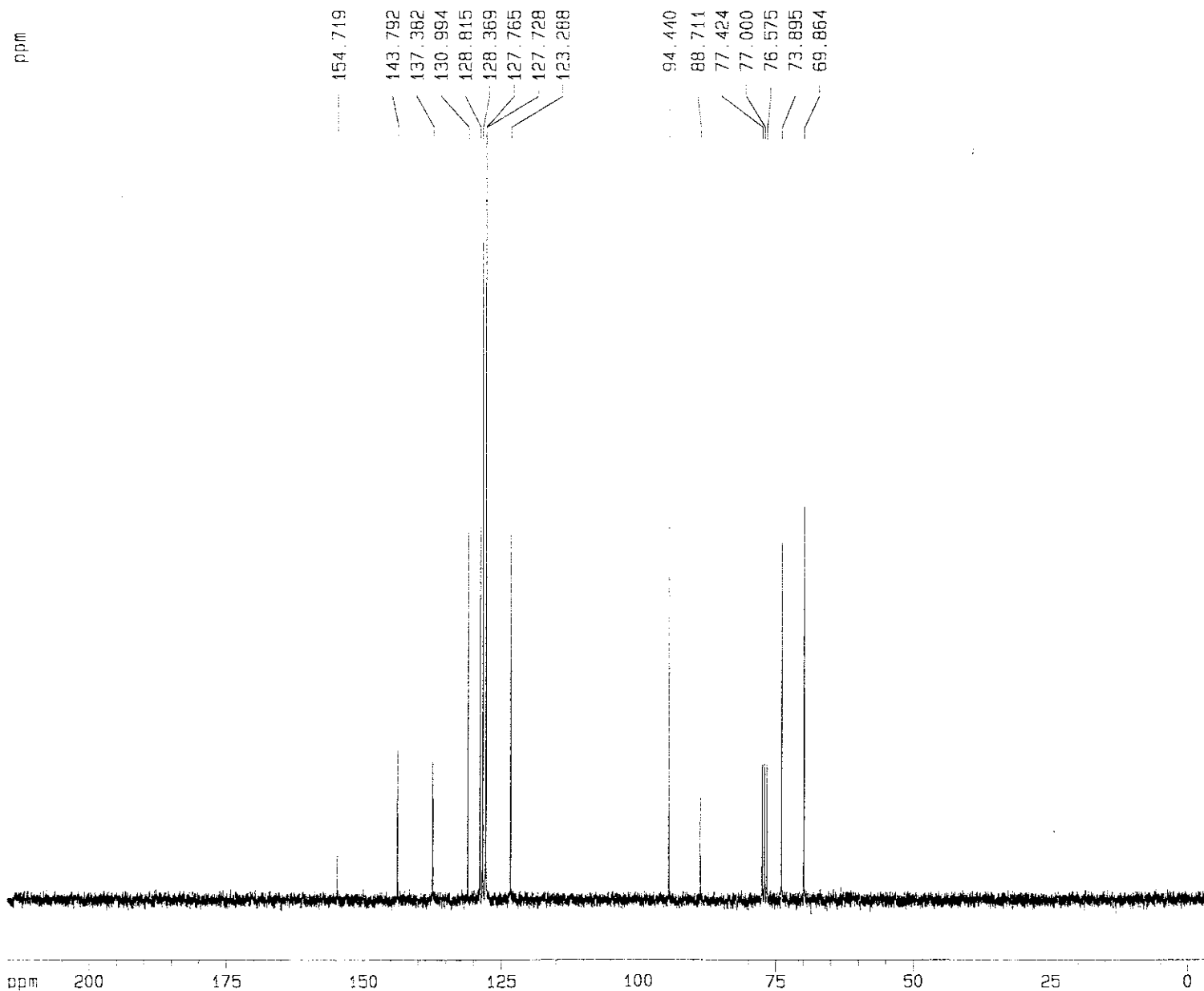
===== CHANNEL f1 =====  
NUC1 1H  
P1 9.50 usec  
PL1 -5.00 dB  
SF01 300.1318534 MHz

F2 - Processing parameters  
SI 32768  
SF 300.1300000 MHz  
WDW nc  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

1D NMR plot parameters  
CX 20.00 cm  
F1P 11.000 ppm  
F1 3301.43 Hz  
F2P -1.000 ppm  
F2 -300.13 Hz  
PPMCM 0.60000 ppm/cm  
HZCM 180.07800 Hz/cm



17a



Current Data Parameters  
NAME PL-0009-09  
EXPNO 2  
PROCNO 1

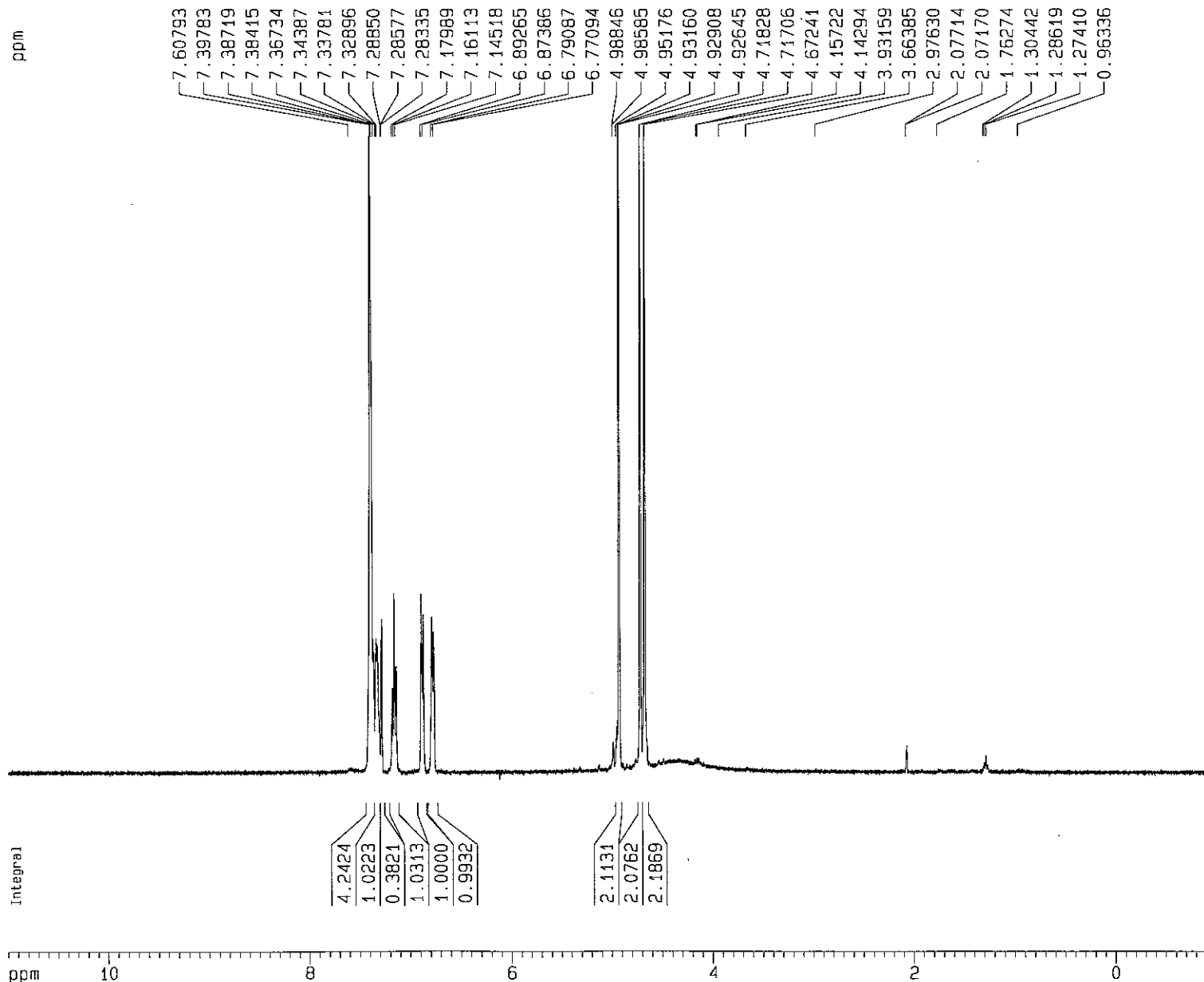
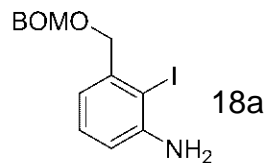
F2 - Acquisition Parameters  
Date\_ 20090509  
Time 12.04  
INSTRUM spect  
PROBHD 5 mm Multinu  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 52  
DS 4  
SWH 10632.393 Hz  
FIDRES 0.287360 Hz  
AQ 1.7400308 sec  
RG 16384  
DW 26.550 usec  
DE 6.00 usec  
TE 300.0 K  
D1 2.0000000 sec  
d11 0.0300000 sec  
d12 0.0000200 sec

===== CHANNEL f1 =====  
NUC1 13C  
P1 11.80 usec  
PL1 0.00 dB  
SFO1 75.4760200 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 110.00 usec  
PL2 0.00 dB  
PL12 17.50 dB  
PL13 17.50 dB  
SFO2 300.1312005 MHz

F2 - Processing parameters  
SI 32768  
SF 75.4677635 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

1D NMR plot parameters  
CX 20.00 cm  
F1P 215.000 ppm  
F1 15225.57 Hz  
F2P -5.000 ppm  
F2 -377.34 Hz  
PRGCM 11 00000 ppm/cm  
HZCM 930.14545 Hz/cm



## Current Data Parameters

NAME PL-Jun17-09  
EXPNO 1  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20090617  
Time 17.59  
INSTRUM spect  
PROBHD 5 mm BBI 1H-B  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8278.146 Hz  
FIDRES 0.126314 Hz  
AQ 3.9584243 sec  
RG 645.1  
DW 60.400 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec

## ===== CHANNEL f1 =====

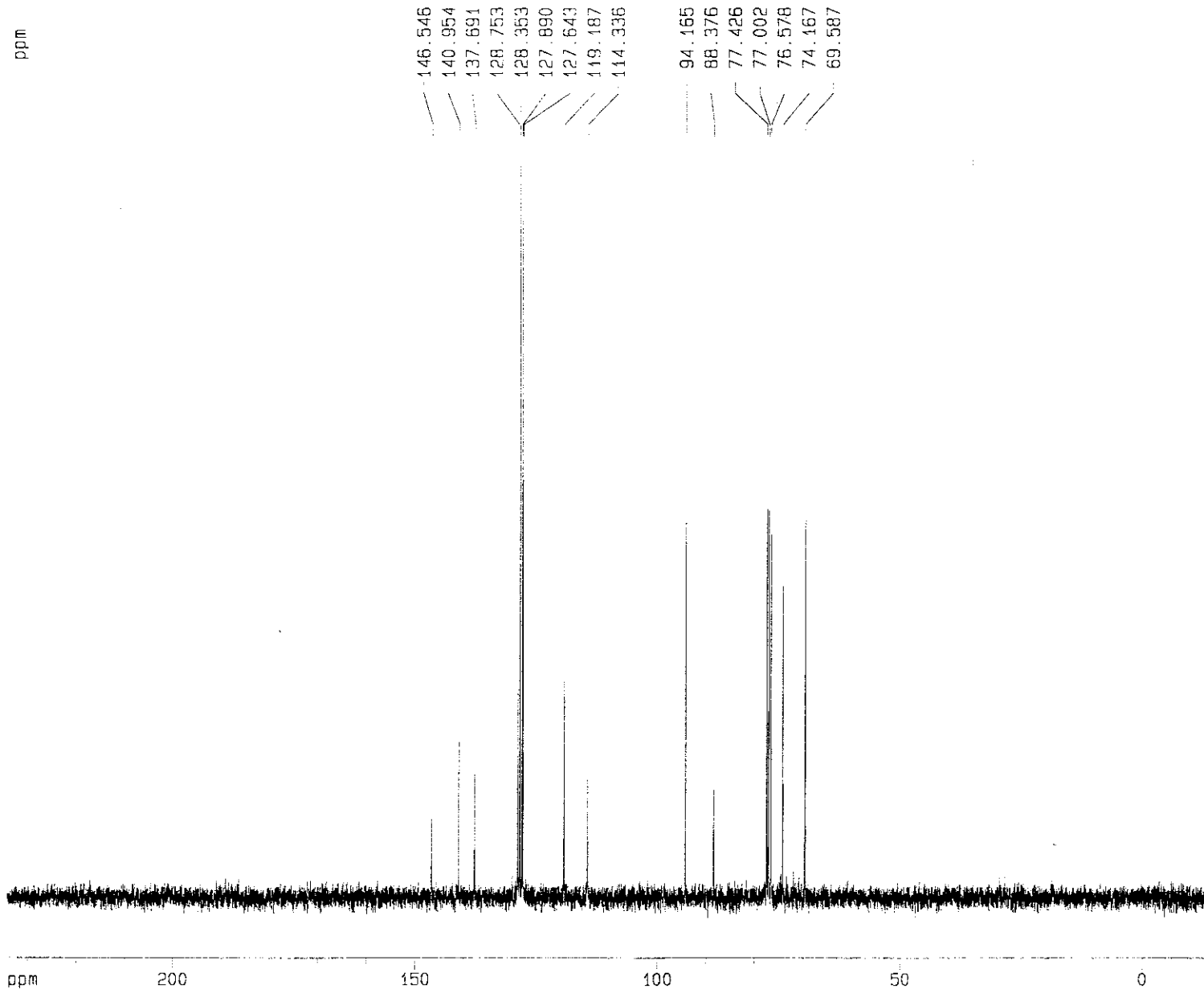
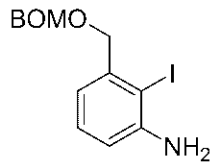
NUC1 1H  
P1 6.45 usec  
PL1 0.00 dB  
SF01 400.1324710 MHz

## F2 - Processing parameters

SI 32768  
SF 400.1300000 MHz  
WDW no  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

## 1D NMR plot parameters

CX 20.00 cm  
F1P 11.000 ppm  
F1 4401.43 Hz  
F2P -1.000 ppm  
F2 -400.13 Hz  
PPMCM 0.60000 ppm/cm  
HZCM 240.07800 Hz/cm



Current Data Parameters  
NAME PL-Jun10-09  
EXPNO 2  
PROCNO 1

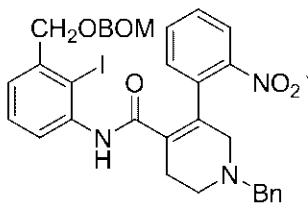
F2 - Acquisition Parameters  
Date\_ 20090510  
Time 15.31  
INSTRUM spect  
PROBHD 5 mm Multinu  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 101  
DS 4  
SWH 18832.393 Hz  
FIDRES 0.287360 Hz  
AQ 1.7400308 sec  
RG 13004  
DW 26.550 usec  
DE 6.00 usec  
TE 300.0 K  
D1 2.0000000 sec  
d11 0.0300000 sec  
d12 0.0000200 sec

===== CHANNEL f1 =====  
NUC1 13C  
P1 11.80 usec  
PL1 0.00 dB  
SFO1 75.4760200 MHz

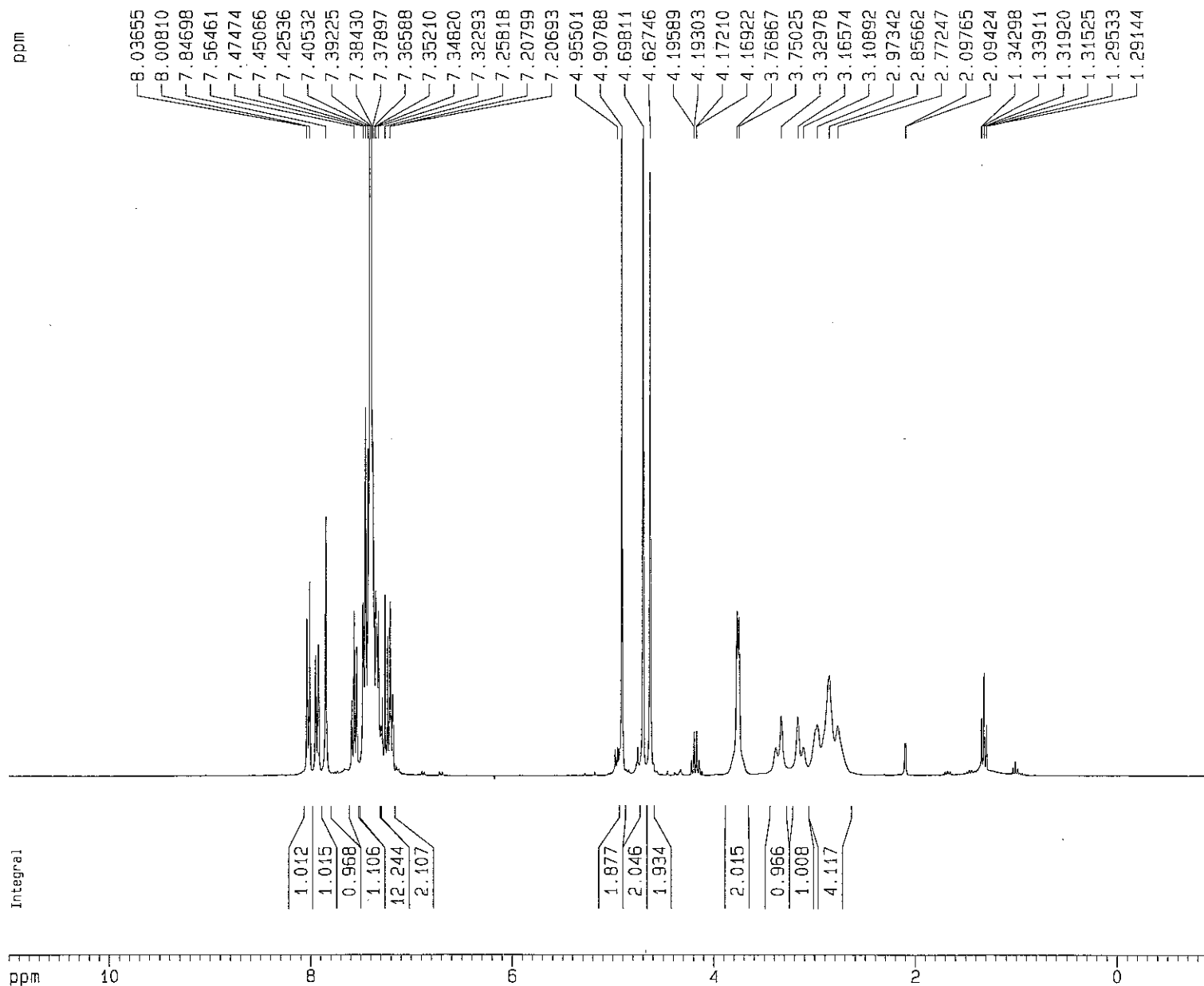
===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 110.00 usec  
PL2 0.00 dB  
PL12 17.50 dB  
PL13 17.50 dB  
SFO2 300.1312005 MHz

F2 - Processing parameters  
SI 32768  
SF 75.4677506 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

1D NMR plot parameters  
CX 20.00 cm  
F1P 234.213 ppm  
F1 17675.52 Hz  
F2P -15.329 ppm  
F2 -1156.88 Hz  
PPMCM 12.47711 ppm/cm  
HZCM 941.61963 Hz/cm



23a



## Current Data Parameters

NAME PL-Jun27-09  
EXPNO 1  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20090627  
Time 14.18  
INSTRUM spect  
PROBHD 5 mm GNP 1H/1  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6172.839 Hz  
FIDRES 0.094190 Hz  
AQ 5.3084660 sec  
RG 35.9  
DW 81.000 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec

## ===== CHANNEL f1 =====

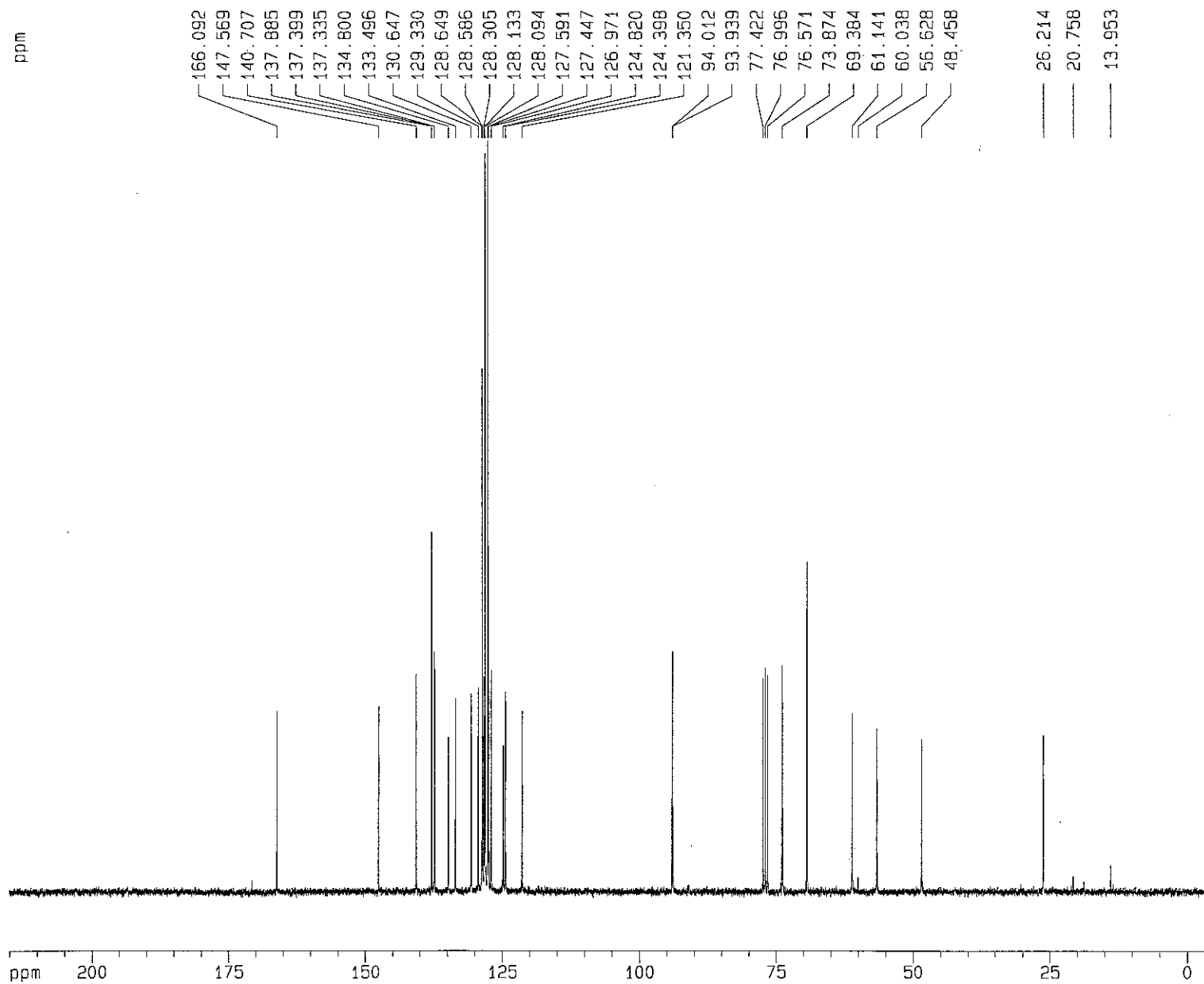
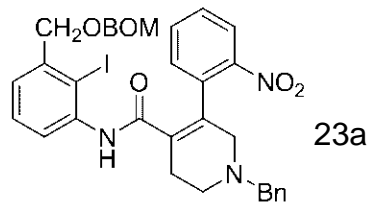
NUC1 1H  
P1 11.70 usec  
PL1 0.00 dB  
SFO1 299.8718518 MHz

## F2 - Processing parameters

SI 32768  
SF 299.870000 MHz  
WDW no  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

## 1D NMR plot parameters

CX 20.00 cm  
F1P 11.000 ppm  
F1 3298.57 Hz  
F2P -1.000 ppm  
F2 -299.87 Hz  
PPMCM 0.60000 ppm/cm  
HZCM 179.92200 Hz/cm



Current Data Parameters  
NAME PL-Jun27-09  
EXPNO 2  
PROCNO 1

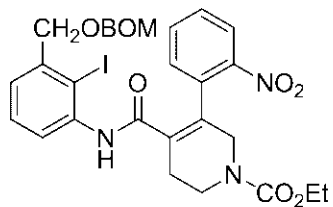
F2 - Acquisition Parameters  
Date\_ 20090627  
Time 14.27  
INSTRUM spect  
PROBHD 5 mm GNP 1H/1  
PULPROG zgpg30  
TD 65536  
SOLVENT CDC13  
NS 154  
DS 4  
SWH 18796.992 Hz  
FIDRES 0.286819 Hz  
AQ 1.7433076 sec  
RG 1024  
DW 26.600 usec  
DE 6.00 usec  
TE 300.0 K  
D1 2.0000000 sec  
D11 0.0300000 sec  
D12 0.0000200 sec

===== CHANNEL f1 =====  
NUC1 13C  
P1 5.40 usec  
PL1 -6.00 dB  
SF01 75.4106357 MHz

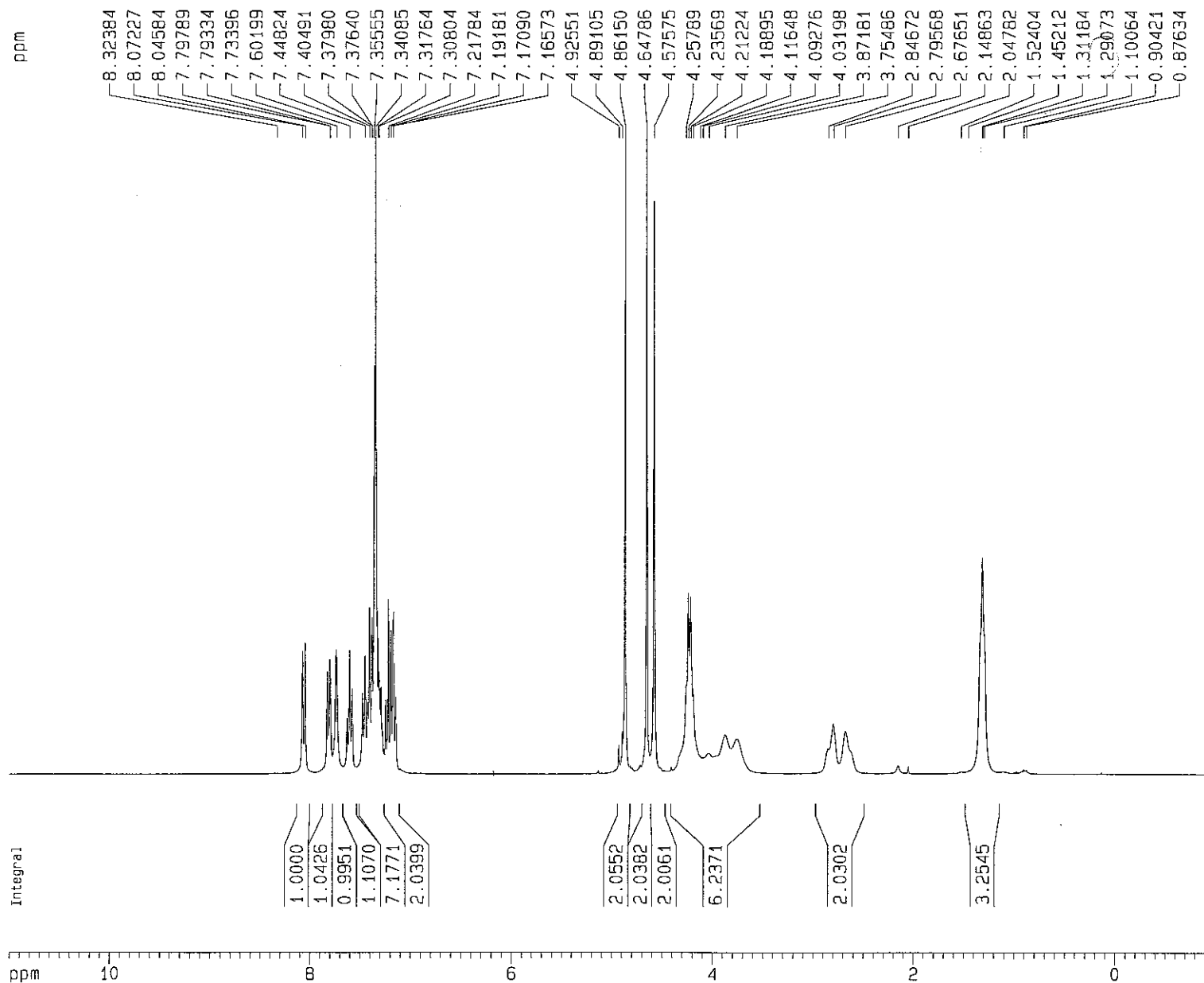
===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 115.00 usec  
PL2 0.00 dB  
PL12 20.00 dB  
PL13 20.00 dB  
SF02 299.8711995 MHz

F2 - Processing parameters  
SI 32768  
SF 75.4024044 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

1D NMR plot parameters  
CX 20.00 cm  
F1P 215.000 ppm  
F1 16211.52 Hz  
F2P -5.000 ppm  
F2 -377.01 Hz  
PPMCM 11.00000 ppm/cm  
HZCM 829.42645 Hz/cm



24a



## Current Data Parameters

NAME PL-Jun29-09  
EXPNO 1  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20090629  
Time 13.51  
INSTRUM spect  
PROBHD 5 mm QNP 1H/1  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6172.839 Hz  
FIDRES 0.094190 Hz  
AQ 5.3084660 sec  
RG 45.3  
DW 81.000 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec

## ===== CHANNEL f1 =====

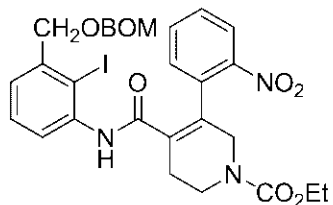
NUC1 1H  
P1 11.70 usec  
PL1 0.00 dB  
SF01 299.6718518 MHz

## F2 - Processing parameters

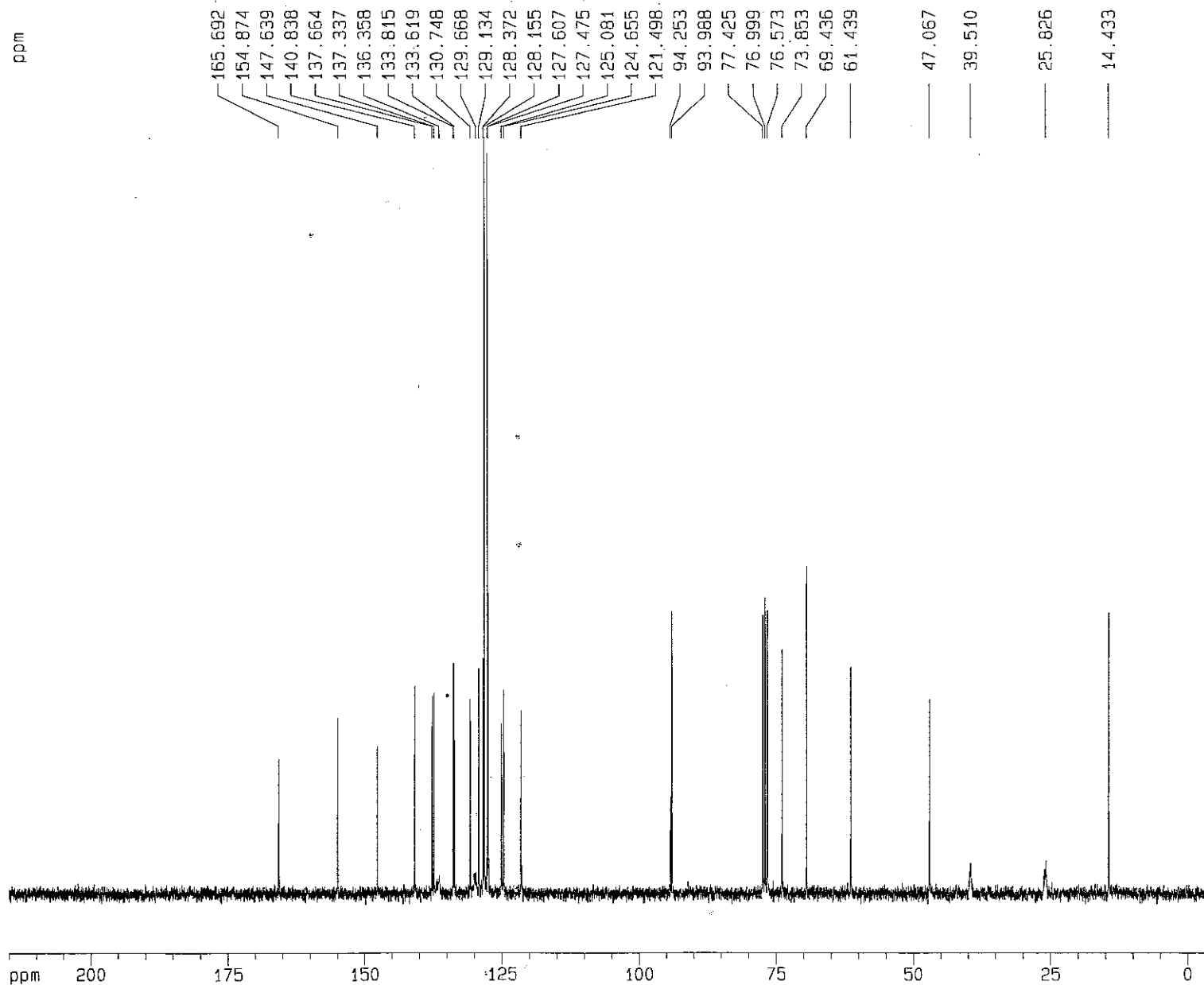
SI 32768  
SF 299.6700000 MHz  
WDW no  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

## 1D NMR plot parameters

CX 20.00 cm  
F1P 11.000 ppm  
F1 3296.57 Hz  
F2P -1.000 ppm  
F2 -299.87 Hz  
PPMCM 0.60000 ppm/cm  
HZCM 179.92200 Hz/cm



24a



Current Data Parameters  
 NAME PL-Jun29-09  
 EXPNO 2  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20090629  
 Time 13.58  
 INSTRUM spect  
 PROBHD 5 mm QNP 1H/1  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 83  
 DS 4  
 SWH 18796.992 Hz  
 FIDRES 0.286819 Hz  
 AQ 1.7433076 sec  
 RG 1024  
 DW 26.600 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 2.0000000 sec  
 D11 0.0300000 sec  
 D12 0.0002000 sec

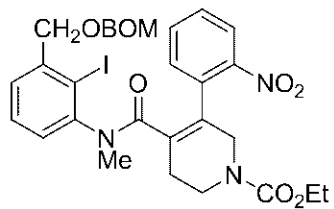
===== CHANNEL f1 =====  
 NUC1 13C  
 P1 5.40 usec  
 PL1 -6.00 dB  
 SF01 75.4106357 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 115.00 usec  
 PL2 0.00 dB  
 PL12 20.00 dB  
 PL13 20.00 dB  
 SF02 299.8711995 MHz

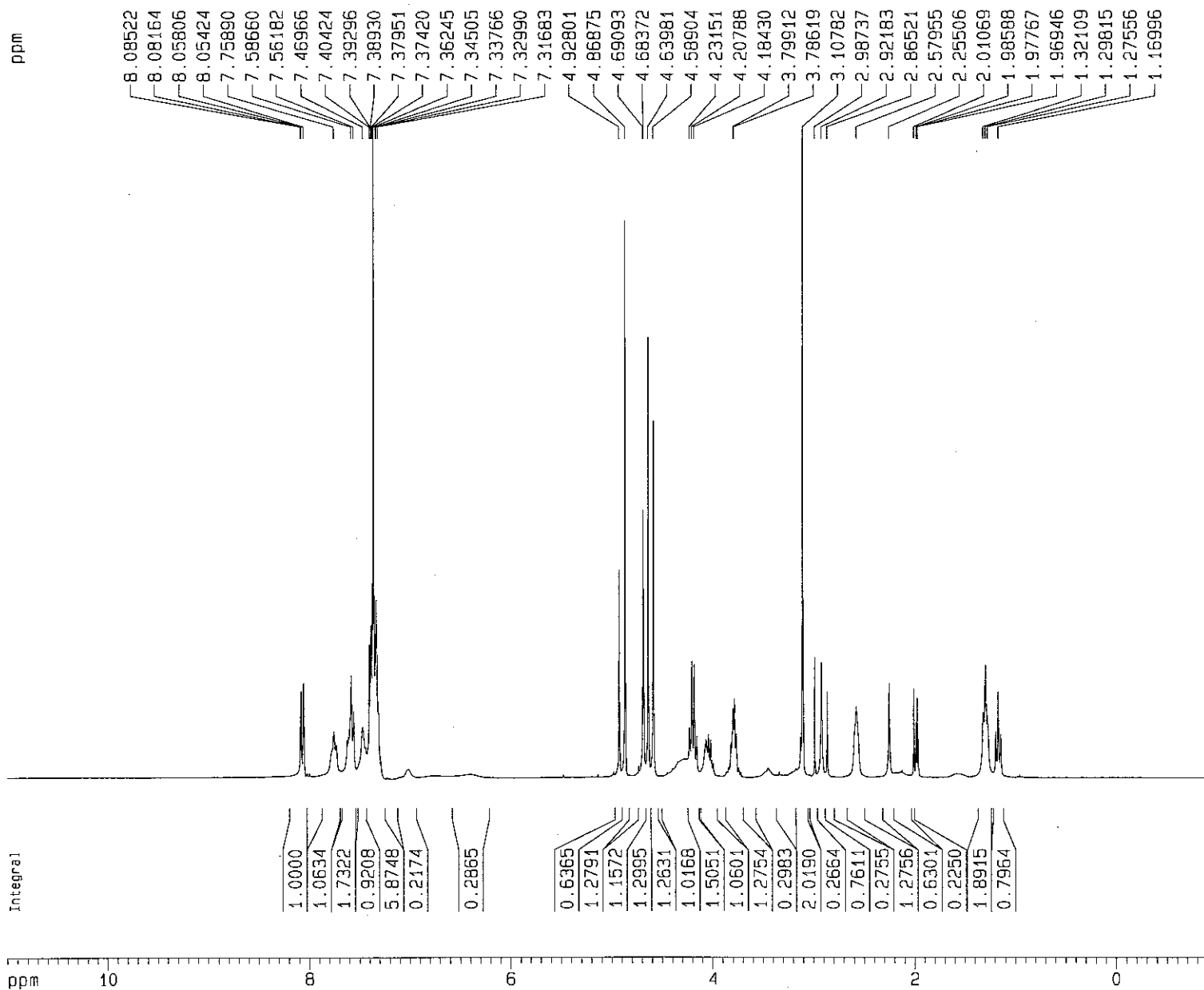
F2 - Processing parameters  
 SI 32768  
 SF 75.4023975 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 215.000 ppm  
 F1 16211.52 Hz  
 F2P -5.000 ppm  
 F2 -377.01 Hz  
 PPMCM 11.00000 ppm/cm  
 HZCM 829.42639 Hz/cm





25a

in CD<sub>3</sub>CN at rt

## Current Data Parameters

NAME PL-Jul15-09  
EXPNO 3  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20090715  
Time 12.54  
INSTRUM spect  
PROBHD 5 mm QNP 1H/1  
PULPROG zg30  
TD 65536  
SOLVENT CD3CN  
NS 16  
DS 2  
SWH 6172.839 Hz  
FIDRES 0.094190 Hz  
AQ 5.3084660 sec  
RG 101.6  
DW 81.000 usec  
DE 6.000 usec  
TE 300.0 K  
D1 1.00000000 sec

## ===== CHANNEL f1 =====

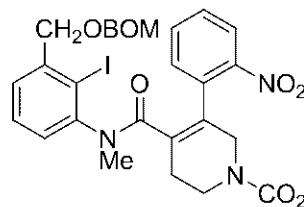
NUC1 1H  
P1 11.70 usec  
PL1 0.00 dB  
SFO1 299.8718518 MHz

## F2 - Processing parameters

SI 32768  
SF 299.8700000 MHz  
WDW no  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

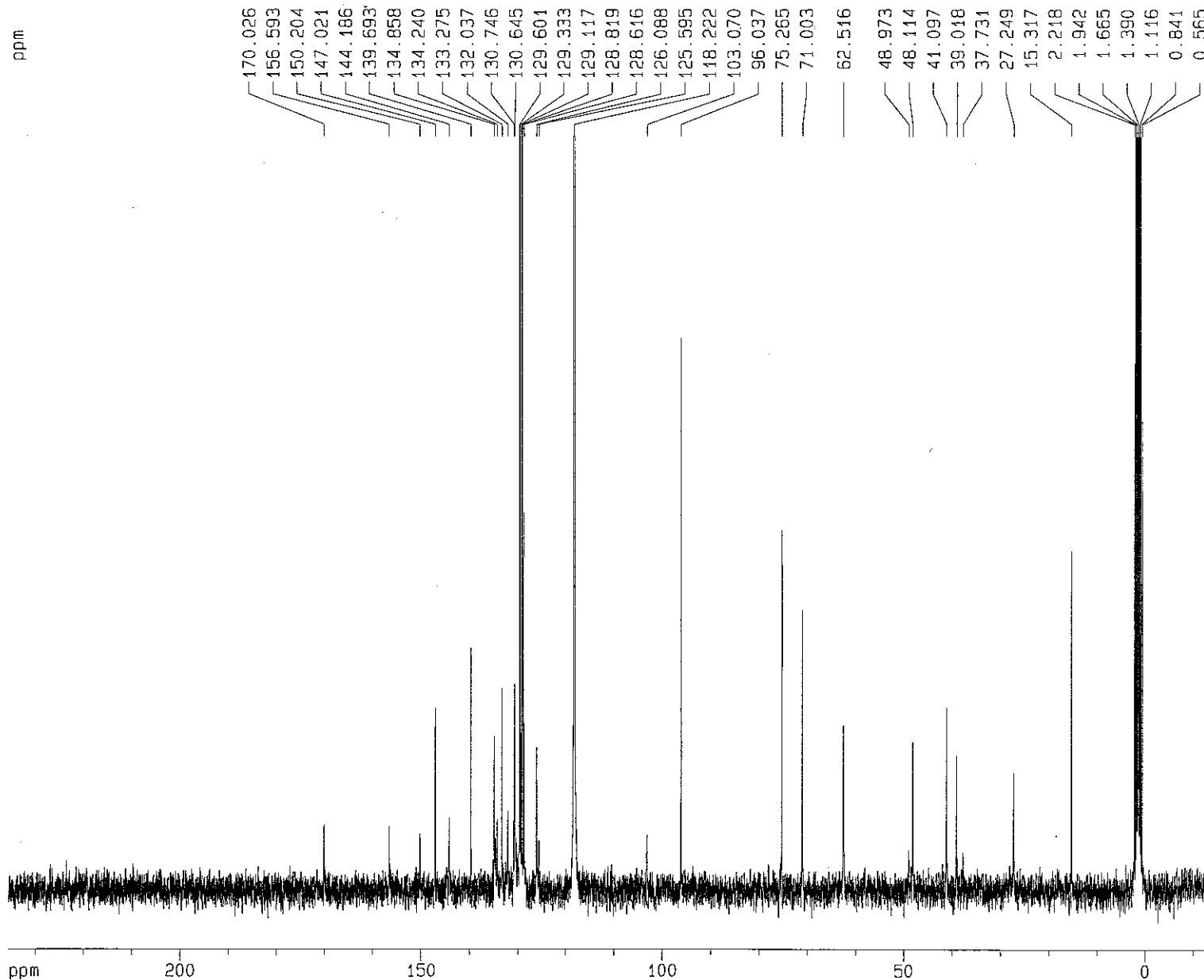
## 1D NMR plot parameters

CX 20.00 cm  
F1P 11.000 ppm  
F1 3298.57 Hz  
F2P -1.000 ppm  
F2 -299.87 Hz  
PPMCM 0.60000 ppm/cm  
HZCM 179.92200 Hz/cm



25a

in CD3CN at 65 degree



## Current Data Parameters

NAME PL-Jul15-09  
EXPNO 5  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20090715  
Time 13.20  
INSTRUM spect  
PROBHD 5 mm GNP 1H/1  
PULPROG zgpg30  
TD 65536  
SOLVENT CD3CN  
NS 636  
DS 4  
SWH 18796.992 Hz  
FIDRES 0.286819 Hz  
AQ 1.7433076 sec  
RG 512  
DW 26.600 usec  
DE 6.00 usec  
TE 300.0 K  
D1 2.0000000 sec  
D11 0.0300000 sec  
D12 0.0000200 sec

## ===== CHANNEL f1 =====

NUC1 13C  
P1 5.40 usec  
PL1 -6.00 dB  
SFO1 75.4106357 MHz

## ===== CHANNEL f2 =====

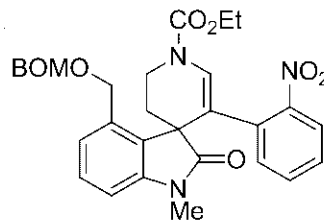
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 115.00 usec  
PL2 0.00 dB  
PL12 20.00 dB  
PL13 20.00 dB  
SFO2 299.8711995 MHz

## F2 - Processing parameters

SI 32768  
SF 75.4022751 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

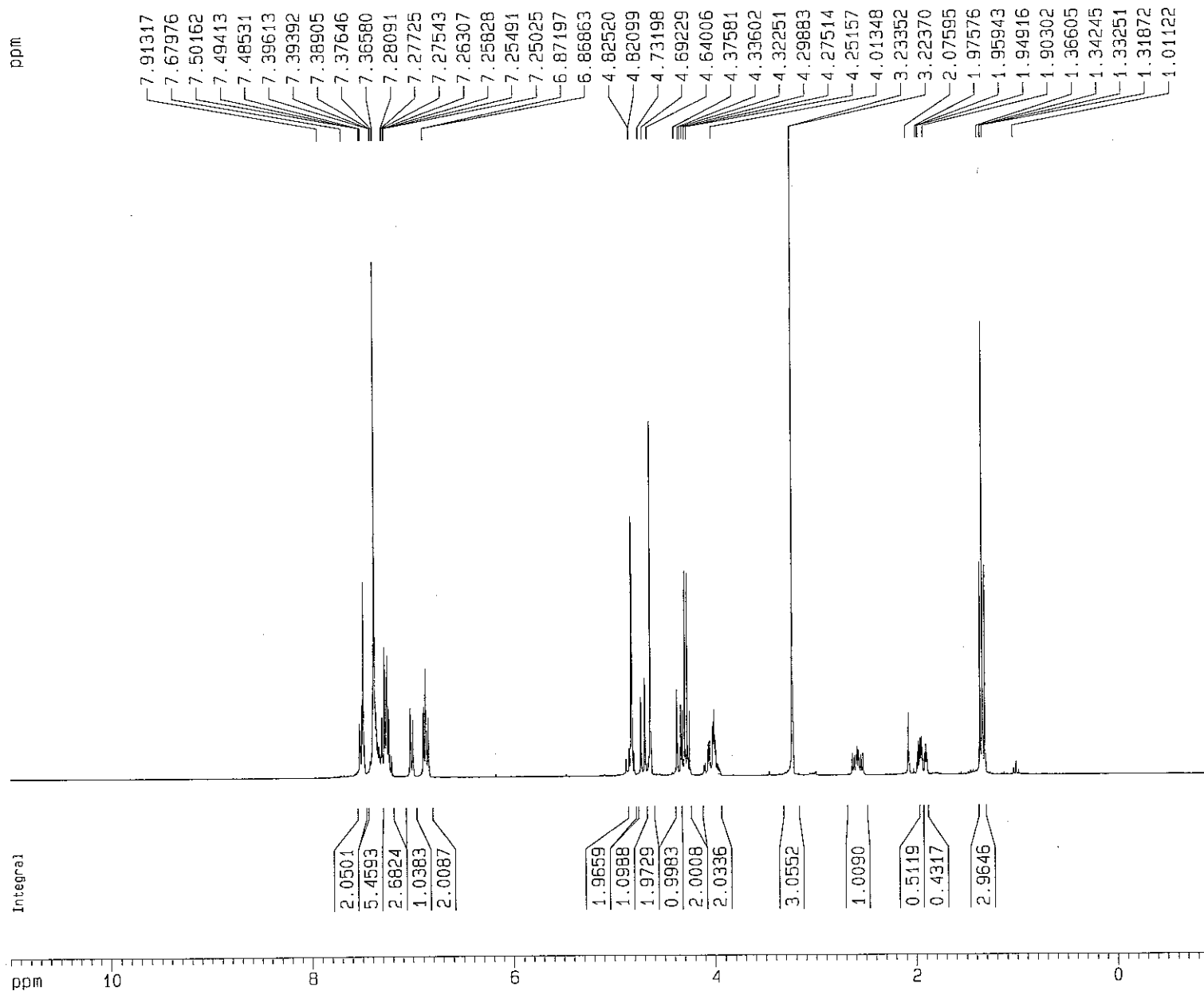
## 1D NMR plot parameters

CX 20.00 cm  
F1P 235.525 ppm  
F1 17759.12 Hz  
F2P -13.765 ppm  
F2 -1037.88 Hz  
PPMCM 12.46447 ppm/cm  
HZCM 939.84961 Hz/cm



26a

65 degree in CD3CN



## Current Data Parameters

NAME PL-Jul12-09  
EXPNO 2  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20090712  
Time 15.47  
INSTRUM spect  
PROBHD 5 mm QNP 1H/1  
PULPROG zg30  
TD 65536  
SOLVENT CD3CN  
NS 16  
DS 2  
SWH 6172.839 Hz  
FIDRES 0.094190 Hz  
AQ 5.3084660 sec  
RG 143.7  
DW 81.000 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec

## ===== CHANNEL f1 =====

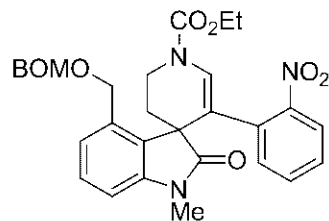
NUC1 1H  
P1 11.70 usec  
PL1 0.00 dB  
SFO1 299.8718518 MHz

## F2 - Processing parameters

SI 32768  
SF 299.8700000 MHz  
WDW no  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

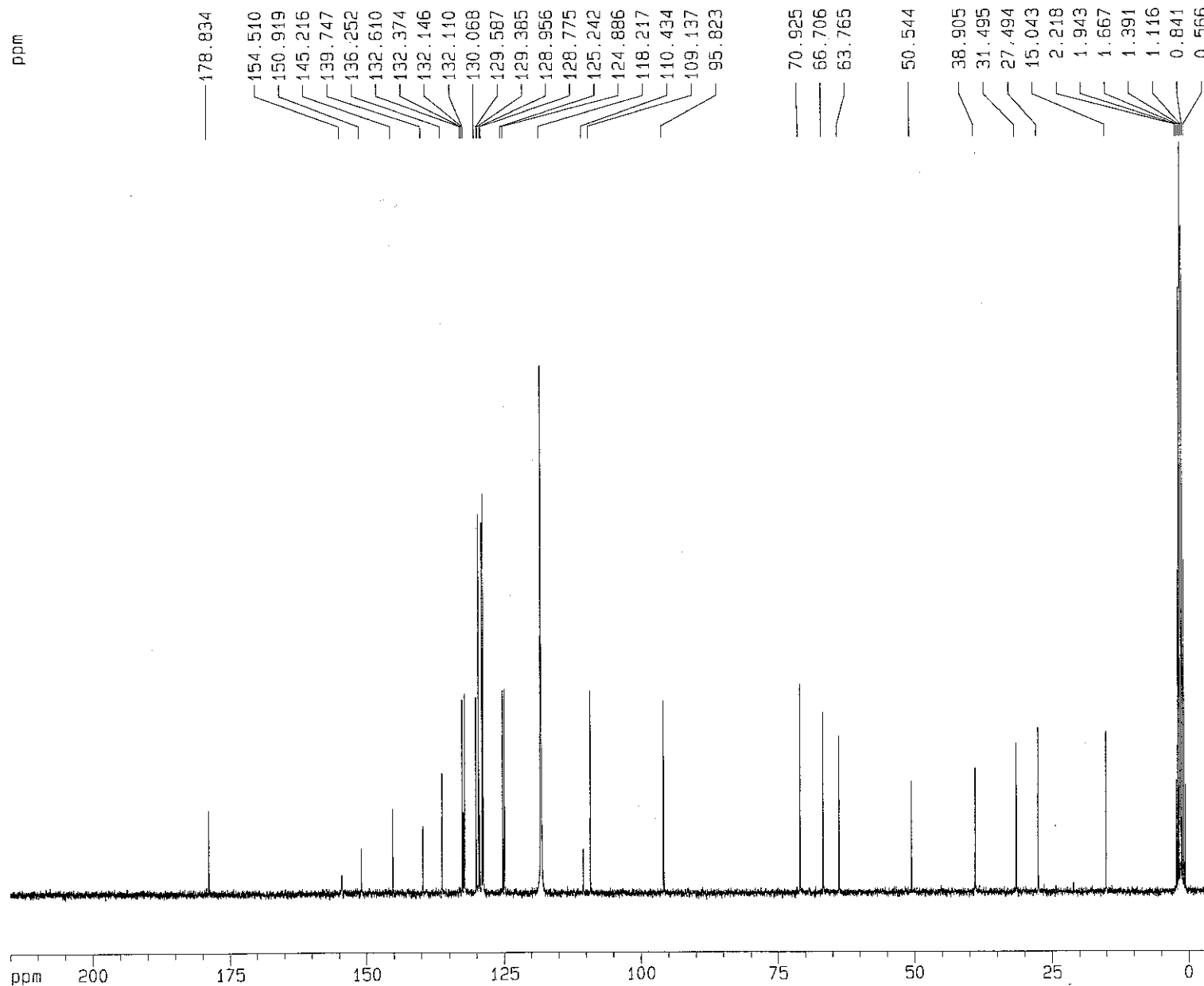
## 1D NMR plot parameters

CX 20.00 cm  
F1P 11.000 ppm  
F1 3298.57 Hz  
F2P -1.000 ppm  
F2 -299.87 Hz  
PPMCM 0.60000 ppm/cm  
HZCM 179.92200 Hz/cm



26a

in CD3CN at 65 degree



## Current Data Parameters

NAME PL-Jul14-09  
EXPNO 2  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20090714  
Time 15.25  
INSTRUM spect  
PROBHD 5 mm GNP 1H/1  
PULPROG zgpg30  
TD 65536  
SOLVENT CD3CN  
NS 604  
DS 4  
SWH 18796.992 Hz  
FIDRES 0.286619 Hz  
AQ 1.7433076 sec  
RG 4096  
DW 26.600 usec  
DE 6.00 usec  
TE 300.0 K  
D1 2.0000000 sec  
D11 0.03000000 sec  
D12 0.00002000 sec

## ===== CHANNEL f1 =====

NUC1 13C  
P1 5.40 usec  
PL1 -6.00 dB  
SFO1 75.4106357 MHz

## ===== CHANNEL f2 =====

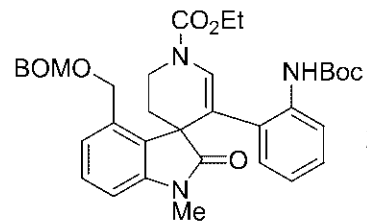
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 115.00 usec  
PL2 0.00 dB  
PL12 20.00 dB  
PL13 20.00 dB  
SFO2 299.8711995 MHz

## F2 - Processing parameters

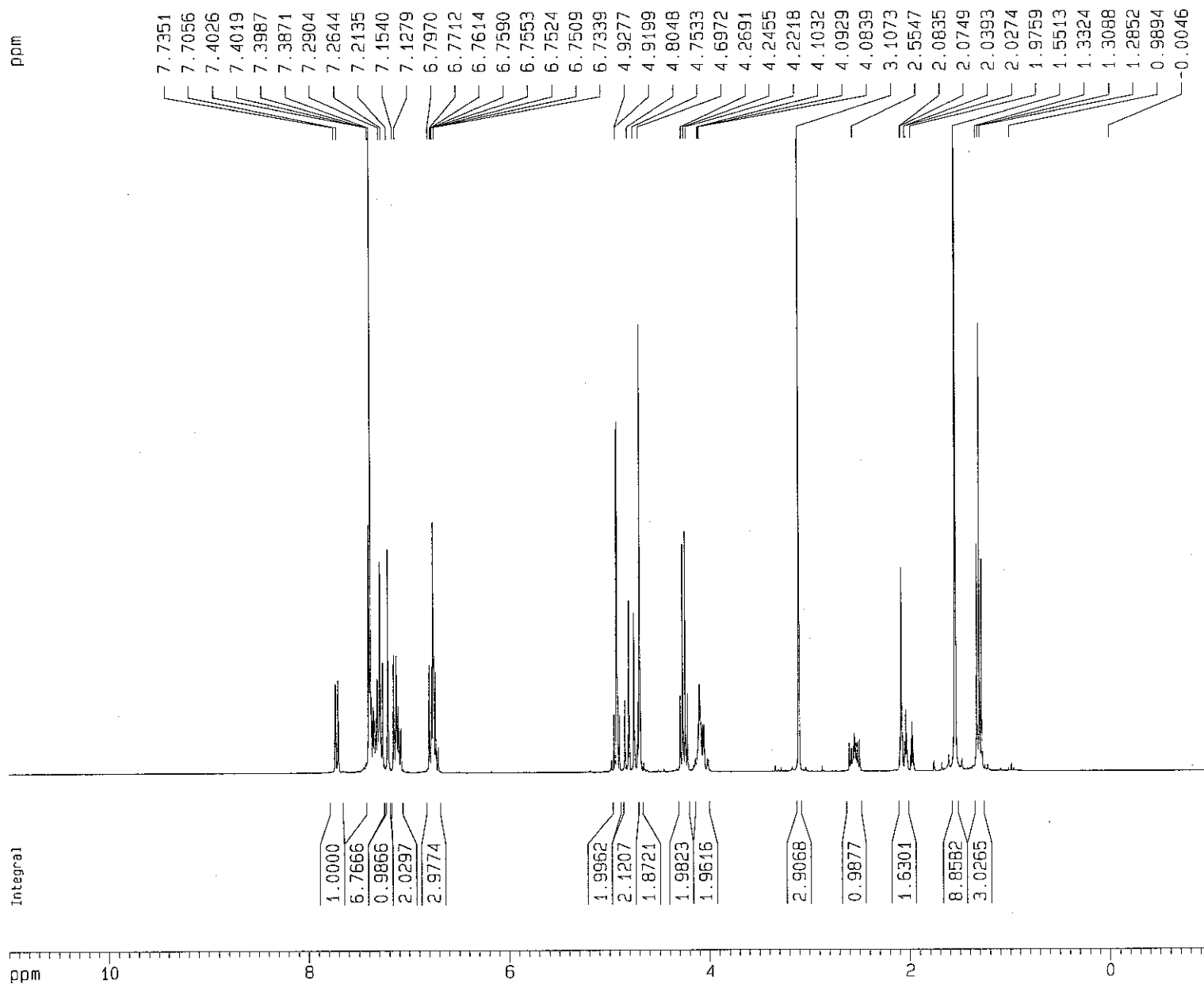
SI 32768  
SF 75.4022751 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

## f0 NMR plot parameters

CX 20.00 cm  
F1P 215.000 ppm  
F1 16211.49 Hz  
F2P -5.000 ppm  
F2 -377.01 Hz  
PPMCM 11.00000 ppm/cm  
HZCM 829.42505 Hz/cm



in CD<sub>3</sub>CN at 65 degree



## Current Data Parameters

NAME PL-Jul14-09  
EXPNO 5  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20090714  
Time 16.26  
INSTRUM spect  
PROBHD 5 mm QNP 1H/1  
PULPROG zg30  
TD 65536  
SOLVENT CD3CN  
NS 16  
DS 2  
SWH 6172.839 Hz  
FIDRES 0.094190 Hz  
AQ 5.3084660 sec  
RG 101.6  
DW 81.000 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec

## ===== CHANNEL f1 =====

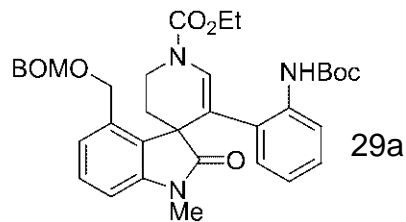
NUC1 1H  
P1 11.70 usec  
PL1 0.00 dB  
SFO1 299.8718518 MHz

## F2 - Processing parameters

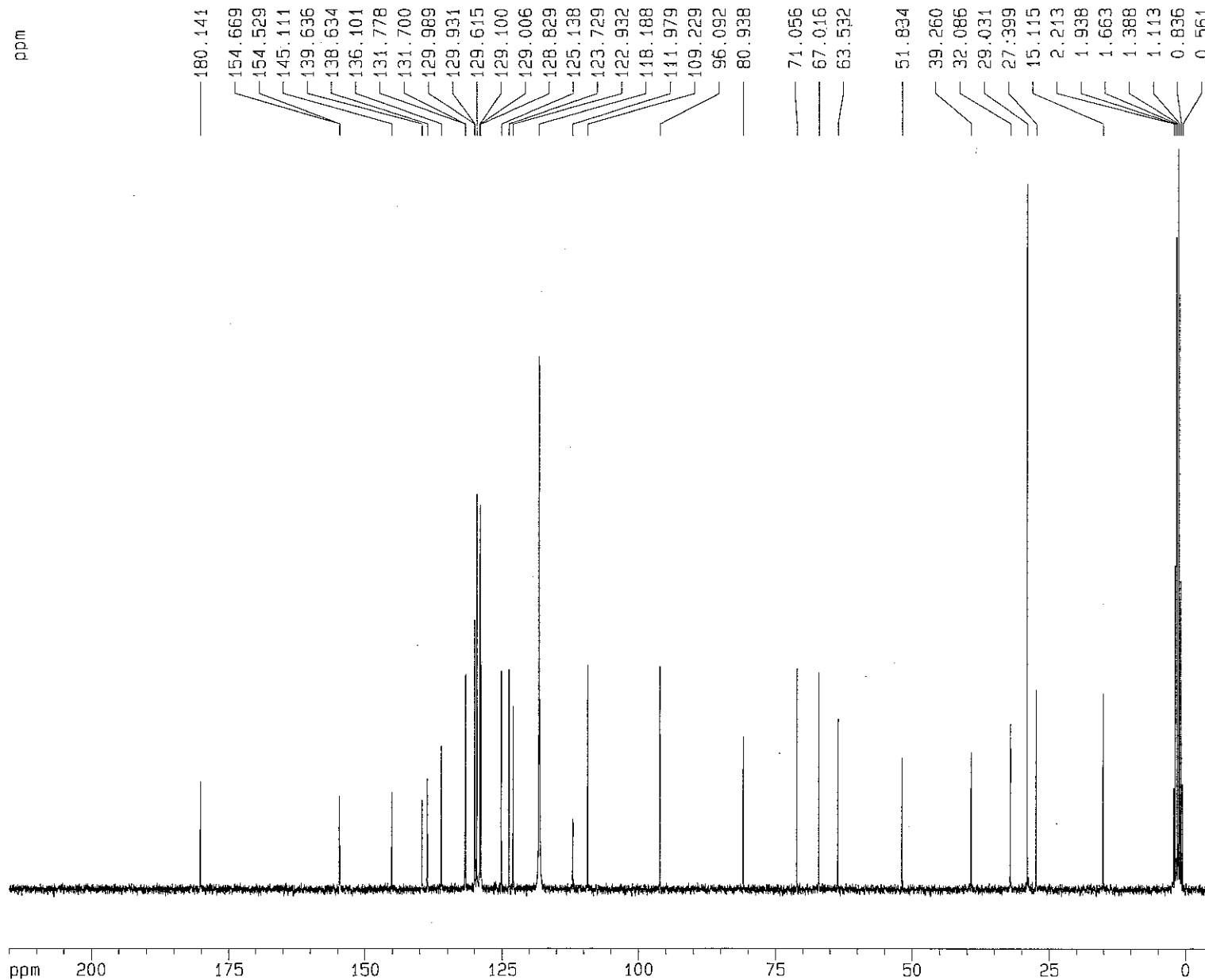
SI 32768  
SF 299.8700000 MHz  
WDW no  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

## 1D NMR plot parameters

CX 20.00 cm  
F1P 11.000 ppm  
F1 3298.57 Hz  
F2P -1.000 ppm  
F2 -299.87 Hz  
PPMCM 0.60000 ppm/cm  
HZCM 179.92200 Hz/cm



in CD3CN at 65 degree



## Current Data Parameters

NAME PL-Jul15-09  
EXPNO 1  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20090715  
Time 12.13  
INSTRUM spect  
PROBHD 5 mm GNP 1H/1  
PULPROG zgpg30  
TD 65536  
SOLVENT CD3CN  
NS 531  
DS 4  
SWH 18796.992 Hz  
FIDRES 0.286819 Hz  
AQ 1.7433076 sec  
RG 512  
DW 26.600 usec  
DE 6.00 usec  
TE 300.0 K  
D1 2.0000000 sec  
D11 0.0300000 sec  
D12 0.0002000 sec

## ===== CHANNEL f1 =====

NUC1 13C  
P1 5.40 usec  
PL1 -6.00 dB  
SFO1 75.4106357 MHz

## ===== CHANNEL f2 =====

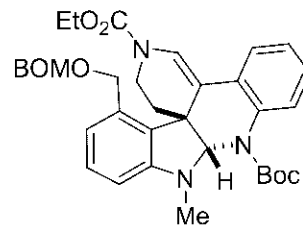
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 115.00 usec  
PL2 0.00 dB  
PL12 20.00 dB  
PL13 20.00 dB  
SFO2 299.8711995 MHz

## F2 - Processing parameters

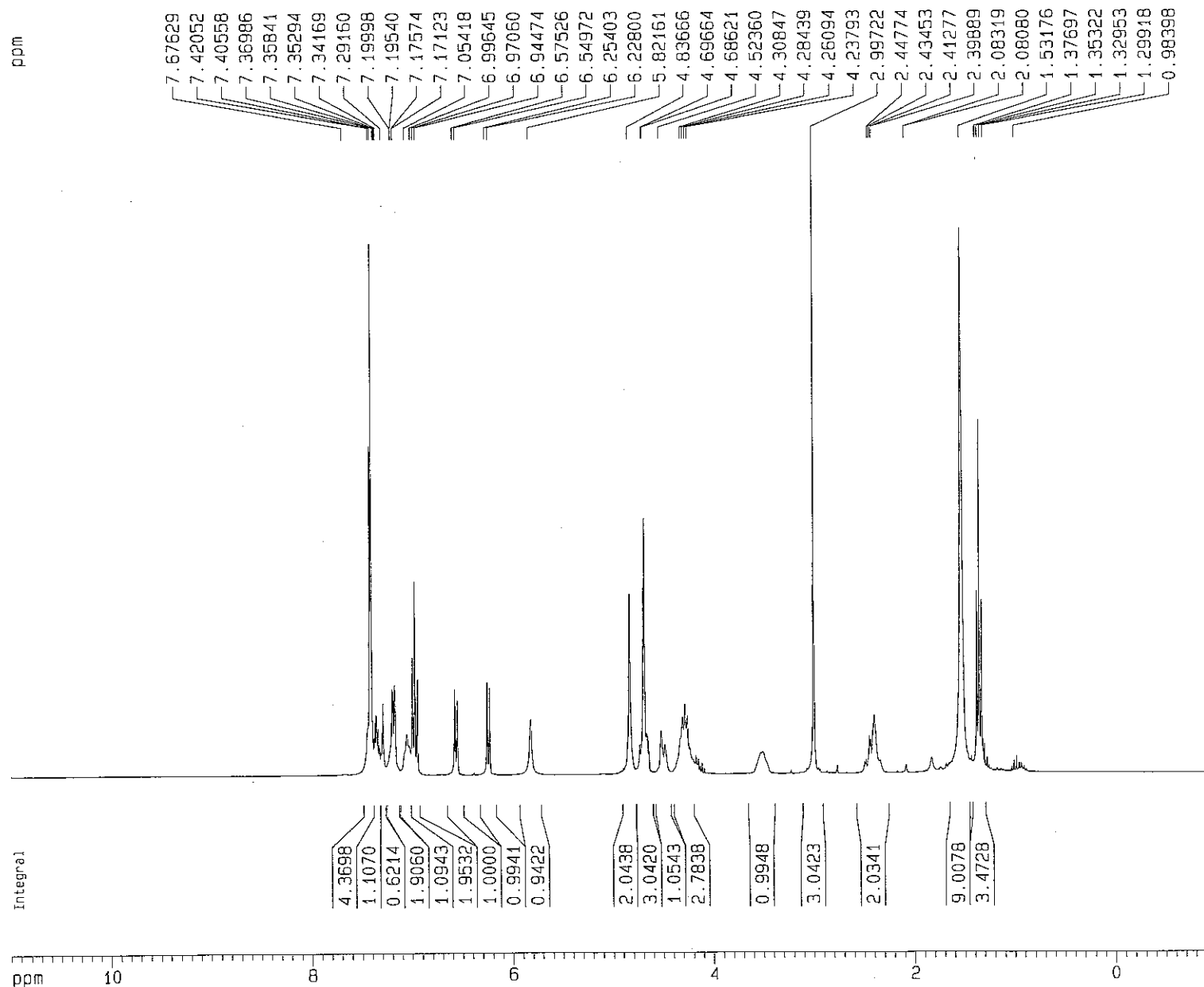
SI 32768  
SF 75.4022757 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

## 1D NMR plot parameters

CX 20.00 cm  
F1P 215.000 ppm  
F1 16211.49 Hz  
F2P -5.000 ppm  
F2 -377.01 Hz  
PPNMC 11.00000 ppm/cm  
HZCM 829.42505 Hz/cm



30a



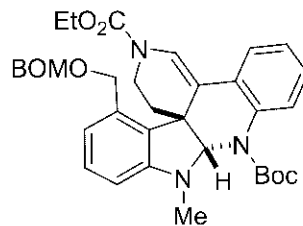
Current Data Parameters  
 NAME PL-Jul17-09  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20090717  
 Time 11.28  
 INSTRUM spect  
 PROBHD 5 mm GNP 1H/1  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDC13  
 NS 16  
 DS 2  
 SWH 6172.839 Hz  
 FIDRES 0.094190 Hz  
 AQ 5.3084660 sec  
 RG 64  
 DW 81.000 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 1.00000000 sec

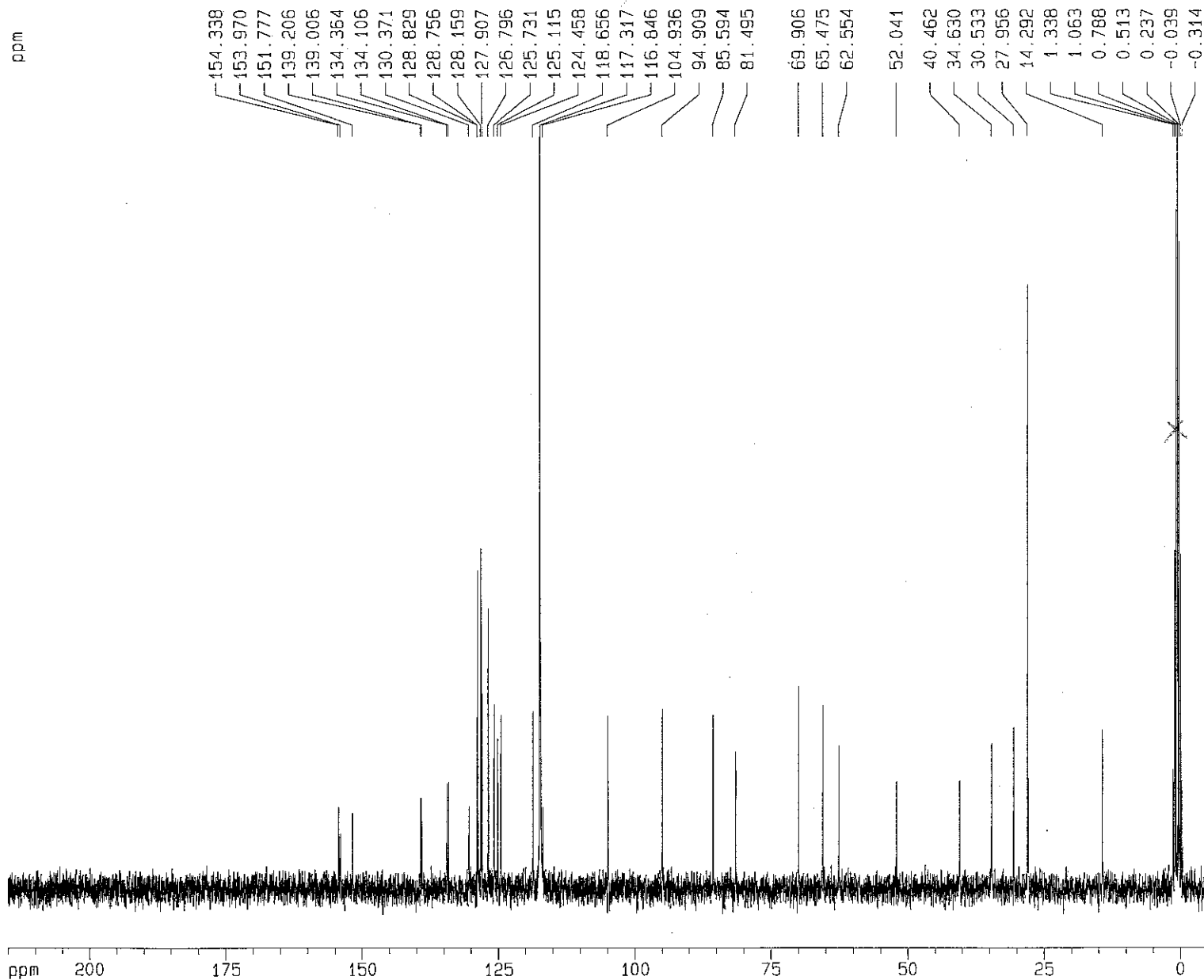
==== CHANNEL f1 =====  
 NUC1 1H  
 P1 11.70 usec  
 PL1 0.00 dB  
 SF01 299.8718518 MHz

F2 - Processing parameters  
 SI 32768  
 SF 299.8700000 MHz  
 WDW no  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 11.000 ppm  
 F1 3298.57 Hz  
 F2P -1.000 ppm  
 F2 -299.87 Hz  
 PPMCM 0.60000 ppm/cm  
 HZCM 179.92200 Hz/cm



30a

in CD<sub>3</sub>CN at 65 degree

## Current Data Parameters

NAME PL-Jul10-09  
EXPNO 3  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20090718  
Time 15.07  
INSTRUM spect  
PROBHD 5 mm GNP 1H/1  
PULPROG zgpg30  
TD 65536  
SOLVENT CD<sub>3</sub>CN  
NS 84  
DS 4  
SWH 18796.992 Hz  
FIDRES 0.286819 Hz  
AQ 1.7433076 sec  
RG 2048  
DW 26.600 usec  
DE 6.00 usec  
TE 300.0 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
D12 0.00002000 sec

===== CHANNEL f1 =====  
NUC1 13C  
P1 5.40 usec  
PL1 -6.00 dB  
SF01 75.4106357 MHz

===== CHANNEL f2 =====  
CPOPRG2 waltz16  
NUC2 1H  
PCPD2 115.00 usec  
PL2 0.00 dB  
PL12 20.00 dB  
PL13 20.00 dB  
SF02 299.8711995 MHz

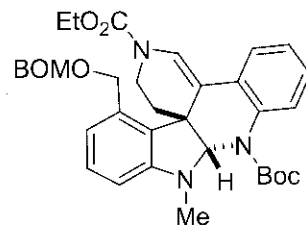
## F2 - Processing parameters

SI 32768  
SF 75.4023410 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

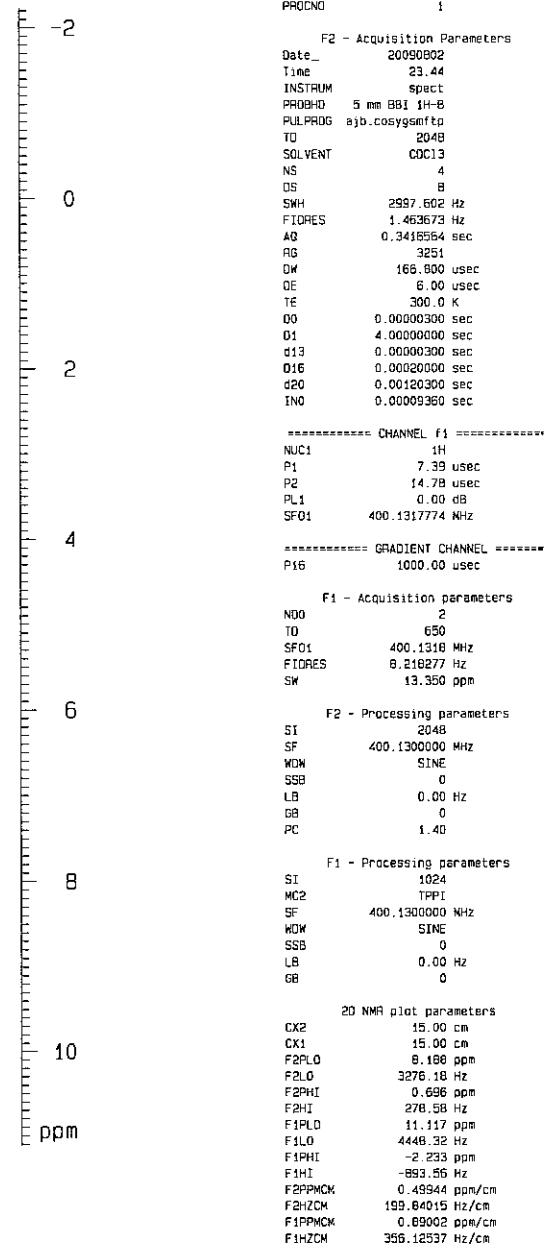
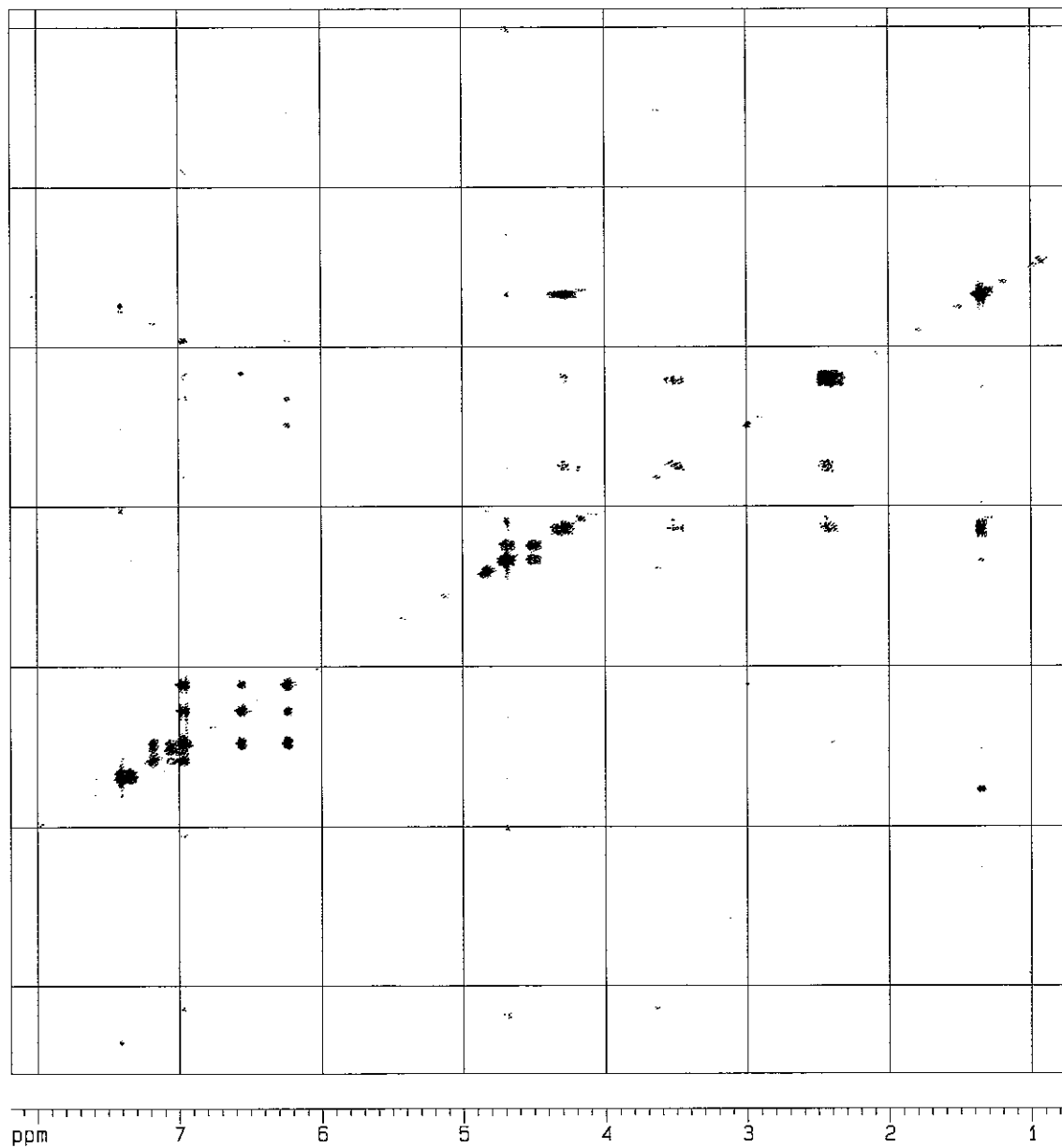
## 1D NMR plot parameters

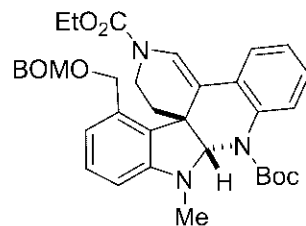
CX 20.00 cm  
F1P 215.000 ppm  
F1 16211.50 Hz  
F2P -5.000 ppm  
F2 -377.01 Hz  
PPMCM 11.00000 ppm/cm  
HZCM 829.42578 Hz/cm



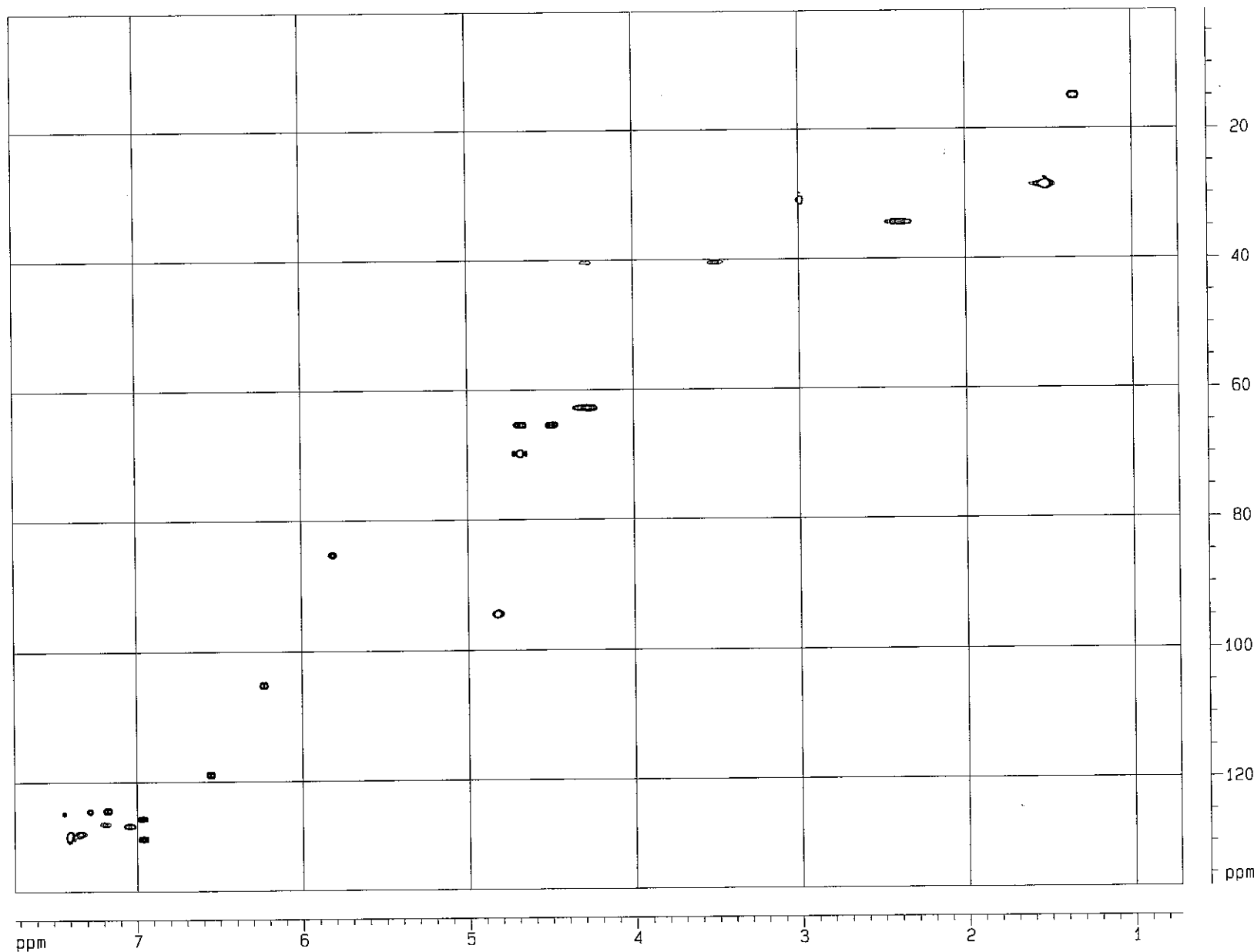


cosy 30a





HMQC 30a



```

Current Data Parameters
NAME      PL-Aug02-09
EXPNO    2
PROCNO    1

F2 - Acquisition Parameters
Date_     20090802
Time      25.03
INSTRUM   spect
PROBHD    5 mm BBI 1H-6
PULPROG   inv4qpts
TD        2048
SOLVENT   CDCl3
NS        4
DS        60
SWH       2897.602 Hz
FIDRES    1.463073 Hz
AQ        0.3415564 sec
RG        7266.2
RW        166.600 usec
DE        6.00 usec
TE        300.2 K
CNS12     145.0000000
d0        0.00000300 sec
d1        1.99831298 sec
d2        0.00344808 sec
d4        0.00172414 sec
d11       0.00000000 sec
d13       0.00000500 sec
d16       0.00000000 sec
d56       0.00052414 sec
d21       0.00224428 sec
IN0       0.00001450 sec

----- CHANNEL f1 -----
NUC1       1H
P1         7.39 usec
P2         14.78 usec
PL1        0.00 dB
SFO1       400.1317774 MHz

----- CHANNEL f2 -----
CPDPRG2    gprp
NUC2       13C
P3         17.00 usec
d4         34.00 usec
PCPD2     84.00 usec
PL2        -6.00 dB
PL12       6.30 dB
SFO2       100.6202332 MHz

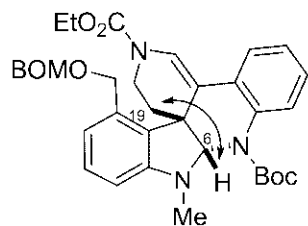
----- GRADIENT CHANNEL -----
GPM1H1     SINE.100
GPM1H2     SINE.100
GPM1H3     SINE.100
GPK1       17.00 %
GPK2       20.00 %
GPK3       25.00 %
GPV1       17.00 %
GPV2       20.00 %
GPV3       25.00 %
GPR1       17.00 %
GPR2       20.00 %
GPR3       25.00 %
F1S        1000.00 usec

F1 - Acquisition parameters
NUC1       13C
TD         256
SFO1       100.6202 MHz
FIDRES     65.541497 Hz
SW         156.751 ppm

F2 - Processing parameters
SI         2048
SF         400.1300000 MHz
WDW        GSIINE
SSB        2
LB         0.00 Hz
GB         0
PC         1.00

F1 - Processing parameters
SI         1024
WDW        TOP1
SF         100.6127290 MHz
WDW        GSIINE
SSB        2
LB         0.00 Hz
GB         0

2D NMR plot parameters
CXP        20.00 cm
CX1        15.00 cm
F2PLD      7.731 ppm
F2L0       3063.22 Hz
F2PH1      0.725 ppm
F2PH2      250.00 Hz
F1PL0      137.121 ppm
F1L0       13796.14 Hz
F1PH1      1.769 ppm
F1PH2      176.57 Hz
F2PRMCA    0.35025 ppm/cm
F2PRMCH    140.14667 Hz/cm
F3PRMCA    9.02216 ppm/cm
F3PRMCH    907.74432 Hz/cm
  
```

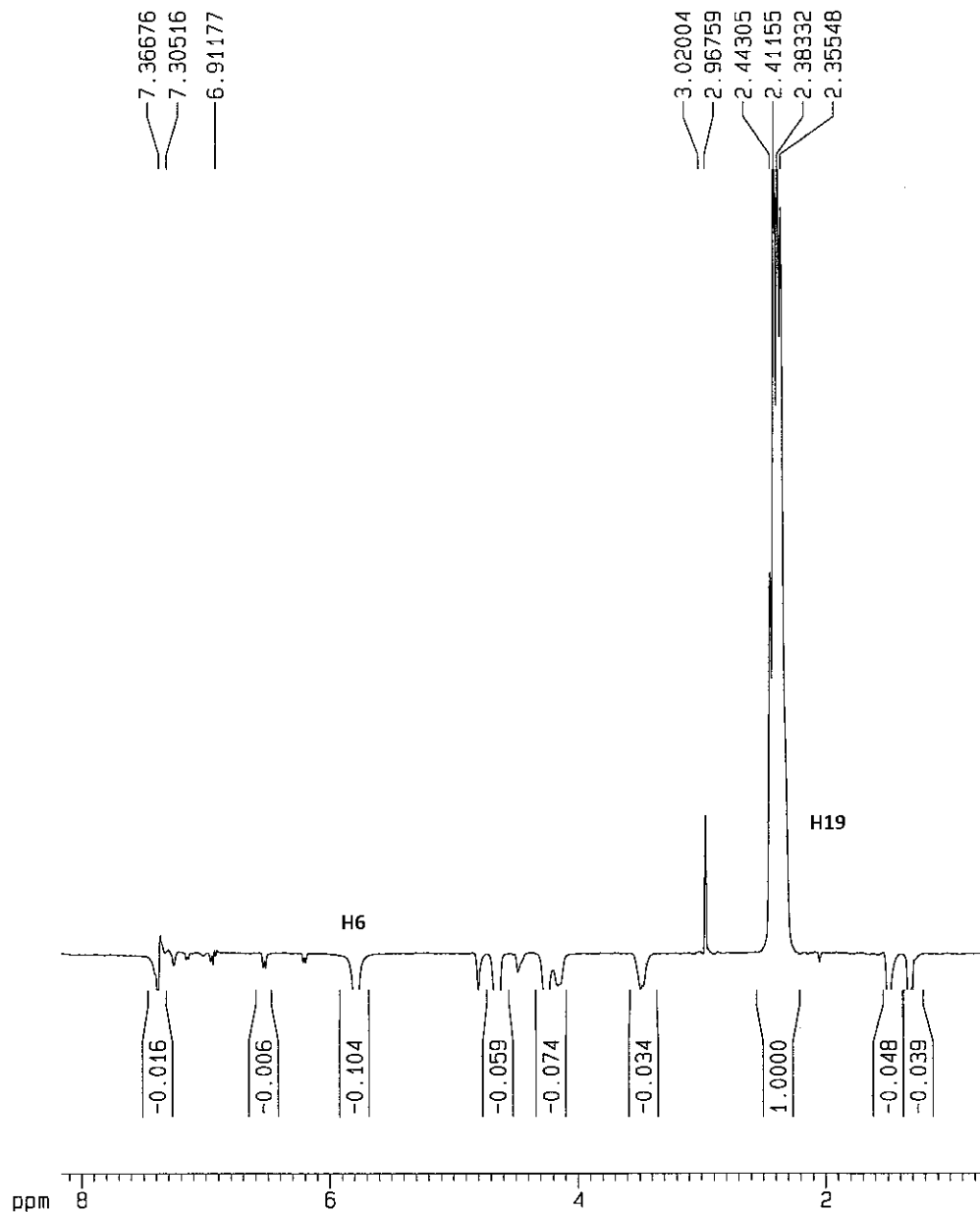


NOESY 30a

ppm

7.36676  
7.30516  
6.91177

3.02004  
2.96759  
2.44305  
2.41155  
2.38332  
2.35548



Integral

Current Data Parameters  
NAME PL-Aug03-09  
EXPNO 4  
PROCNO 6

F2 - Acquisition Parameters

Date\_ 20090803  
Time 2.55  
INSTRUM spect  
PROBHD 5 mm BBI 1H-B  
PULPROG noesygptp  
TD 2048  
SOLVENT CDCl3  
NS 8  
DS 8  
SWH 2997.602 Hz  
FIDRES 1.463673 Hz  
AQ 0.3416564 sec  
RG 71.8  
DW 166.800 usec  
DE 6.00 usec  
TE 300.0 K  
d0 0.00000300 sec  
D1 4.00000000 sec  
D8 0.80000001 sec  
D16 0.00020000 sec  
d20 0.39880002 sec  
IN0 0.00012496 sec

===== CHANNEL f1 =====

NUC1 1H  
P1 7.39 usec  
P2 14.78 usec  
PL1 0.00 dB  
SFO1 400.1317774 MHz

===== GRADIENT CHANNEL =====

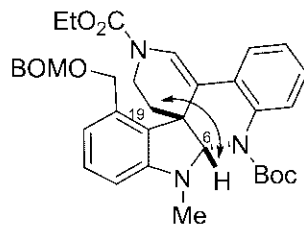
GPNAM1 sine.100  
GPNAM2 sine.100  
GPX1 0.00 %  
GPX2 0.00 %  
GPY1 0.00 %  
GPY2 0.00 %  
GPZ1 40.00 %  
GPZ2 -40.00 %  
P16 1000.00 usec

F2 - Processing parameters

SI 2048  
SF 400.1300092 MHz  
WDW QSINE  
SSB 2  
LB 0.00 Hz  
GB 0  
PC 1.00

1D NMR plot parameters

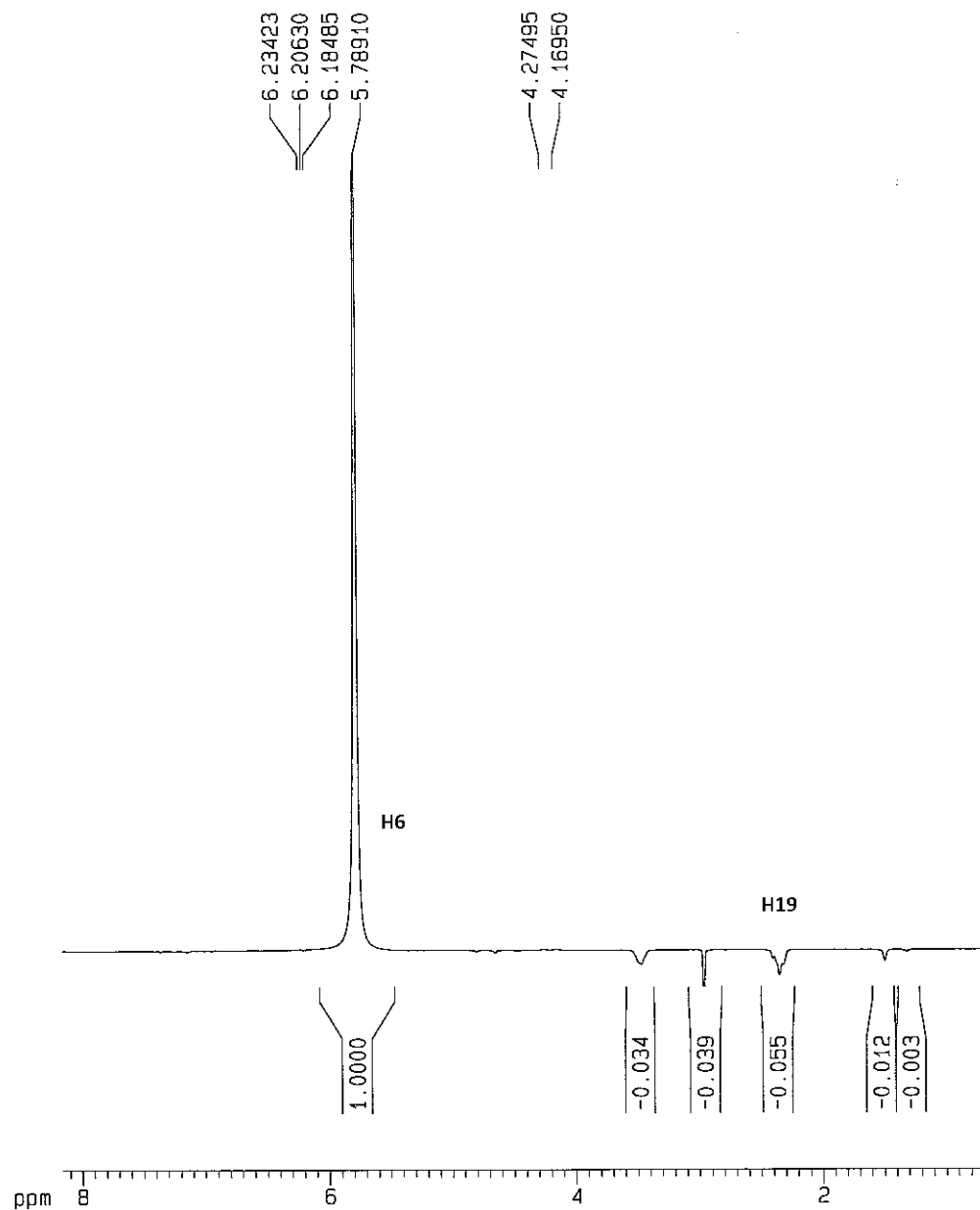
CX 20.00 cm  
F1P 11.000 ppm  
F1 4401.43 Hz  
F2P -1.000 ppm  
F2 -400.13 Hz  
PPMCM 0.60000 ppm/cm  
HZCM 240.07800 Hz/cm



NOESY

30a

ppm



Integral

## Current Data Parameters

NAME PL-Aug03-09  
EXPNO 4  
PROCNO 2

## F2 - Acquisition Parameters

Date\_ 20090803  
Time 2.55  
INSTRUM spect  
PROBHD 5 mm BBI 1H-8  
PULPROG noesygptp  
TD 2048  
SOLVENT CDCl3  
NS 8  
DS 8  
SWH 2997.602 Hz  
FIDRES 1.463673 Hz  
AQ 0.3416564 sec  
RG 71.8  
DW 166.800 usec  
DE 6.00 usec  
TE 300.0 K  
d0 0.00000300 sec  
D1 4.00000000 sec  
D8 0.80000001 sec  
D16 0.00020000 sec  
d20 0.39880002 sec  
IN0 0.00012496 sec

## ===== CHANNEL f1 =====

NUC1 1H  
P1 7.39 usec  
P2 14.78 usec  
PL1 0.00 dB  
SF01 400.1317774 MHz

## ===== GRADIENT CHANNEL =====

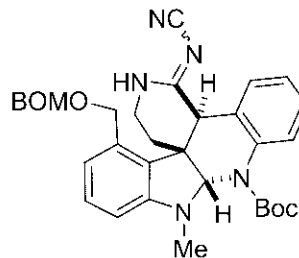
GPNAME1 sine.100  
GPNAME2 sine.100  
GPX1 0.00 %  
GPX2 0.00 %  
GPY1 0.00 %  
GPY2 0.00 %  
GPZ1 40.00 %  
GPZ2 -40.00 %  
P16 1000.00 usec

## F2 - Processing parameters

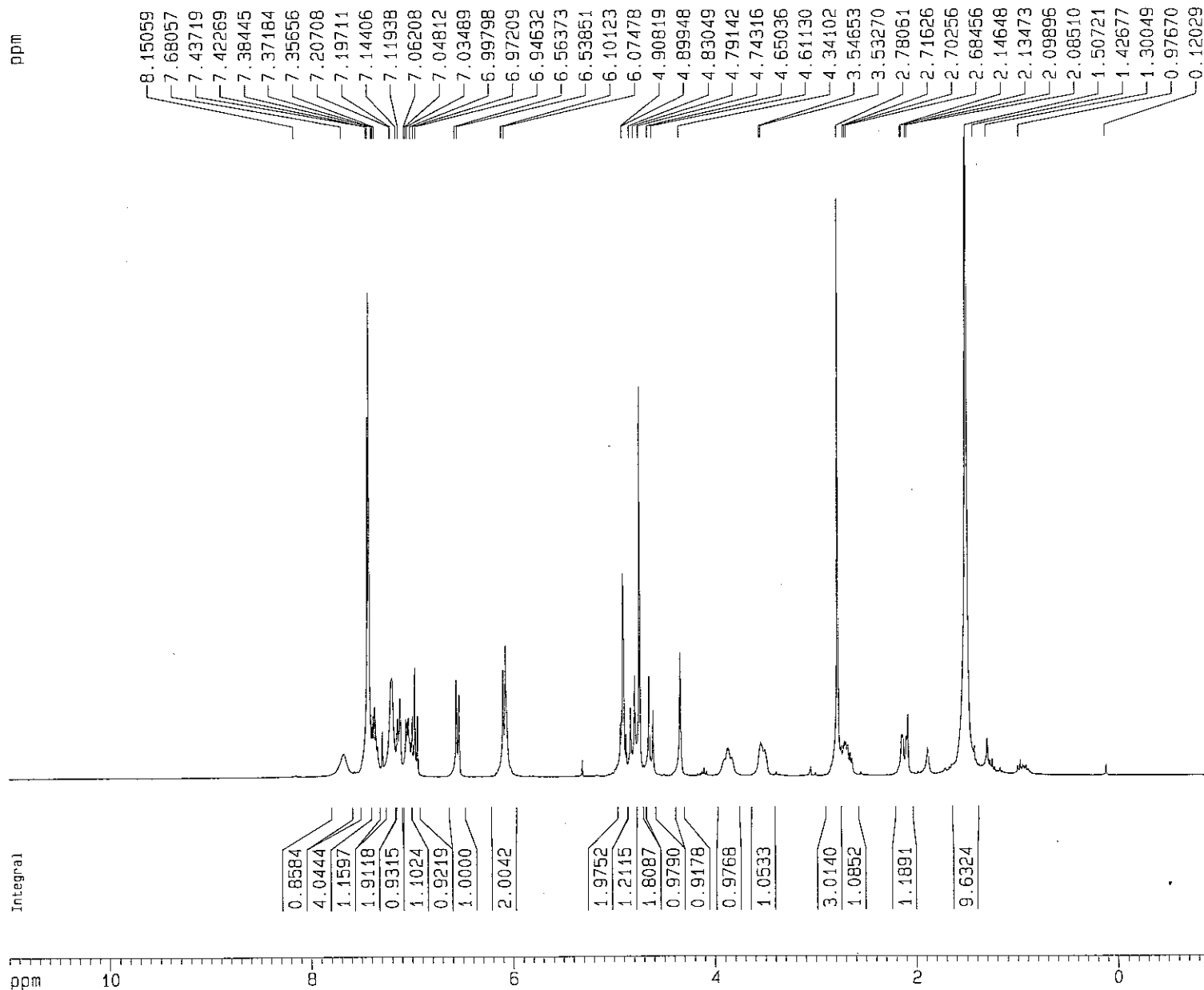
SI 2048  
SF 400.1300092 MHz  
WDW GSINE  
SSB 2  
LB 0.00 Hz  
GB 0  
PC 1.00

## 1D NMR plot parameters

CX 20.00 cm  
F1P 11.000 ppm  
F1 4401.43 Hz  
F2P -1.000 ppm  
F2 -400.13 Hz  
PPMCM 0.60000 ppm/cm  
HZCM 240.07800 Hz/cm



47



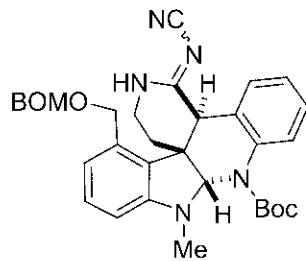
Current Data Parameters  
 NAME PL-Jul23-09  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20090723  
 Time 12.56  
 INSTAUM spect  
 PROBHD 5 mm GNP 1H/1  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 16  
 DS 2  
 SWH 6172.839 Hz  
 FIDRES 0.094190 Hz  
 AQ 5.3084660 sec  
 RG 114  
 DW 81.000 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 1.0000000 sec

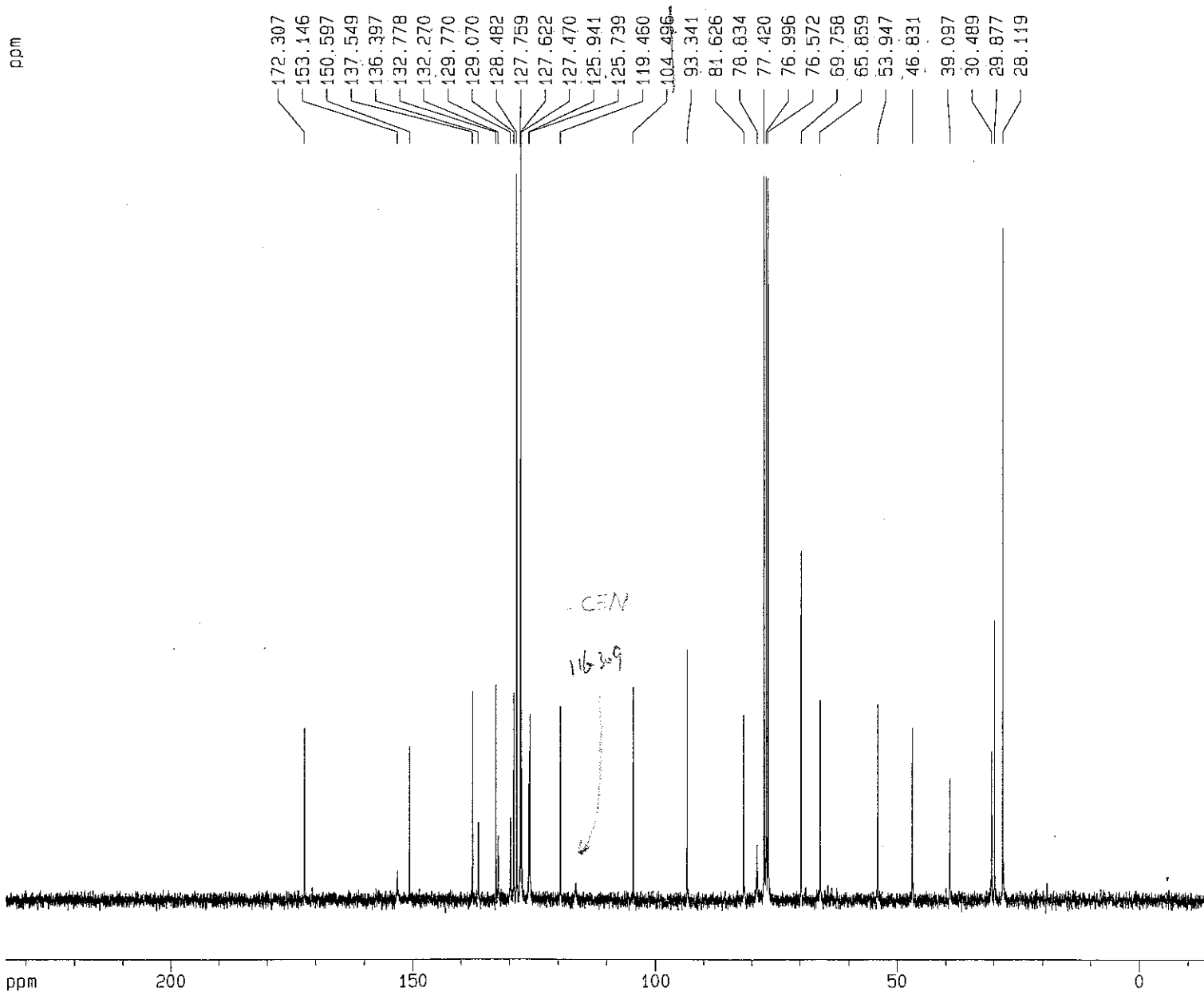
===== CHANNEL f1 =====  
 NUC1 1H  
 P1 11.70 usec  
 PL1 0.00 dB  
 SFO1 299.8718518 MHz

F2 - Processing parameters  
 SI 32768  
 SF 299.8700000 MHz  
 WDW no  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 11.000 ppm  
 F1 3298.57 Hz  
 F2P -1.000 ppm  
 F2 -299.87 Hz  
 PPMCM 0.60000 ppm/cm  
 HZCM 179.92200 Hz/cm



47



## Current Data Parameters

NAME PL-Ju123-09  
EXPNO 2  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20090723  
Time 13.42  
INSTRUM spect  
PROBHD 5 mm GNP 1H/1  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 759  
DS 4  
SWH 18796.992 Hz  
FIDRES 0.286819 Hz  
AQ 1.7433076 sec  
RG 4096  
DW 26.600 usec  
DE 6.00 usec  
TE 300.0 K  
D1 2.0000000 sec  
D11 0.0300000 sec  
D12 0.0002000 sec

## ===== CHANNEL f1 =====

NUC1 13C  
P1 5.40 usec  
PL1 -6.00 dB  
SF01 75.4106357 MHz

## ===== CHANNEL f2 =====

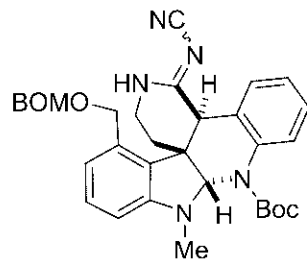
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 115.00 usec  
PL2 0.00 dB  
PL12 20.00 dB  
PL13 20.00 dB  
SF02 299.8711995 MHz

## F2 - Processing parameters

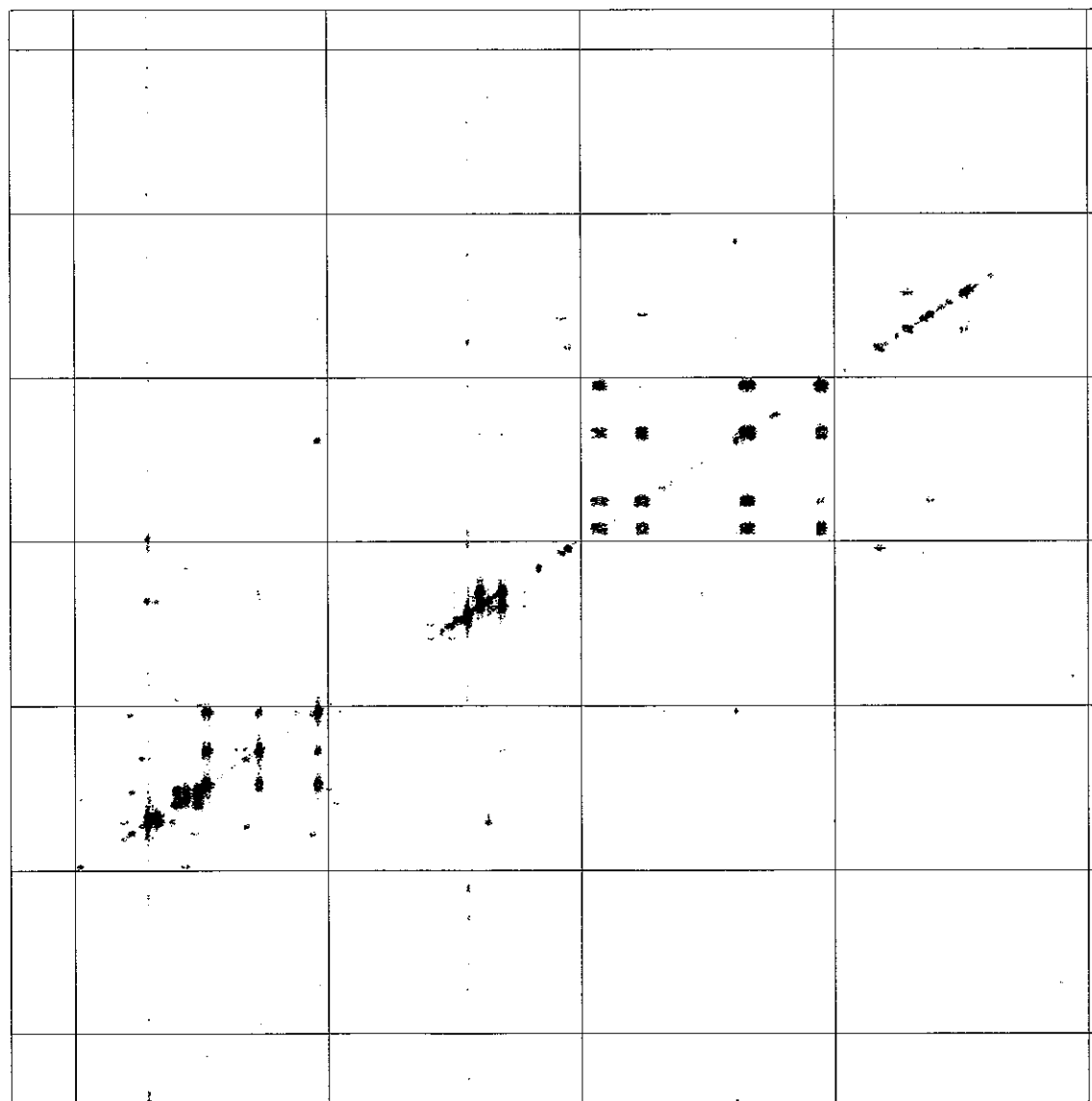
SI 32768  
SF 75.4023826 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

## 1D NMR plot parameters

CX 20.00 cm  
F1P 234.099 ppm  
F1 17651.62 Hz  
F2P -15.190 ppm  
F2 -1145.37 Hz  
PPMCM 12.46445 ppm/cm  
HZCM 939.84949 Hz/cm



47  
COSY



ppm 10 8 6 4 2

-2  
0  
2  
4  
6  
8  
10  
ppm

Current Data Parameters  
NAME PL-Aug01-09  
EXPNO 8  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20090802  
Time 1.04  
INSTRUM spect  
PROBHD 5 mm BBI 1H-8  
PULPROG ajb.cosygsm1tp  
TD 2048  
SOLVENT CDCl3  
NS 4  
DS 8  
SWH 3453.039 Hz  
FIDRES 1.586054 Hz  
AQ 0.2966004 sec  
RG 3649.1  
DM 144.800 usec  
DE 6.00 usec  
TE 300.0 K  
DO 0.0000300 sec  
D1 4.0000000 sec  
d13 0.0000300 sec  
D16 0.0002000 sec  
d20 0.00120300 sec  
IN0 0.0009360 sec

\*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
NUC1 1H  
P1 7.50 usec  
P2 15.00 usec  
PL1 0.00 dB  
SFO1 400.1316765 MHz

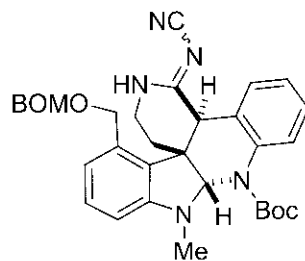
\*\*\*\*\* GRADIENT CHANNEL \*\*\*\*\*  
P16 1000.00 usec

F1 - Acquisition parameters  
ND0 2  
TD 650  
SFO1 400.1317 MHz  
FIDRES 8.218277 Hz  
SW 19.350 ppm

F2 - Processing parameters  
SI 2048  
SF 400.1300000 MHz  
WDW SINE  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.40

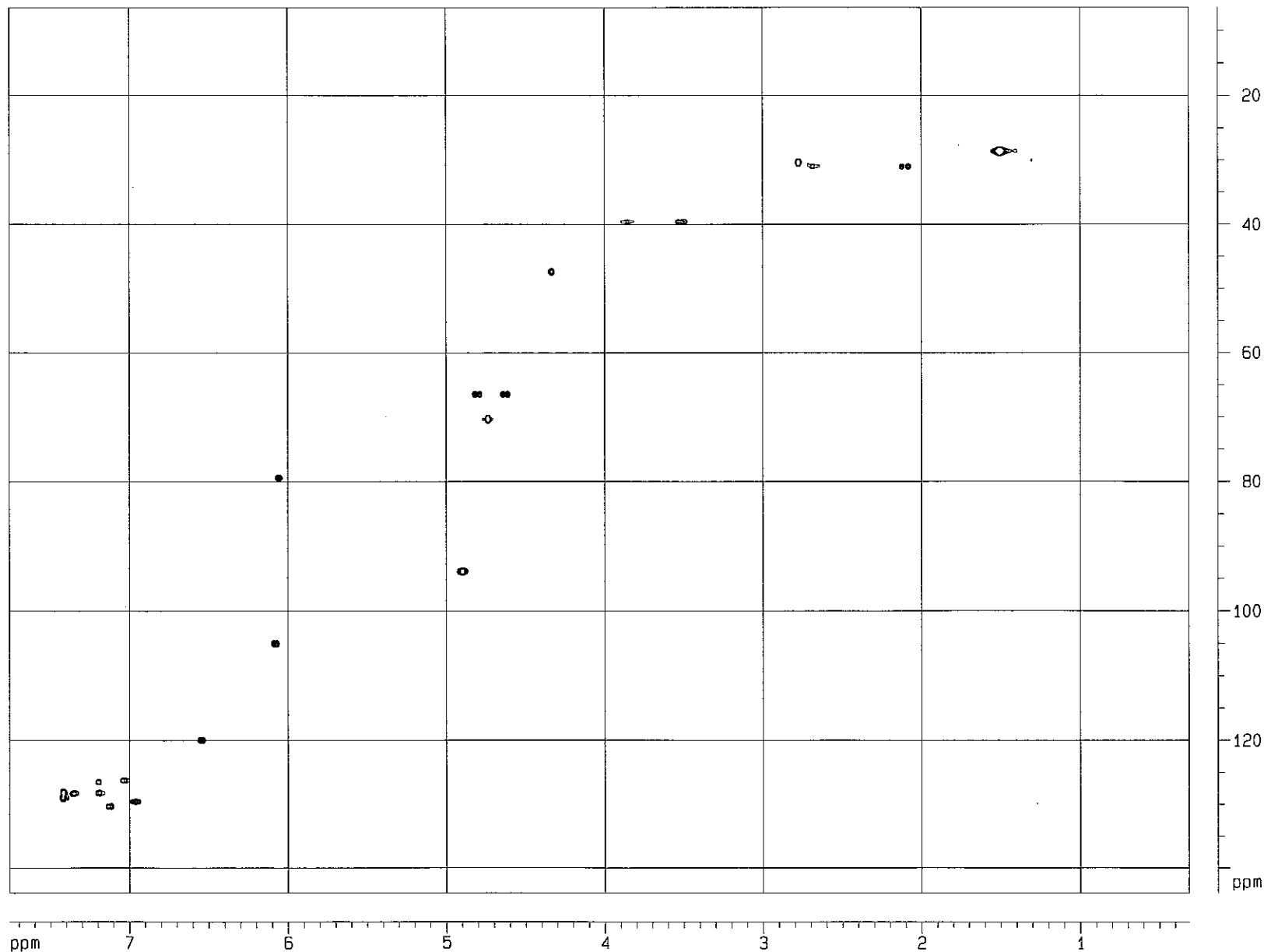
F1 - Processing parameters  
SI 1024  
MC2 TPP1  
SF 400.1300000 MHz  
WDW SINE  
SSB 0  
LB 0.00 Hz  
GB 0

2D NMR plot parameters  
CX2 15.00 cm  
CX1 15.00 cm  
F2PLO 8.505 ppm  
F2LO 3403.06 Hz  
F2PHI -0.125 ppm  
F2HI -49.97 Hz  
F1PLO 10.865 ppm  
F1LO 4347.48 Hz  
F1PHI -2.485 ppm  
F1HI -994.40 Hz  
F2PPMCM 0.57532 ppm/cm  
F2HZCM 230.20259 Hz/cm  
F1PPMCM 0.89002 ppm/cm  
F1HZCM 356.12537 Hz/cm



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HMQC



Current Data Parameters  
 NAME PL-Aug01-09  
 EXPNO 6  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20090801  
 Time 22.14  
 INSTRUM spect  
 PROBHD 5 mm BBI 1H-6  
 PULPROG inv4cp1p  
 TD 2048  
 SOLVENT CDCl3  
 NS 4  
 DS 80  
 SNH 3453.035 Hz  
 FIDRES 1.859504 Hz  
 AQ 0.2565004 sec  
 RG 3648.1  
 DM 144.600 usec  
 DE 6.00 usec  
 TE 300.0 K  
 CNST2 145.000000  
 dd 0.0000300 sec  
 d1 1.39631298 sec  
 d2 0.00344628 sec  
 d4 0.00172414 sec  
 d11 0.03000000 sec  
 d13 0.00000300 sec  
 d16 0.00000000 sec  
 d20 0.00002414 sec  
 d21 0.00224426 sec  
 IND 0.00001490 sec

----- CHANNEL f1 -----  
 NUC1 1H  
 P1 7.50 usec  
 p2 15.00 usec  
 PL1 0.00 dB  
 SFO1 400.1316765 MHz

----- CHANNEL f2 -----  
 CPDPRG2 gmp  
 NUC2 13C  
 P3 17.00 usec  
 p4 34.00 usec  
 PCPD2 64.00 usec  
 PL2 -6.00 dB  
 PL12 6.30 dB  
 SFO2 100.6262532 MHz

----- GRADIENT CHANNEL -----  
 GPMAX1 SINE 100  
 GPMAX2 SINE 100  
 GPMAX3 SINE 100  
 GPC1 17.00 %  
 GPC2 20.00 %  
 GPC3 25.00 %  
 GPC4 17.00 %  
 GPC5 20.00 %  
 GPC6 25.00 %  
 GPC7 17.00 %  
 GPC8 20.00 %  
 GPC9 25.00 %  
 GPC10 1000.00 usec

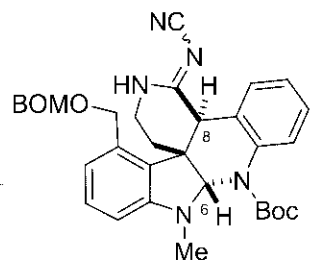
F1 - Acquisition parameters  
 TD 4  
 SF01 100.6262 MHz  
 FIDRES 65.541017 Hz  
 SN 158.751 ppm

F2 - Processing parameters  
 SI 2048  
 SF 400.1300000 MHz  
 WDW GSINE  
 SSB 2  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

F1 - Processing parameters  
 SI 1024  
 MC2 TPP1  
 SF 100.6127290 MHz  
 WDW GSINE  
 SSB 2  
 LB 0.00 Hz  
 GB 0

2D NMR plot parameters  
 CX2 50.00 cm  
 CX1 15.00 cm  
 F2PLD 7.759 ppm  
 F2L0 3404.63 Hz  
 F2PH 3.315 ppm  
 F2M1 125.37 Hz  
 F1PLD 143.961 ppm  
 F1L0 14484.32 Hz  
 F1PH 0.349 ppm  
 F1M1 638.76 Hz  
 F2PPMCM 0.37229 ppm/cm  
 F2HZCM 148.90289 Hz/cm  
 F1PPMCM 8.17416 ppm/cm  
 F1HZCM 828.03729 Hz/cm

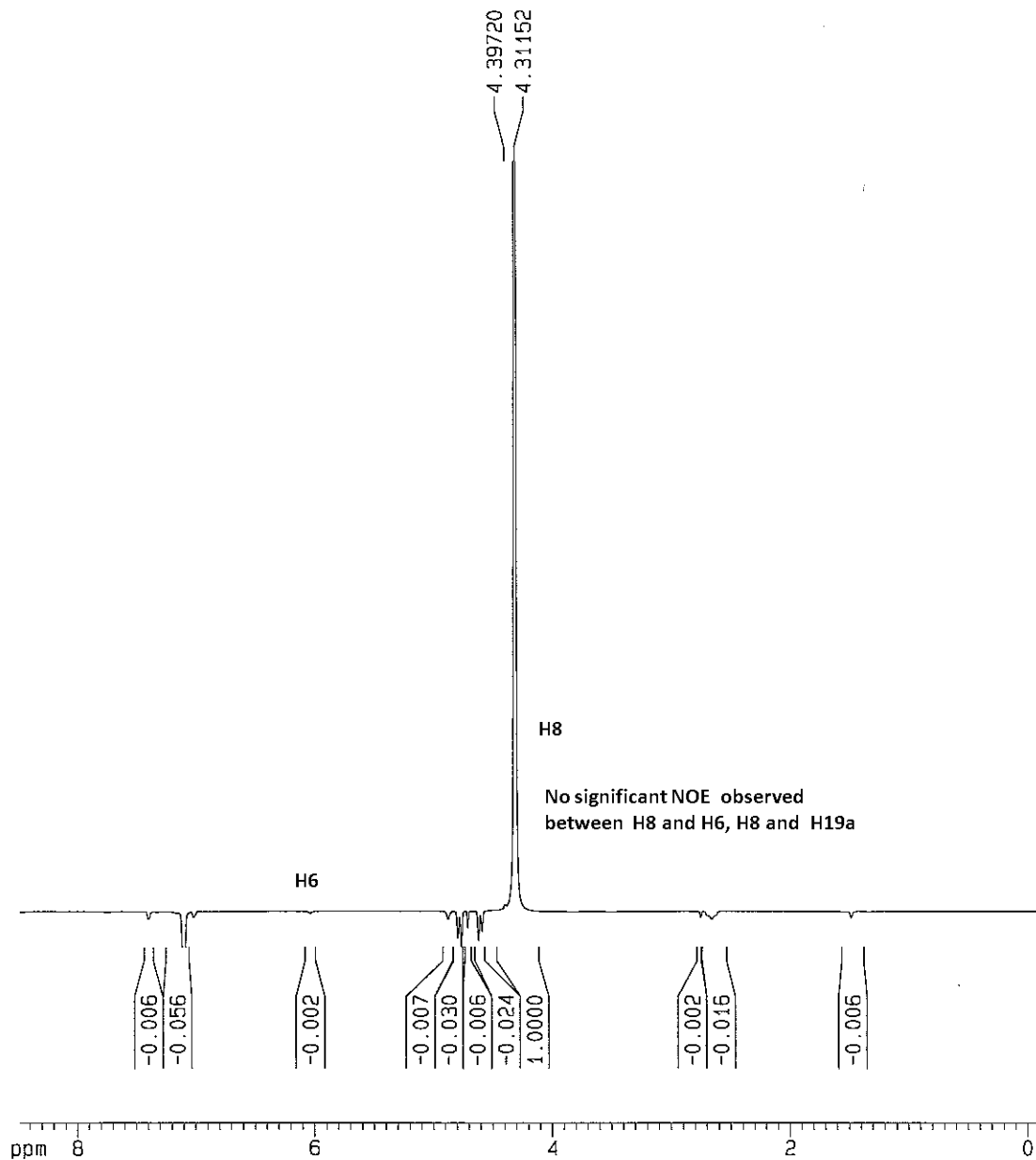




NOESY 47

ppm

Integral



Current Data Parameters  
 NAME PL-Aug01-09  
 EXPNO 9  
 PROCNO 2

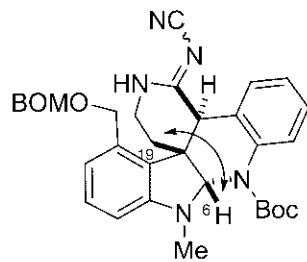
F2 - Acquisition Parameters  
 Date\_ 20090802  
 Time 4.13  
 INSTRUM spect  
 PROBHD 5 mm BBI 1H-8  
 PULPROG noesygtp  
 TD 2048  
 SOLVENT CDCl3  
 NS 8  
 DS 8  
 SWH 3453.039 Hz  
 FIDRES 1.686054 Hz  
 AQ 0.2966004 sec  
 RG 64  
 DW 144.800 usec  
 DE 6.00 usec  
 TE 300.0 K  
 d0 0.0000300 sec  
 D1 4.0000000 sec  
 DB 0.8000001 sec  
 D16 0.0002000 sec  
 d20 0.39880002 sec  
 IN0 0.00012496 sec

===== CHANNEL F1 =====  
 NUC1 1H  
 P1 7.50 usec  
 P2 15.00 usec  
 PL1 0.00 dB  
 SFO1 400.1316765 MHz

===== GRADIENT CHANNEL =====  
 GPNAM1 sine.100  
 GPNAM2 sine.100  
 GPX1 0.00 %  
 GPX2 0.00 %  
 GPY1 0.00 %  
 GPY2 0.00 %  
 GPZ1 40.00 %  
 GPZ2 -40.00 %  
 P16 1000.00 usec

F2 - Processing parameters  
 SI 2048  
 SF 400.1300092 MHz  
 NDW QSINE  
 SSB 2  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 11.000 ppm  
 F1 4401.43 Hz  
 F2P -1.000 ppm  
 F2 -400.13 Hz  
 PPMCM 0.60000 ppm/cm  
 HZCM 240.07800 Hz/cm

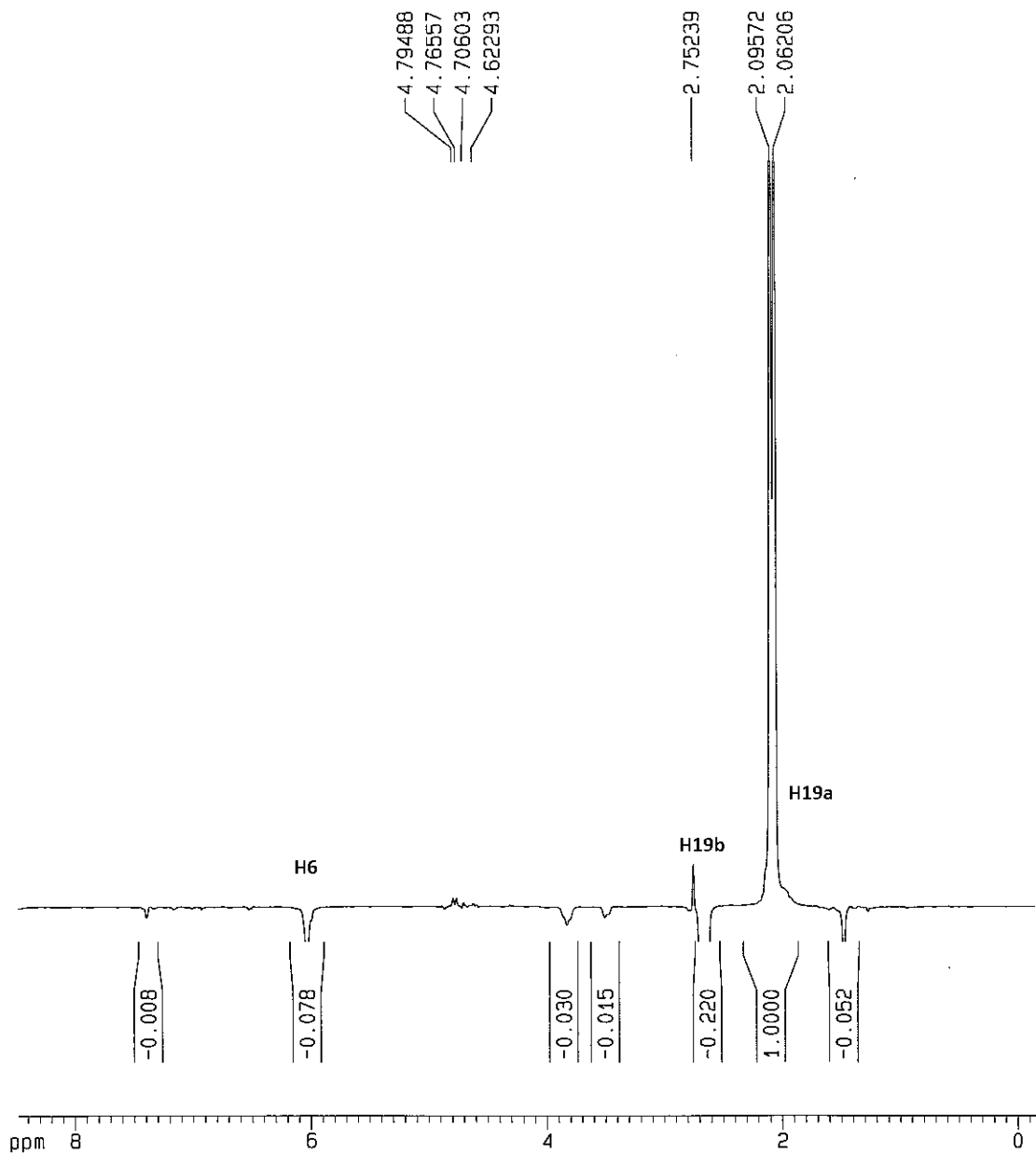


NOESY 47

ppm

4.79488  
4.76557  
4.70603  
4.62293

2.75239  
2.09572  
2.06206



Integral

Current Data Parameters  
NAME PL-Aug01-09  
EXPNO 9  
PROCNO 5

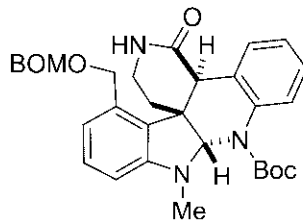
F2 - Acquisition Parameters  
Date\_ 20090802  
Time 4.13  
INSTRUM spect  
PROBHD 5 mm BBI 1H-8  
PULPROG noesygptp  
TD 2048  
SOLVENT CDCl3  
NS 8  
DS 8  
SWH 3453.039 Hz  
FIDRES 1.686054 Hz  
AQ 0.2966004 sec  
RG 64  
DW 144.800 usec  
DE 6.00 usec  
TE 300.0 K  
d0 0.00000300 sec  
D1 4.00000000 sec  
DB 0.80000001 sec  
D16 0.00020000 sec  
d20 0.39880002 sec  
IN0 0.00012496 sec

----- CHANNEL f1 -----  
NUC1 1H  
P1 7.50 usec  
P2 15.00 usec  
PL1 0.00 dB  
SFO1 400.1316765 MHz

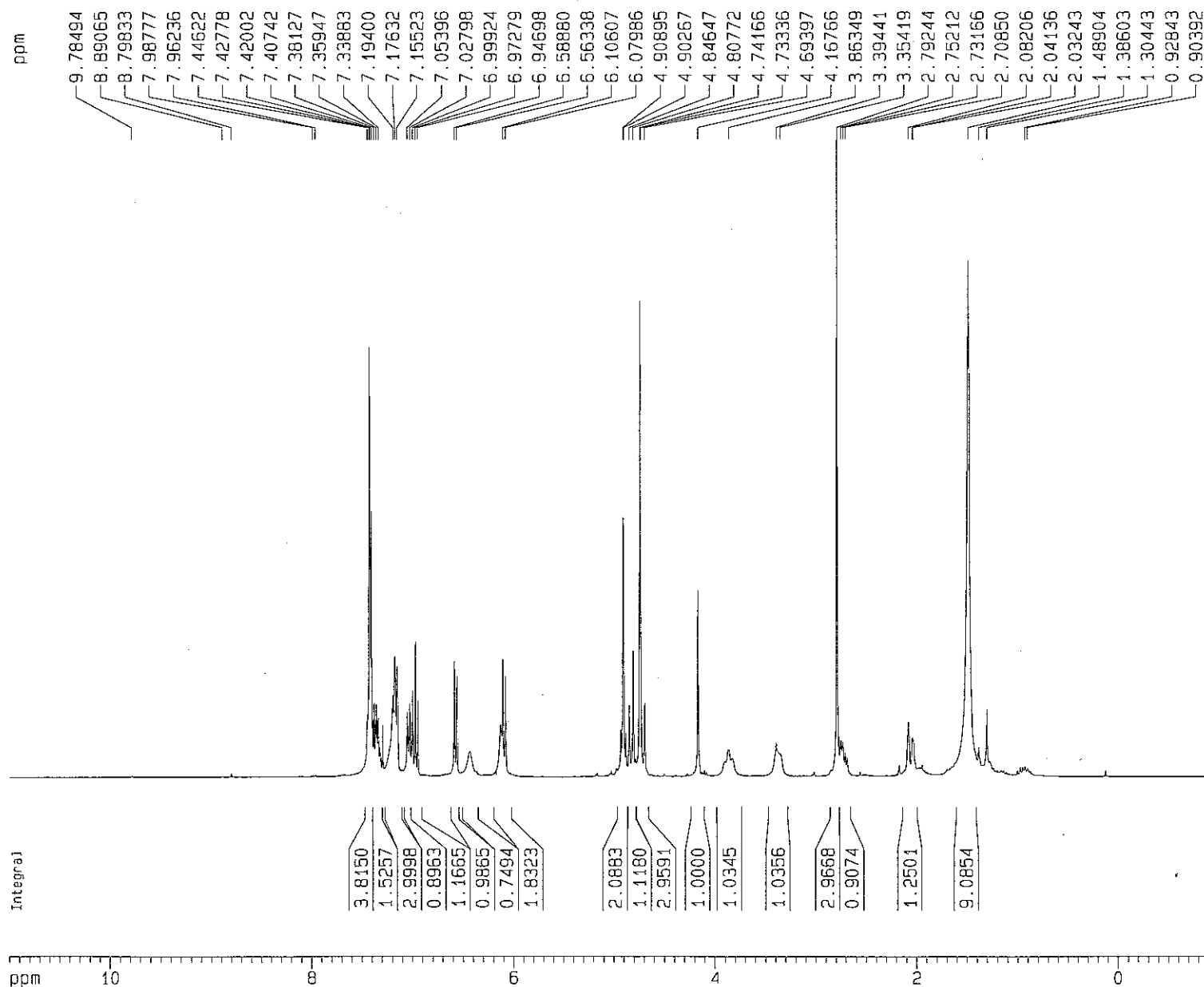
----- GRADIENT CHANNEL -----  
GPNAM1 sine.100  
GPNAM2 sine.100  
GPX1 0.00 %  
GPX2 0.00 %  
GPY1 0.00 %  
GPY2 0.00 %  
GPZ1 40.00 %  
GPZ2 -40.00 %  
P16 1000.00 usec

F2 - Processing parameters  
SI 2048  
SF 400.1300092 MHz  
WDW QSINE  
SSB 2  
LB 0.00 Hz  
GB 0  
PC 1.00

1D NMR plot parameters  
CX 20.00 cm  
F1P 11.000 ppm  
F1 4401.43 Hz  
F2P -1.000 ppm  
F2 -400.13 Hz  
PPHCH 0.60000 ppm/cm  
HZCH 240.07800 Hz/cm



48



## Current Data Parameters

NAME PL-Ju130-09  
EXPNO 6  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20090730  
Time 20.34  
INSTRUM spect  
PROBHD 5 mm GNP 1H/1  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6172.839 Hz  
FIDRES 0.094190 Hz  
AQ 5.3084660 sec  
RG 90.5  
DW 81.000 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec

## ===== CHANNEL f1 =====

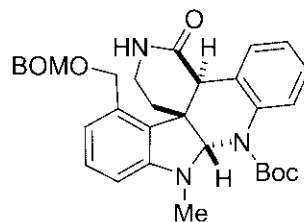
NUC1 1H  
P1 11.70 usec  
PL1 0.00 dB  
SFO1 299.8718518 MHz

## F2 - Processing parameters

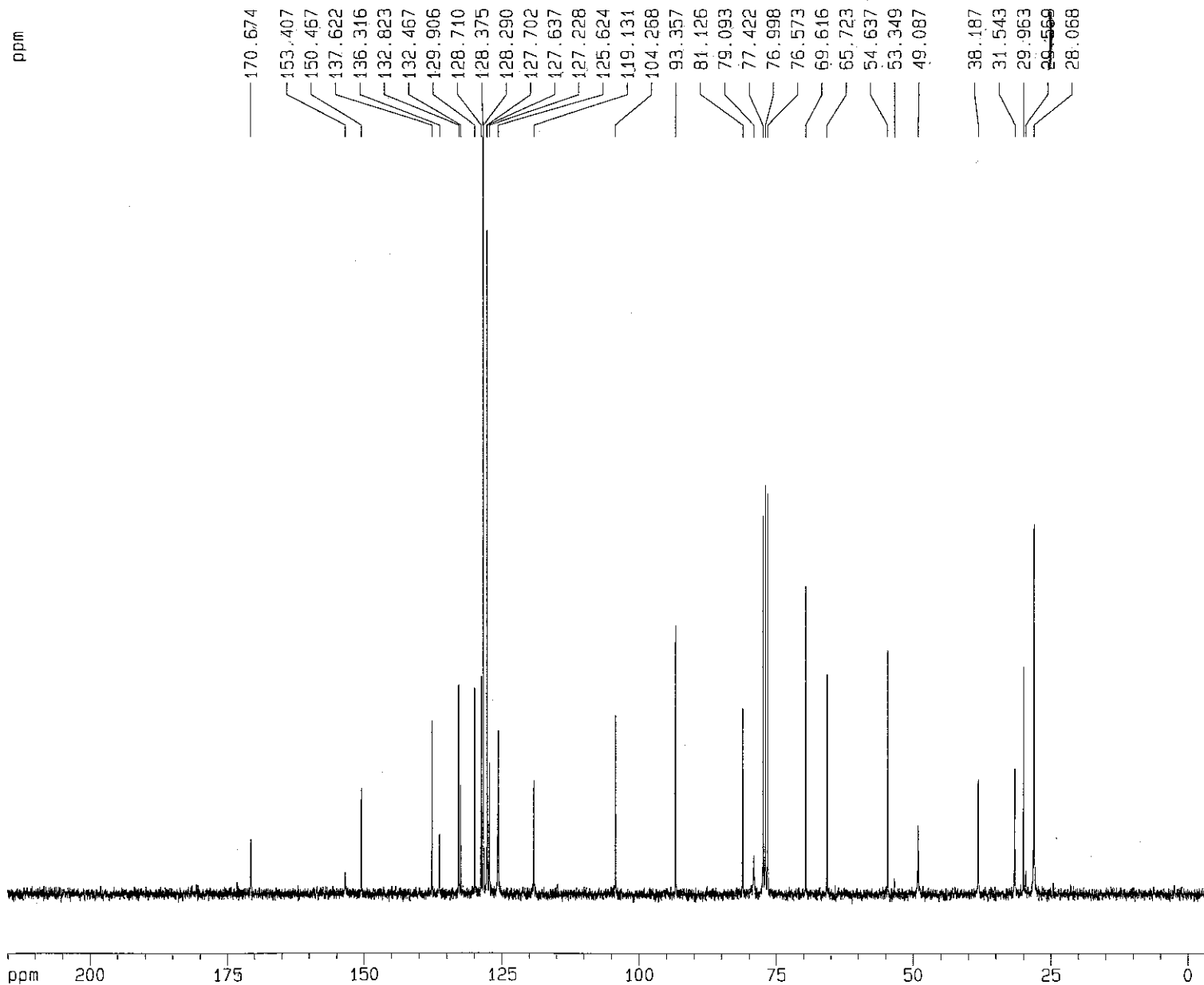
SI 32768  
SF 299.8700000 MHz  
WDW no  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

## 1D NMR plot parameters

CX 20.00 cm  
F1P 11.000 ppm  
F1 3298.57 Hz  
F2P -1.000 ppm  
F2 -299.87 Hz  
PPMCM 0.60000 ppm/cm  
HZCM 179.92200 Hz/cm



48



Current Data Parameters  
 NAME PL-Ju124-09  
 EXPNO 1  
 PROCNO 1

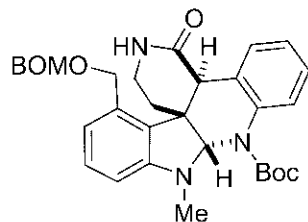
F2 - Acquisition Parameters  
 Date\_ 20090724  
 Time 13.47  
 INSTRUM spect  
 PROBHD 5 mm GNP 1H/1  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDC13  
 NS 401  
 DS 4  
 SWH 18796.992 Hz  
 FIDRES 0.286819 Hz  
 AQ 1.7433076 sec  
 RG 812.7  
 DW 26.600 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 2.00000000 sec  
 D11 0.03000000 sec  
 D12 0.00002000 sec

----- CHANNEL f1 -----  
 NUC1 13C  
 P1 5.40 usec  
 PL1 -6.00 dB  
 SFO1 75.4106357 MHz

----- CHANNEL f2 -----  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 115.00 usec  
 PL2 0.00 dB  
 PL12 20.00 dB  
 PL13 20.00 dB  
 SFO2 299.8711995 MHz

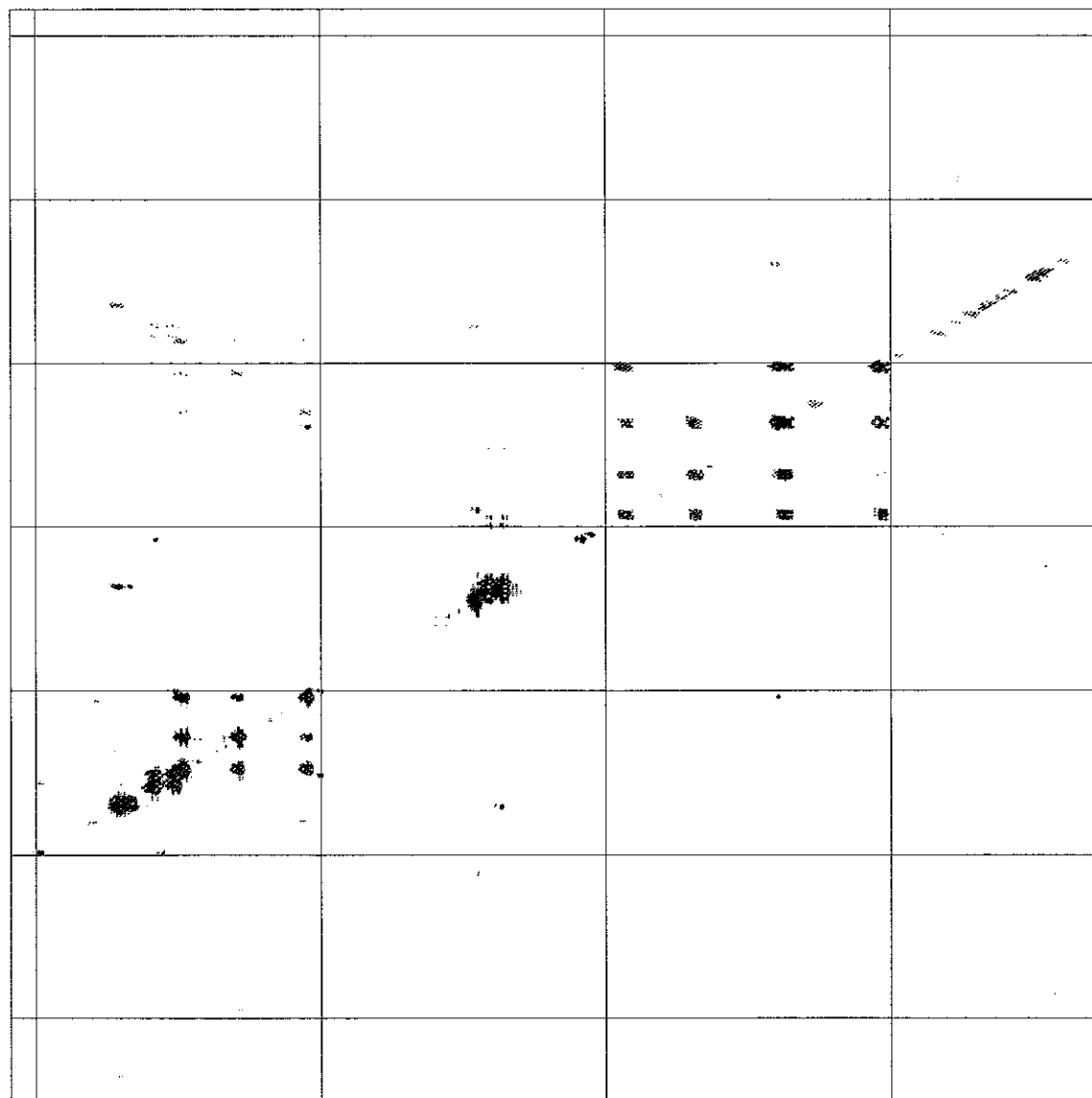
F2 - Processing parameters  
 SI 32768  
 SF 75.4023549 MHz  
 HDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 215.000 ppm  
 F1 16211.51 Hz  
 F2P -5.000 ppm  
 F2 -377.01 Hz  
 PPMCM 11.00000 ppm/cm  
 HZCM 829.42621 Hz/cm



48

COSY

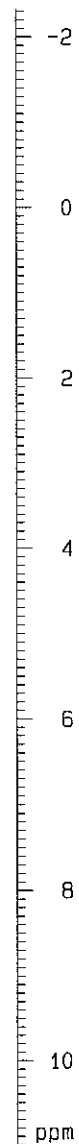


ppm

6

4

2



Current Data Parameters  
 NAME PL-Aug02-09  
 EXPNO 3  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20090802  
 Time 12.49  
 INSTRUM spect  
 PROBHD 5 mm BBI 1H-B  
 PULPROG ajb.cosygmftp  
 TO 2048  
 SOLVENT CDCl3  
 NS 4  
 DS 8  
 SWH 3063.726 Hz  
 FIDRES 1.495960 Hz  
 AQ 0.3342836 sec  
 RG 4597.6  
 OW 163.200 usec  
 OE 6.00 usec  
 TE 300.0 K  
 D0 0.00000300 sec  
 D1 4.00000000 sec  
 d13 0.00000300 sec  
 D16 0.00020000 sec  
 d20 0.00120300 sec  
 INO 0.0009360 sec

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 7.39 usec  
 P2 14.78 usec  
 PL1 0.00 dB  
 SFO1 400.1317398 MHz

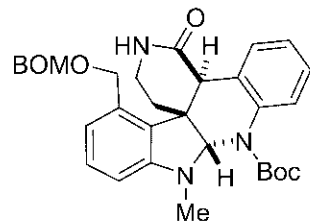
===== GRADIENT CHANNEL =====  
 P16 1000.00 usec

F1 - Acquisition parameters  
 NDO 2  
 TO 650  
 SFO1 400.1317 MHz  
 FIDRES 8.216277 Hz  
 SW 13.350 ppm

F2 - Processing parameters  
 SI 2048  
 SF 400.1300000 MHz  
 WDW SINE  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.40

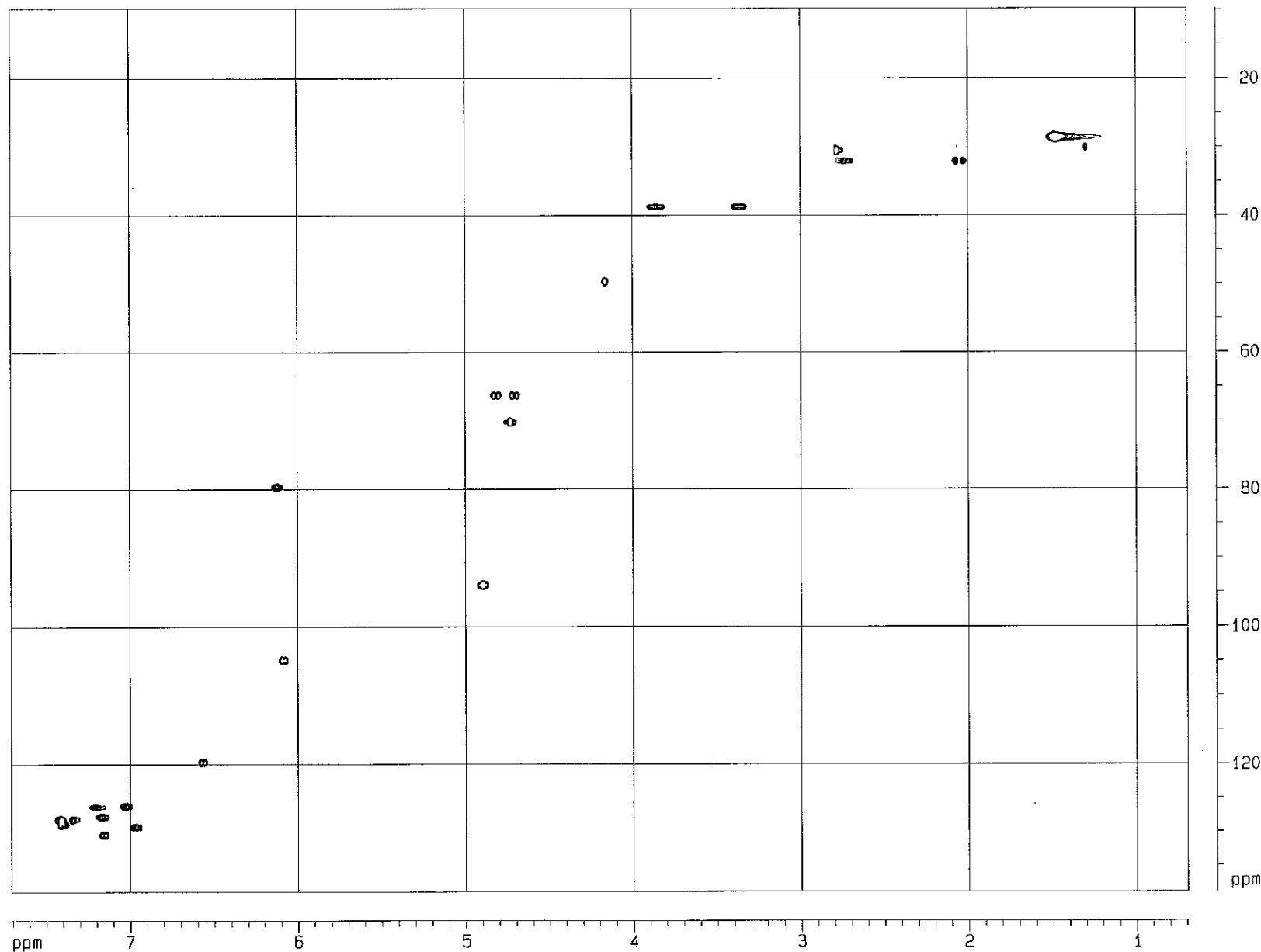
F1 - Processing parameters  
 SI 1024  
 MC2 TPPI  
 SF 400.1300000 MHz  
 WDW SINE  
 SSB 0  
 LB 0.00 Hz  
 GB 0

2D NMR plot parameters  
 CX2 15.00 cm  
 CX1 15.00 cm  
 F2PL0 8.176 ppm  
 F2L0 3271.63 Hz  
 F2PHI 0.520 ppm  
 F2HI 207.90 Hz  
 F1PL0 11.023 ppm  
 F1L0 4410.71 Hz  
 F1PHI -2.327 ppm  
 F1HI -831.16 Hz  
 F2PMCH 0.51046 ppm/cm  
 F2HZC 204.24837 Hz/cm  
 F1PMCH 0.85002 ppm/cm  
 F1HZC 356.12537 Hz/cm



HMQC

48



Current Data Parameters  
 NAME PL-Aug02-09  
 SAMPID 2  
 PRDCNO 1

F2 - Acquisition Parameters  
 Date\_ 20090802  
 Time 12.02  
 INSTRUM spect  
 PROBHD 5 mm BBI 1H-2  
 PULPROG InvAgg16  
 TO 2045  
 SOLVENT CDCl3  
 NS 4  
 DS 80  
 SWH 2063.726 Hz  
 FIDRES 1.425901 Hz  
 AQ 0.3342836 sec  
 RG 7288.2  
 RW 163.200 usec  
 DE 6.00 usec  
 TE 300.0 K  
 CNSTE 145.000000  
 d0 0.0000000 sec  
 d1 1.9951298 sec  
 d2 0.0004428 sec  
 d4 0.00172414 sec  
 d11 0.0300000 sec  
 d13 0.0000000 sec  
 d16 0.0000000 sec  
 d20 0.00052414 sec  
 d21 0.00224428 sec  
 INO 0.00001490 sec

----- CHANNEL f1 -----  
 NUC1 1H  
 P1 7.35 usec  
 p2 14.78 usec  
 PL1 0.00 dB  
 SF01 400.1317598 MHz

----- CHANNEL f2 -----  
 CPDPRG2 gprp  
 NUC2 13C  
 P3 17.00 usec  
 p4 34.00 usec  
 PPRG2 64.00 usec  
 PL2 -5.00 dB  
 PL12 6.30 dB  
 SFO2 100.6202332 MHz

----- GRADIENT CHANNEL -----  
 GPMAX1 SINE.100  
 GPMAX2 SINE.100  
 GPMAX3 SINE.100  
 GPX1 17.00 %  
 GPX2 30.00 %  
 GPX3 25.00 %  
 GPY1 17.00 %  
 GPY2 20.00 %  
 GPY3 25.00 %  
 GPZ1 17.00 %  
 GPZ2 20.00 %  
 GPZ3 25.00 %  
 P16 1000.00 usec

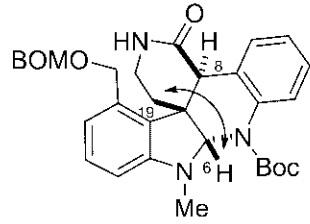
F1 - Acquisition parameters  
 MD 4  
 TD 256  
 SFO1 100.6202332 MHz  
 FIDRES 65.541907 Hz  
 SK 188.751 ppm

F2 - Processing parameters  
 SI 2045  
 SF 400.1300000 MHz  
 KW OSINE  
 SSB 2  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

F1 - Processing parameters  
 S1 1024  
 MC2 TPPI  
 SF 100.6127780 MHz  
 KW OSINE  
 SSB 2  
 LB 0.00 Hz  
 GB 0

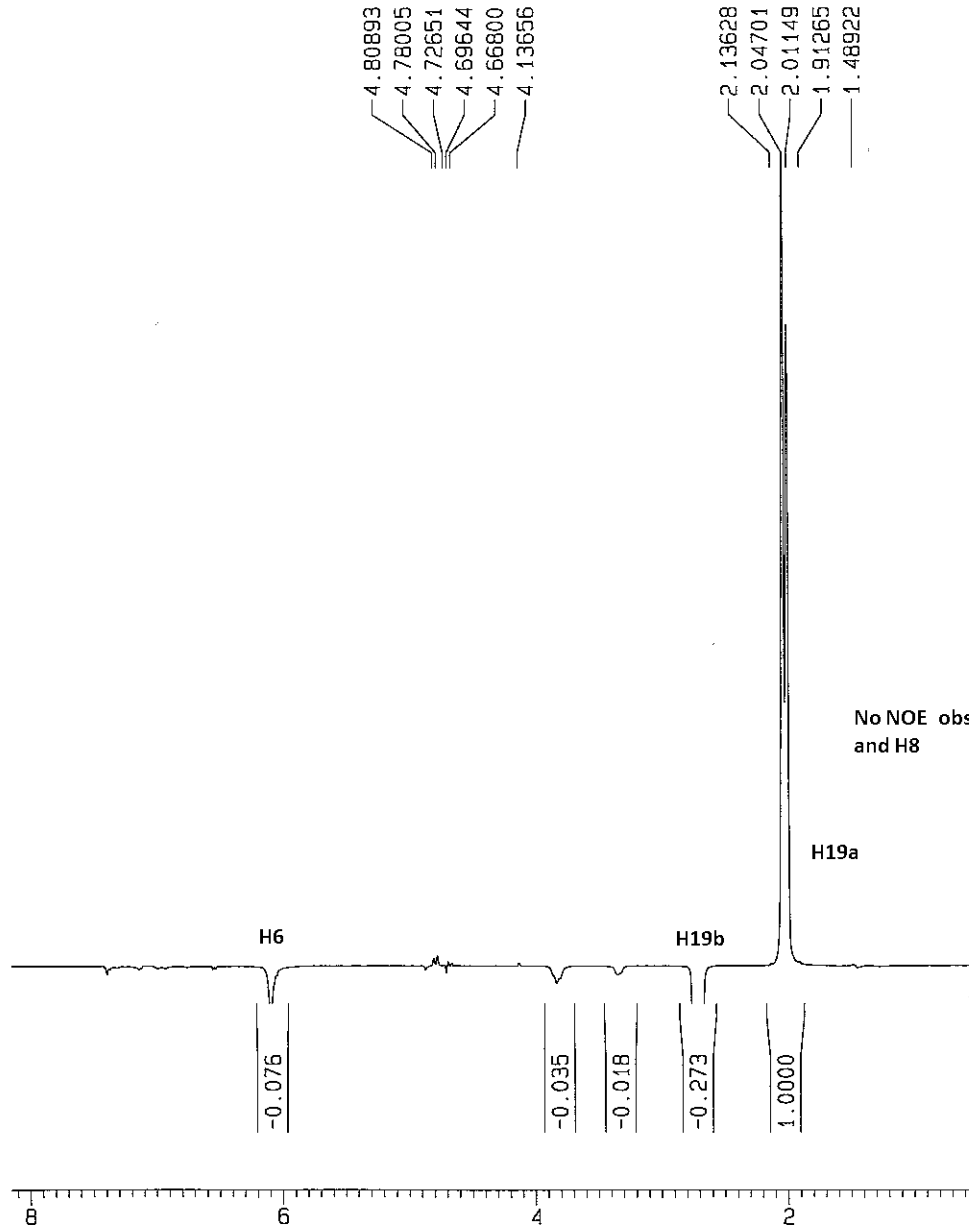
2D NMR plot parameters  
 CX2 20.00 cm  
 CX1 16.00 cm  
 F2PL0 7.769 ppm  
 F2LD 3084.63 Hz  
 F2PHI 0.655 ppm  
 F2H1 278.21 Hz  
 F1PL0 138.913 ppm  
 F1LD 13076.37 Hz  
 F1PHI 0.765 ppm  
 F1H1 982.65 Hz  
 F2PPMCH 0.35885 ppm/cm  
 F2AZCM 140.34101 Hz/cm  
 F1PPMCH 6.60560 ppm/cm  
 F1AZCM 656.23505 Hz/cm

ppm



48

4.80893  
4.78005  
4.72651  
4.69644  
4.66800  
4.13656  
2.13628  
2.04701  
2.01149  
1.91265  
1.48922



Current Data Parameters  
NAME PL-Aug02-09  
EXPNO 4  
PROCNO 6

F2 - Acquisition Parameters  
Date\_ 20090802  
Time 16.00  
INSTRUM spect  
PROBHD 5 mm BBI 1H-B  
PULPROG noesygtp  
TD 2048  
SOLVENT CDC13  
NS 8  
DS 8  
SWH 3063.726 Hz  
FIDRES 1.495960 Hz  
AQ 0.3342836 sec  
RG 71.8  
DW 163.200 usec  
DE 6.00 usec  
TE 300.0 K  
d0 0.0000300 sec  
D1 4.0000000 sec  
D8 0.6000001 sec  
D16 0.0002000 sec  
d20 0.39880002 sec  
IN0 0.00012496 sec

===== CHANNEL f1 =====  
NUC1 1H  
P1 7.39 usec  
P2 14.78 usec  
PL1 0.00 dB  
SFO1 400.1317398 MHz

===== GRADIENT CHANNEL =====  
GPNAM1 sine.100  
GPNAM2 sine.100  
GPX1 0.00 %  
GPX2 0.00 %  
GPY1 0.00 %  
GPY2 0.00 %  
GPZ1 40.00 %  
GPZ2 -40.00 %  
P16 1000.00 usec

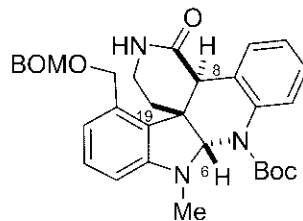
F2 - Processing parameters  
SI 2048  
SF 400.130092 MHz  
WDW QSINE  
SSB 2  
LB 0.00 Hz  
GB 0  
PC 1.00

1D NMR plot parameters  
CX 20.00 cm  
F1P 11.000 ppm  
F1 4401.43 Hz  
F2P -1.000 ppm  
F2 -400.13 Hz  
PPMCM 0.60000 ppm/cm  
HZCM 240.07800 Hz/cm

ppm

Integral

ppm



48

ppm

6.99197

4.24514

4.13849

2.01308

H8

No significant NOE observed  
between H8 and H6, H8 and H19a

-0.005

-0.079

-0.007

-0.039

-0.043

1.0000

-0.025

ppm

8

6

4

2

Current Data Parameters  
NAME PL-Aug02-09  
EXPNO 4  
PROCNO 2

F2 - Acquisition Parameters  
Date\_ 20090802  
Time 16.00  
INSTRUM spect  
PROBHD 5 mm BBI 1H-8  
PULPROG noesygptp  
TD 2048  
SOLVENT CDCl3  
NS 8  
DS 6  
SWH 3063.726 Hz  
FIDRES 1.495960 Hz  
AQ 0.3342636 sec  
RG 71.6  
DW 163.200 usec  
DE 6.00 usec  
TE 300.0 K  
d0 0.0000300 sec  
D1 4.0000000 sec  
DB 0.80000001 sec  
D16 0.00020000 sec  
d20 0.39880002 sec  
INO 0.00012496 sec

===== CHANNEL f1 =====  
NUC1 1H  
P1 7.39 usec  
P2 14.78 usec  
PL1 0.00 dB  
SFO1 400.1317396 MHz

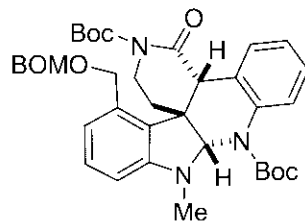
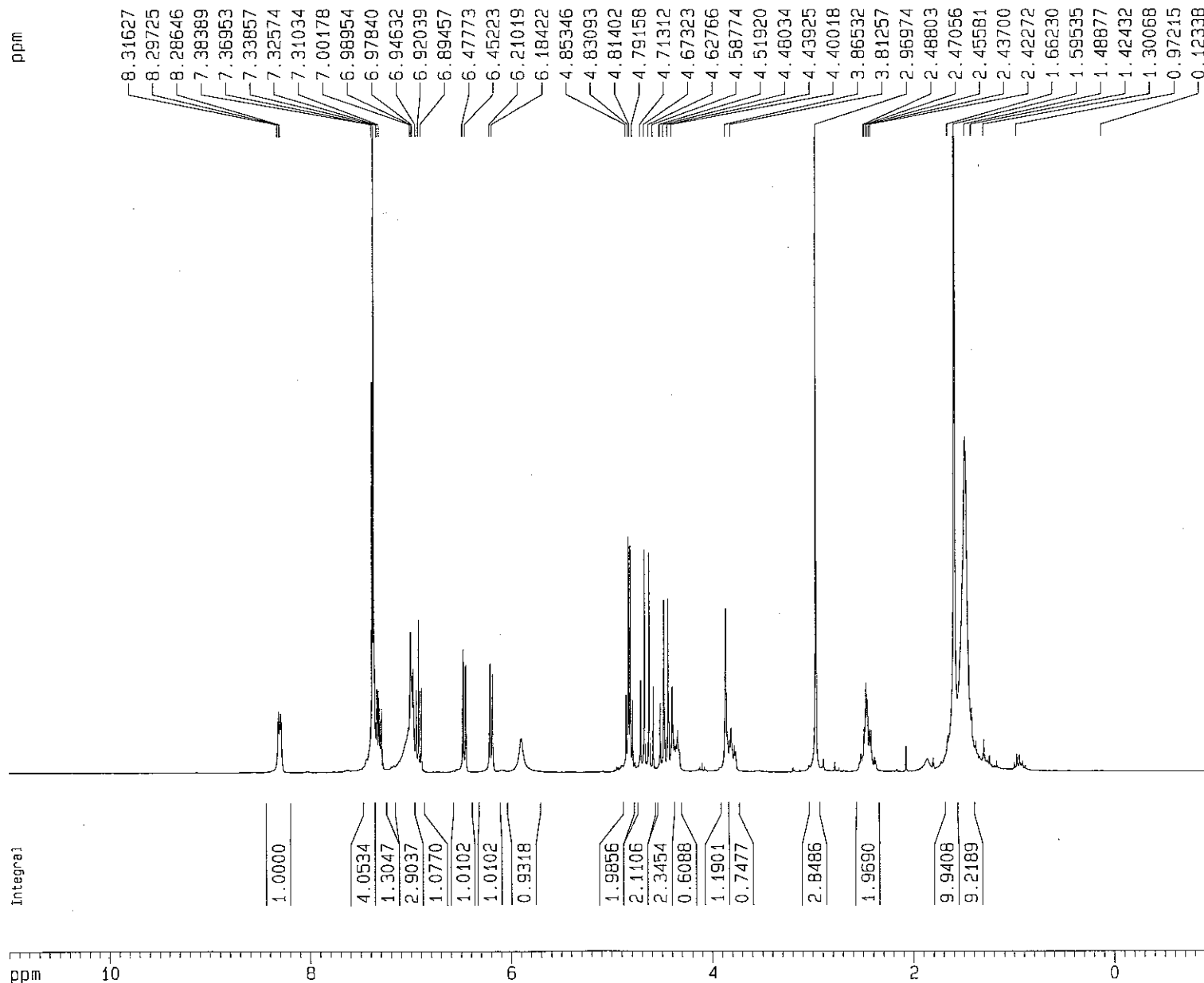
===== GRADIENT CHANNEL =====  
GPNAM1 sine.100  
GPNAM2 sine.100  
GPX1 0.00 %  
GPX2 0.00 %  
GPY1 0.00 %  
GPY2 0.00 %  
GPZ1 40.00 %  
GPZ2 -40.00 %  
P16 1000.00 usec

F2 - Processing parameters  
SI 2048  
SF 400.1300092 MHz  
WDW QSINE  
SSB 2  
LB 0.00 Hz  
GB 0  
PC 1.00

1D NMR plot parameters  
CX 20.00 cm  
F1P 11.000 ppm  
F1 4401.43 Hz  
F2P -1.000 ppm  
F2 -400.13 Hz  
PPMCM 0.60000 ppm/cm  
HZCM 240.07800 Hz/cm

Integral



49  
minor

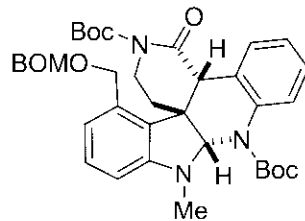
Current Parameters  
 NAME PL-Jul30-09  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20090730  
 Time 15.39  
 INSTRUM spect  
 PROBHD 5 mm QNP 1H/1  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 16  
 DS 2  
 SWH 6172.839 Hz  
 FIDRES 0.094190 Hz  
 AQ 5.3084660 sec  
 RG 64  
 DW 81.000 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 1.00000000 sec

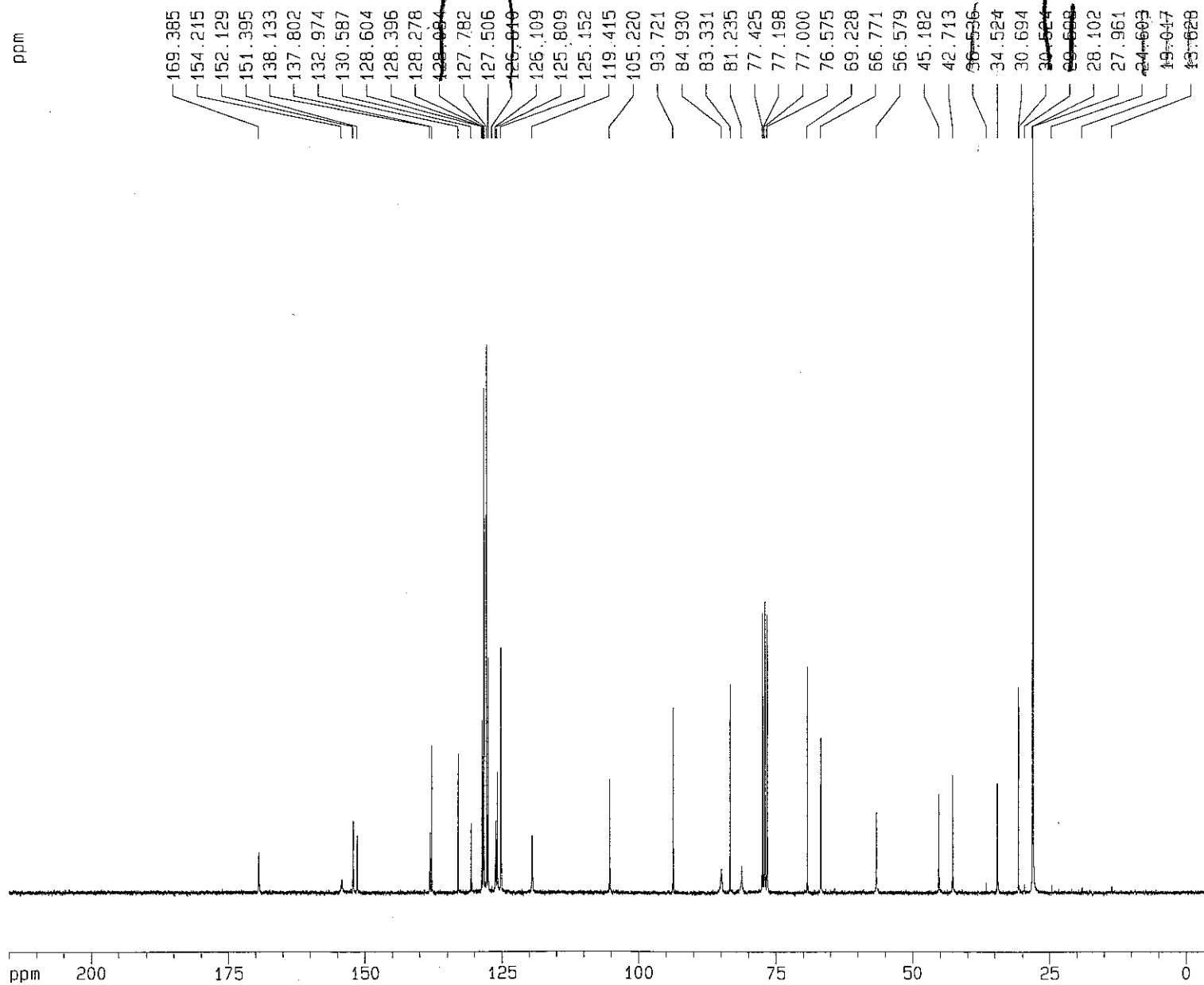
===== CHANNEL f1 =====  
 NUC1 1H  
 P1 11.70 usec  
 PL1 0.00 dB  
 SFO1 299.8718518 MHz

F2 - Processing parameters  
 SI 32768  
 SF 299.8700000 MHz  
 WDW no  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 11.000 ppm  
 F1 3298.57 Hz  
 F2P -1.000 ppm  
 F2 -299.87 Hz  
 PPMCM 0.60000 ppm/cm  
 HZCM 179.92200 Hz/cm



49  
minor



Current Data Parameters  
NAME PL-Jul30-09  
EXPNO 2  
PROCNO 1

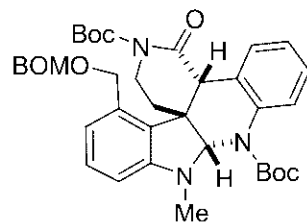
F2 - Acquisition Parameters  
Date\_ 20090730  
Time 18.03  
INSTRUM spect  
PROBHD 5 mm GNP 1H/1  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 2271  
DS 4  
SWH 18796.992 Hz  
FIDRES 0.286819 Hz  
AQ 1.7433076 sec  
RG 812.7  
DW 26.600 usec  
DE 6.00 usec  
TE 300.0 K  
D1 2.0000000 sec  
D11 0.0300000 sec  
D12 0.0002000 sec

===== CHANNEL f1 =====  
NUC1 13C  
P1 5.40 usec  
PL1 -6.00 dB  
SF01 75.4106357 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 115.00 usec  
PL2 0.00 dB  
PL12 20.00 dB  
PL13 20.00 dB  
SF02 299.8711995 MHz

F2 - Processing parameters  
SI 32768  
SF 75.4023820 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

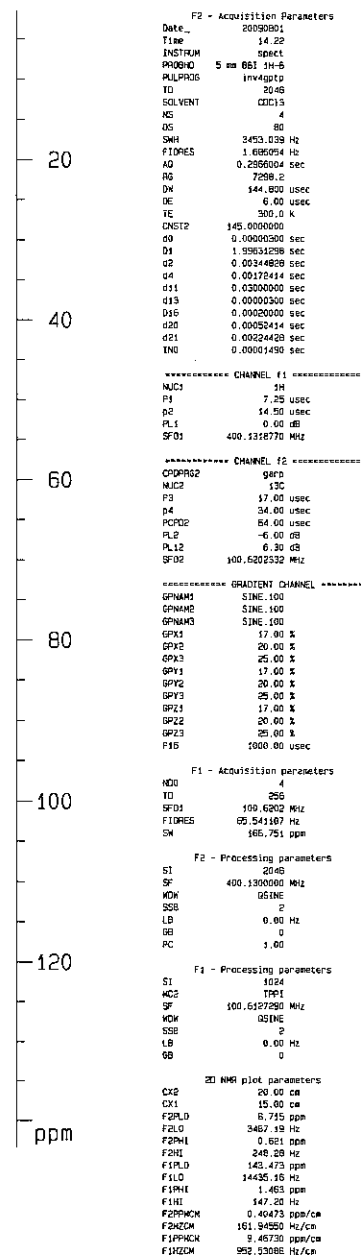
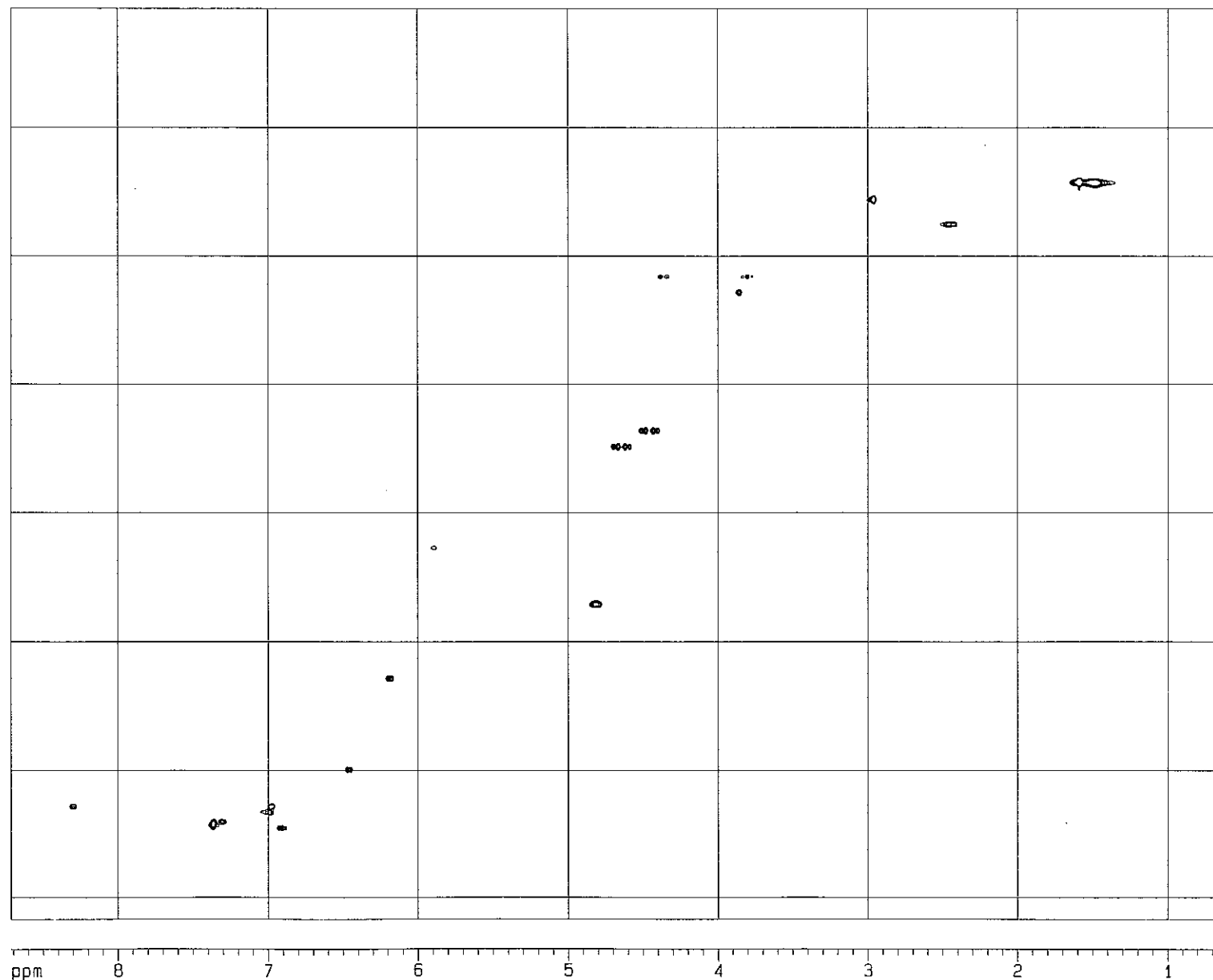
1D NMR plot parameters  
CX 20.00 cm  
F1P 215.000 ppm  
F1 16211.51 Hz  
F2P -5.000 ppm  
F2 -377.01 Hz  
PPMCM 11.00000 ppm/cm  
HZCM 829.42621 Hz/cm

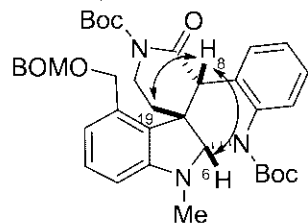


HMQC

49  
minor

INV46STP

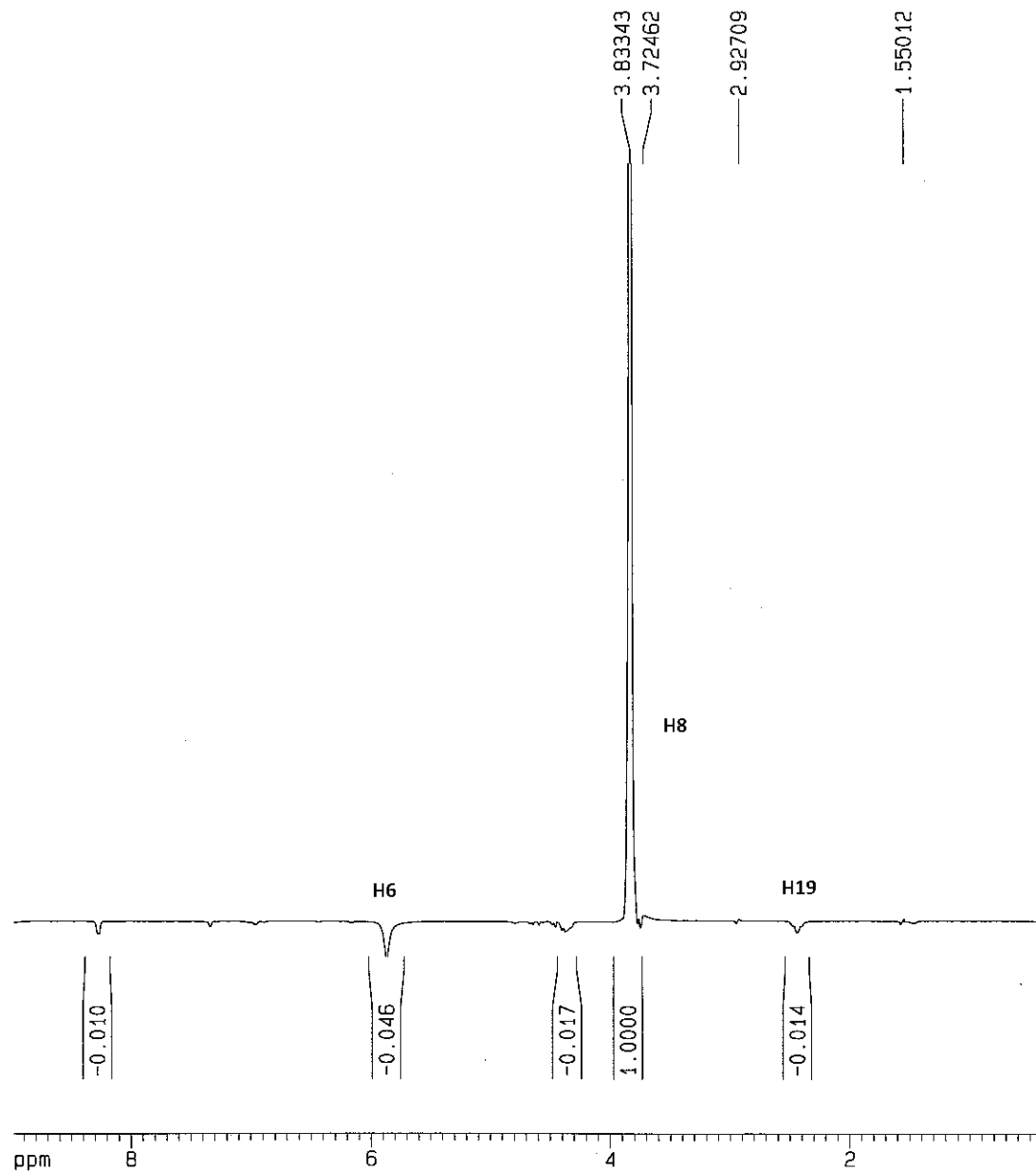




NOESY

49  
minor

ppm



Current Data Parameters  
 NAME PL-Aug01-09  
 EXPNO 3  
 PROCNO 6

F2 - Acquisition Parameters  
 Date\_ 20090801  
 Time 15.03  
 INSTRUM spect  
 PROBHD 5 mm BBI 1H-8  
 PULPROG noesygtp  
 TD 2048  
 SOLVENT CDCl3  
 NS 8  
 DS 8  
 SWH 3453.039 Hz  
 FIDRES 1.686054 Hz  
 AQ 0.2966004 sec  
 RG 64  
 DW 144.800 usec  
 DE 6.00 usec  
 TE 300.0 K  
 d0 0.0000300 sec  
 D1 4.0000000 sec  
 DB 0.8000001 sec  
 D16 0.0002000 sec  
 d20 0.39880002 sec  
 IN0 0.00012496 sec

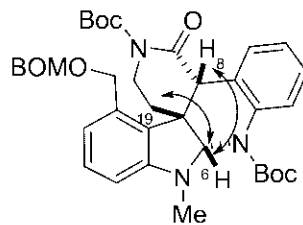
===== CHANNEL f1 =====  
 NUC1 1H  
 P1 7.25 usec  
 P2 14.50 usec  
 PL1 0.00 dB  
 SF01 400.1318770 MHz

===== GRADIENT CHANNEL =====  
 GPNAM1 sine.100  
 GPNAM2 sine.100  
 GPX1 0.00 %  
 GPX2 0.00 %  
 GPY1 0.00 %  
 GPY2 0.00 %  
 GPZ1 40.00 %  
 GPZ2 -40.00 %  
 P16 1000.00 usec

F2 - Processing parameters  
 SI 2048  
 SF 400.130092 MHz  
 WDW QSINE  
 SSB 2  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 11.000 ppm  
 F1 4401.43 Hz  
 F2P -1.000 ppm  
 F2 -400.13 Hz  
 PPMCM 0.60000 ppm/cm  
 HZCM 240.07800 Hz/cm

Integral

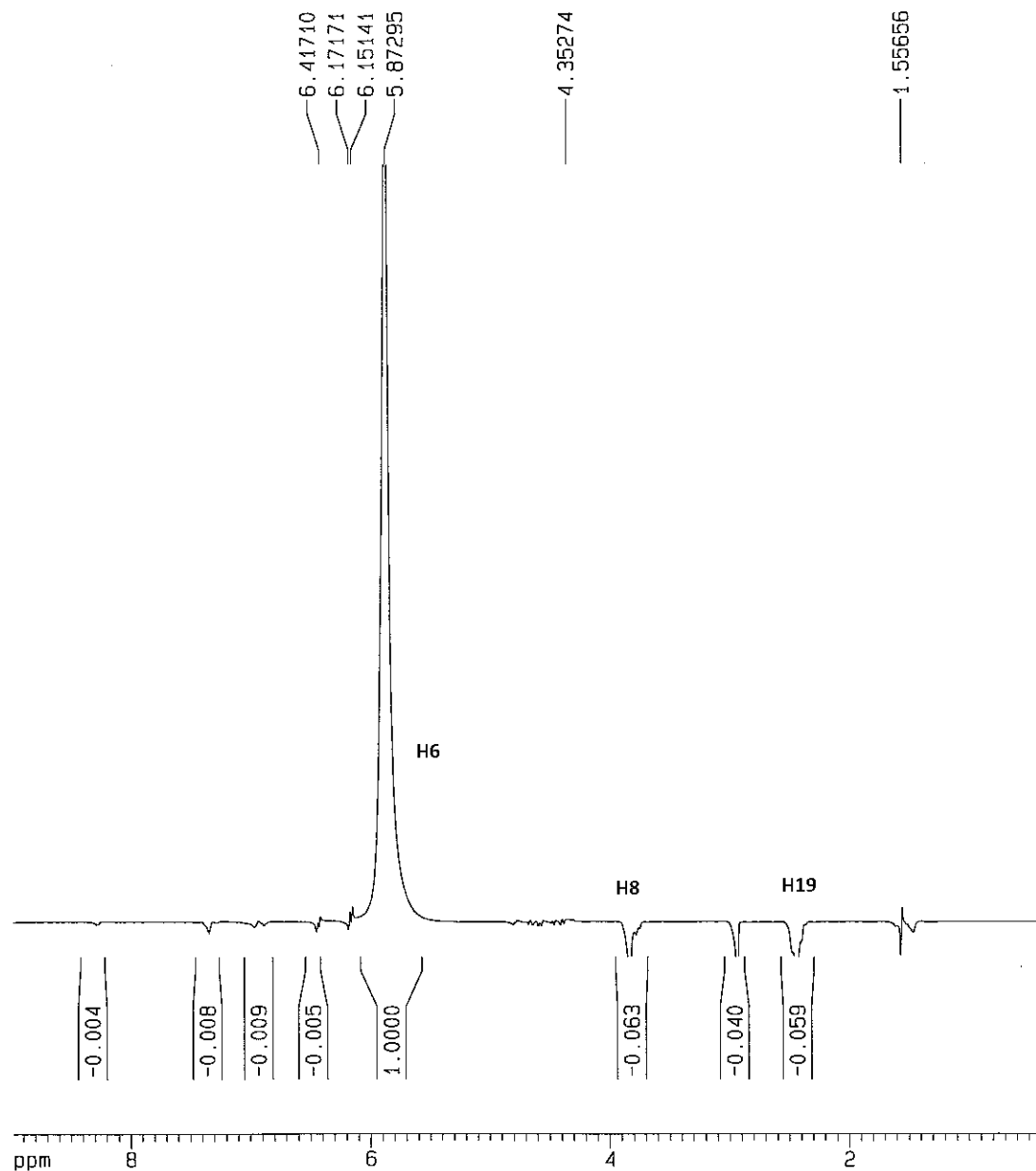


NOESY

49  
minor

ppm

Integral



Current Data Parameters  
NAME PL-Aug01-09  
EXPNO 3  
PROCNO 2

## F2 - Acquisition Parameters

Date\_ 20090801  
Time 15.03  
INSTRUM spect  
PROBHD 5 mm BBI 1H-6  
PULPROG noesygptp  
TD 2048  
SOLVENT CDC13  
NS 8  
DS 8  
SWH 3453.039 Hz  
FIDRES 1.686054 Hz  
AQ 0.2966004 sec  
RG 64  
DW 144.800 usec  
DE 6.00 usec  
TE 300.0 K  
d0 0.00000300 sec  
D1 4.00000000 sec  
D8 0.80000001 sec  
D16 0.00020000 sec  
d20 0.39880002 sec  
INO 0.00012496 sec

## ===== CHANNEL f1 =====

NUC1 1H  
P1 7.25 usec  
P2 14.50 usec  
PL1 0.00 dB  
SF01 400.1318770 MHz

## ===== GRADIENT CHANNEL =====

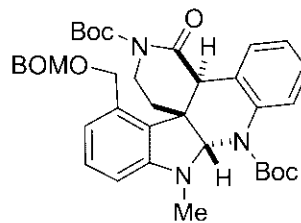
GPNAME1 sine.100  
GPNAME2 sine.100  
GPX1 0.00 %  
GPX2 0.00 %  
GPY1 0.00 %  
GPY2 0.00 %  
GPZ1 40.00 %  
GPZ2 -40.00 %  
P15 1000.00 usec

## F2 - Processing parameters

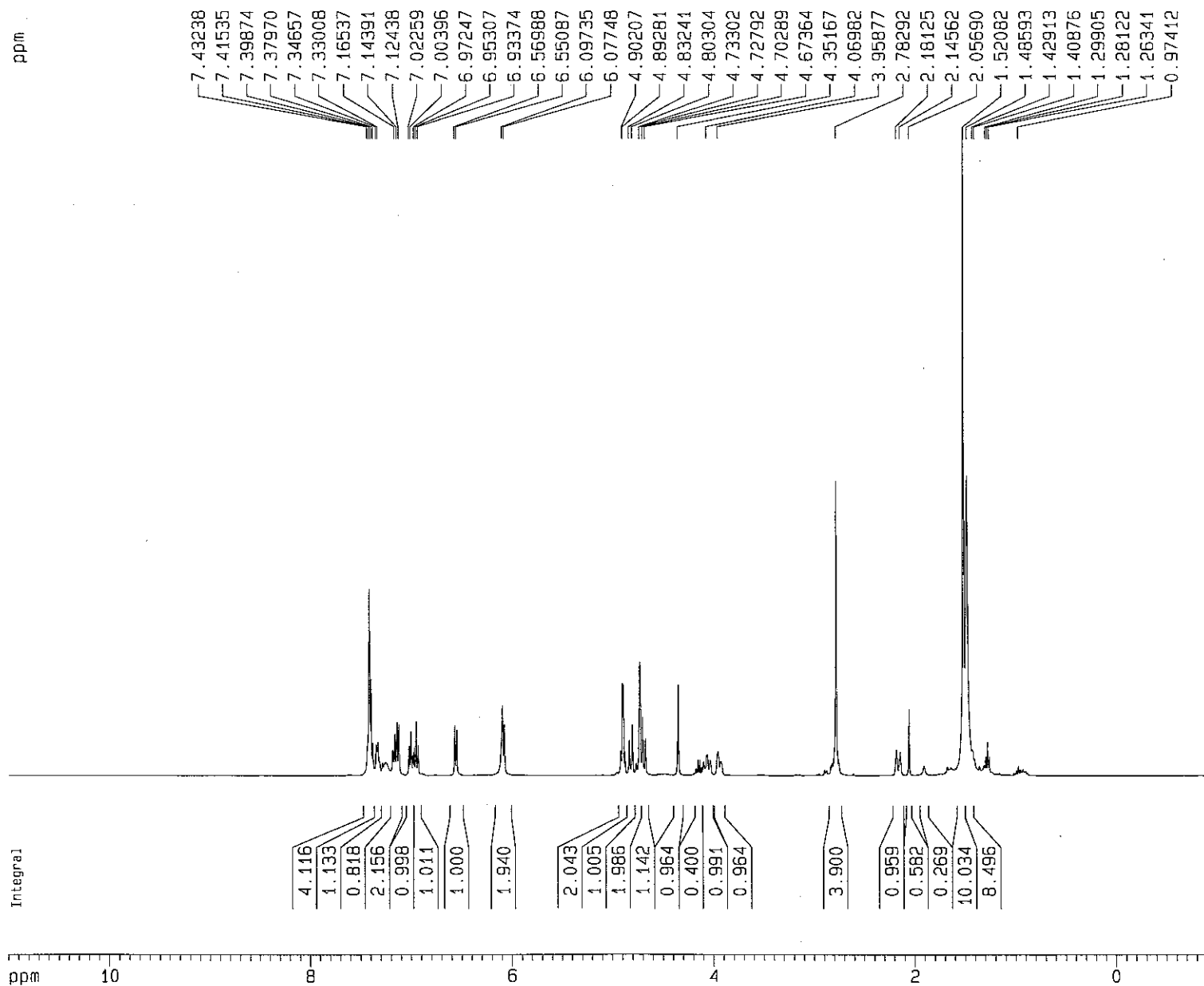
SI 2048  
SF 400.1300092 MHz  
WDW QSINE  
SSB 2  
LB 0.00 Hz  
GB 0

## 1D NMR plot parameters

CX 20.00 cm  
F1P 11.000 ppm  
F1 4401.43 Hz  
F2P -1.000 ppm  
F2 -400.13 Hz  
PPMCM 0.60000 ppm/cm  
HZCM 240.07800 Hz/cm



49  
major



## Current Data Parameters

NAME PL-Jul29-09  
EXPNO 2  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20090729  
Time 13.25  
INSTRUM spect  
PROBHD 5 mm BBI 1H-8  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8278.146 Hz  
FIDRES 0.126314 Hz  
AQ 3.9584243 sec  
RG 16  
DW 60.400 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec

## ===== CHANNEL f1 =====

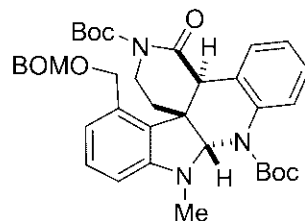
NUC1 1H  
P1 6.45 usec  
PL1 0.00 dB  
SFO1 400.1324710 MHz

## F2 - Processing parameters

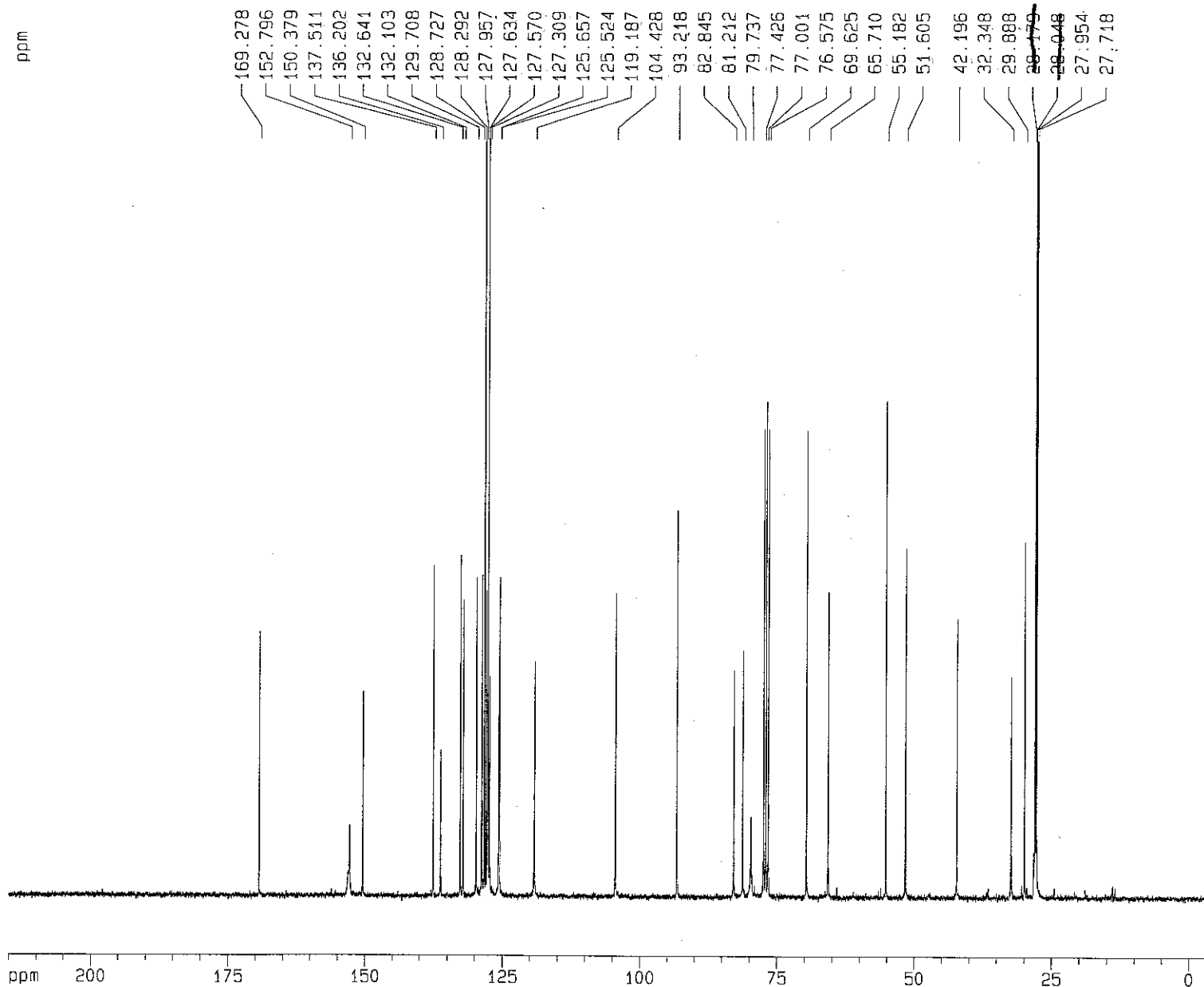
SI 32768  
SF 400.1300000 MHz  
WDW no  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

## 1D NMR plot parameters

CX 20.00 cm  
F1P 11.000 ppm  
F1 4401.43 Hz  
F2P -1.000 ppm  
F2 -400.13 Hz  
PPMCM 0.60000 ppm/cm  
HZCM 240.07800 Hz/cm



49  
major



Current Data Parameters  
NAME PL-Jul130-09  
EXPNO 5  
PROCNO 1

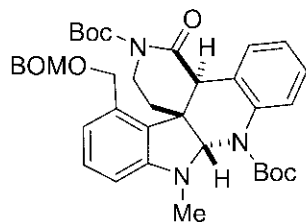
F2 - Acquisition Parameters  
Date\_ 20090730  
Time 19.15  
INSTRUM spect  
PROBHD 5 mm GNP 1H/1  
PULPROG zgpg30  
TD 65536  
SOLVENT CDC13  
NS 1553  
DS 4  
SWH 18796.992 Hz  
FIDRES 0.286819 Hz  
AQ 1.7433076 sec  
RG 4096  
DW 26.600 usec  
DE 6.00 usec  
TE 300.0 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
D12 0.00002000 sec

==== CHANNEL f1 =====  
NUC1 13C  
P1 5.40 usec  
PL1 -6.00 dB  
SF01 75.4106357 MHz

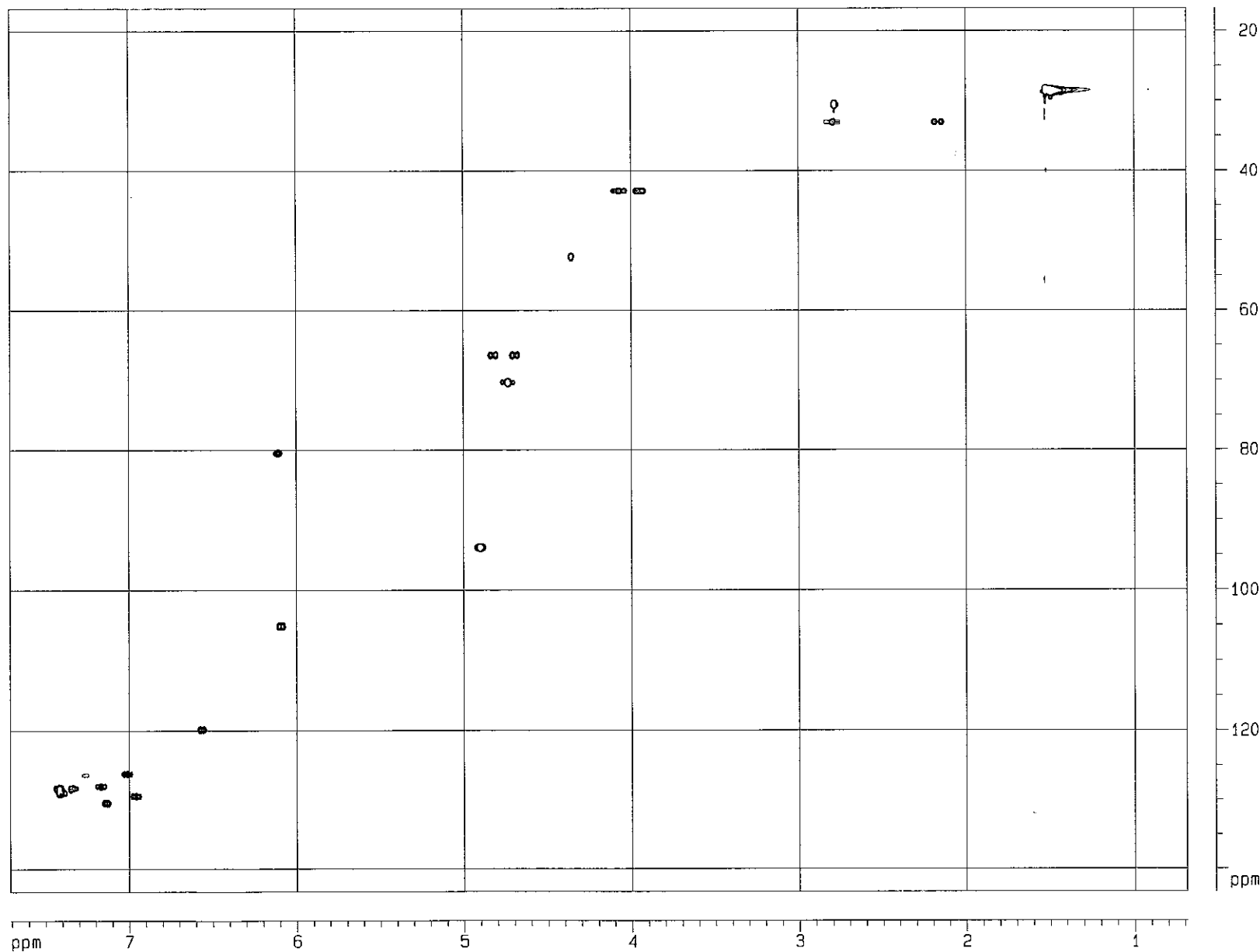
==== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 115.00 usec  
PL2 0.00 dB  
PL12 20.00 dB  
PL13 20.00 dB  
SF02 299.8711995 MHz

F2 - Processing parameters  
SI 32768  
SF 75.4023929 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

1D NMR plot parameters  
CX 20.00 cm  
F1P 215.000 ppm  
F1 16211.51 Hz  
F2P -5.000 ppm  
F2 -377.01 Hz  
PPMCM 11.00000 ppm/cm  
HZCM 829.42627 Hz/cm



HMQC

49  
major

```

Current Data Parameters
NAME      PL-Jul30-09
EXPNO    2
PROCNO   1

F2 - Acquisition Parameters
Date_    20090731
Time     20.01
INSTRUM  spect
PROBHD   5 mm BBI 1H-6
PULPROG  jvkgpzt
TD        2048
SOLVENT  CDCl3
NS        4
DS        60
SMH       3063.726 Hz
FIDRES   1.439960 Hz
AQ        0.334235 sec
RG         3545.1
DK        163.200 usec
DE         6.00 usec
TE        300.2 K
CQST2    145.000000
d0         0.0000300 sec
d1         1.99631298 sec
d2         0.00344808 sec
d4         0.00172404 sec
d11        0.0300000 sec
d13        0.0000300 sec
d16        0.0002000 sec
d20        0.0005414 sec
d21        0.0024426 sec
dN0        0.0001490 sec

===== CHANNEL f1 =====
NUC1      1H
P1         7.30 usec
P2        14.78 usec
PL1        0.00 dB
SFO1      400.1517534 MHz

===== CHANNEL f2 =====
CPDPRG2  gpgp
NUC2      13C
P3         17.00 usec
p4         34.00 usec
PCPD2     64.00 usec
PL2        -6.00 dB
PL12       6.30 dB
SFO2      100.6202332 MHz

===== GRADIENT CHANNEL =====
GRNAM1    SINE.100
GRNAM2    SINE.100
GRNAM3    SINE.100
GPX1       17.00 %
GPX2       20.00 %
GPX3       25.00 %
GPY1       17.00 %
GPY2       20.00 %
GPY3       25.00 %
GFZ1       17.00 %
GFZ2       20.00 %
GFZ3       25.00 %
P16        1000.00 usec

F1 - Acquisition parameters
N00        4
TD         256
SFO1      100.6202 MHz
FIDRES     65.541107 Hz
SN         166.751 ppm

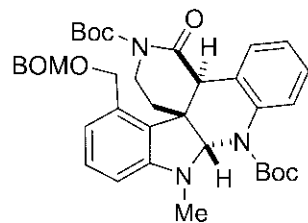
F2 - Processing parameters
SI         2048
SF         400.150000 MHz
WDW        0SINE
SSB        2
LB         0.00 Hz
GB         0
PC         1.00

F1 - Processing parameters
SI         1024
MC2        TRF1
SF         100.627290 MHz
WDW        0SINE
SSB        2
LB         0.00 Hz
GB         0

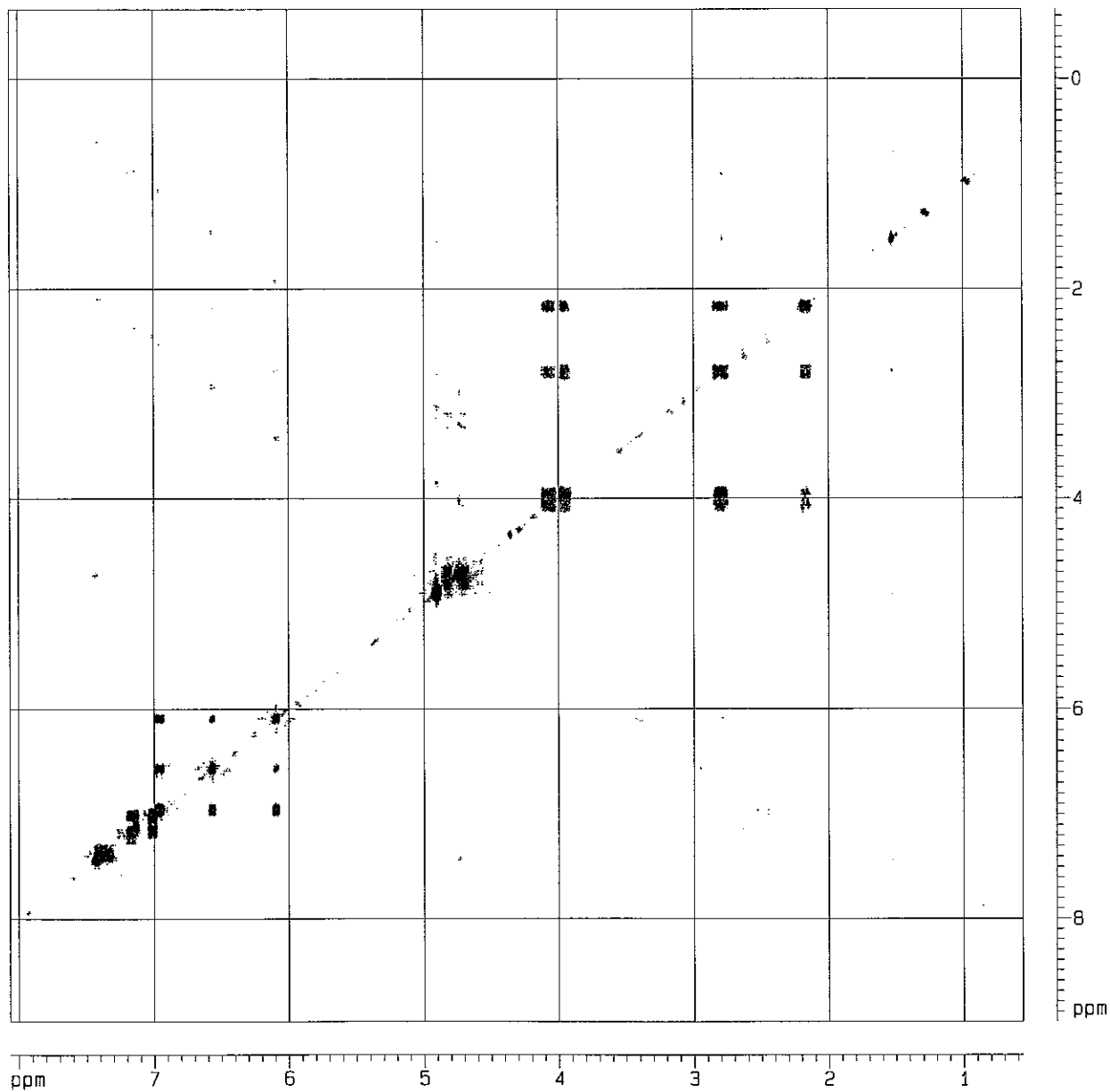
2D NMR plot parameters
CX2        20.00 cm
CX1        15.00 cm
FAPLD      7.709 ppm
FAPL0      3084.77 Hz
F2PH1     0.608 ppm
F2PH2     275.36 Hz
F1PLD     143.310 ppm
F1L0      14418.78 Hz
F1PH1     16.771 ppm
F1PH2     1587.42 Hz
F2MCM      0.35106 ppm/cm
F2M0M     140.47663 Hz/cm
F1MCM      8.42988 ppm/cm
F1M0M     648.75752 Hz/cm

```





49  
cosy major



Current Data Parameters  
NAME PL-Jul10-09  
EXPNO 4  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20090731  
Time 22.54  
INSTRUM spect  
PROBHD 5 mm BBI 1H-B  
PULPROG ajb.cosygmftp  
TD 2048  
SOLVENT CDCl3  
NS 4  
DS 0  
SWH 3063.726 Hz  
FIDRES 1.495960 Hz  
AQ 0.3342836 sec  
RG 2886.3  
DN 163.200 usec  
DE 6.00 usec  
TE 300.0 K  
D0 0.0000300 sec  
D1 4.0000000 sec  
d13 0.0000300 sec  
d16 0.0020000 sec  
d20 0.00120300 sec  
IN0 0.0009360 sec

----- CHANNEL f1 -----  
MUC1 jh  
P1 7.39 usec  
P2 14.78 usec  
PL1 0.00 dB  
SFO1 400.1317534 MHz

----- GRADIENT CHANNEL -----  
P16 1000.00 usec

F1 - Acquisition parameters  
ND0 2  
TD 650  
SFO1 400.1318 MHz  
FIDRES 8.218277 Hz  
SW 13.360 ppm

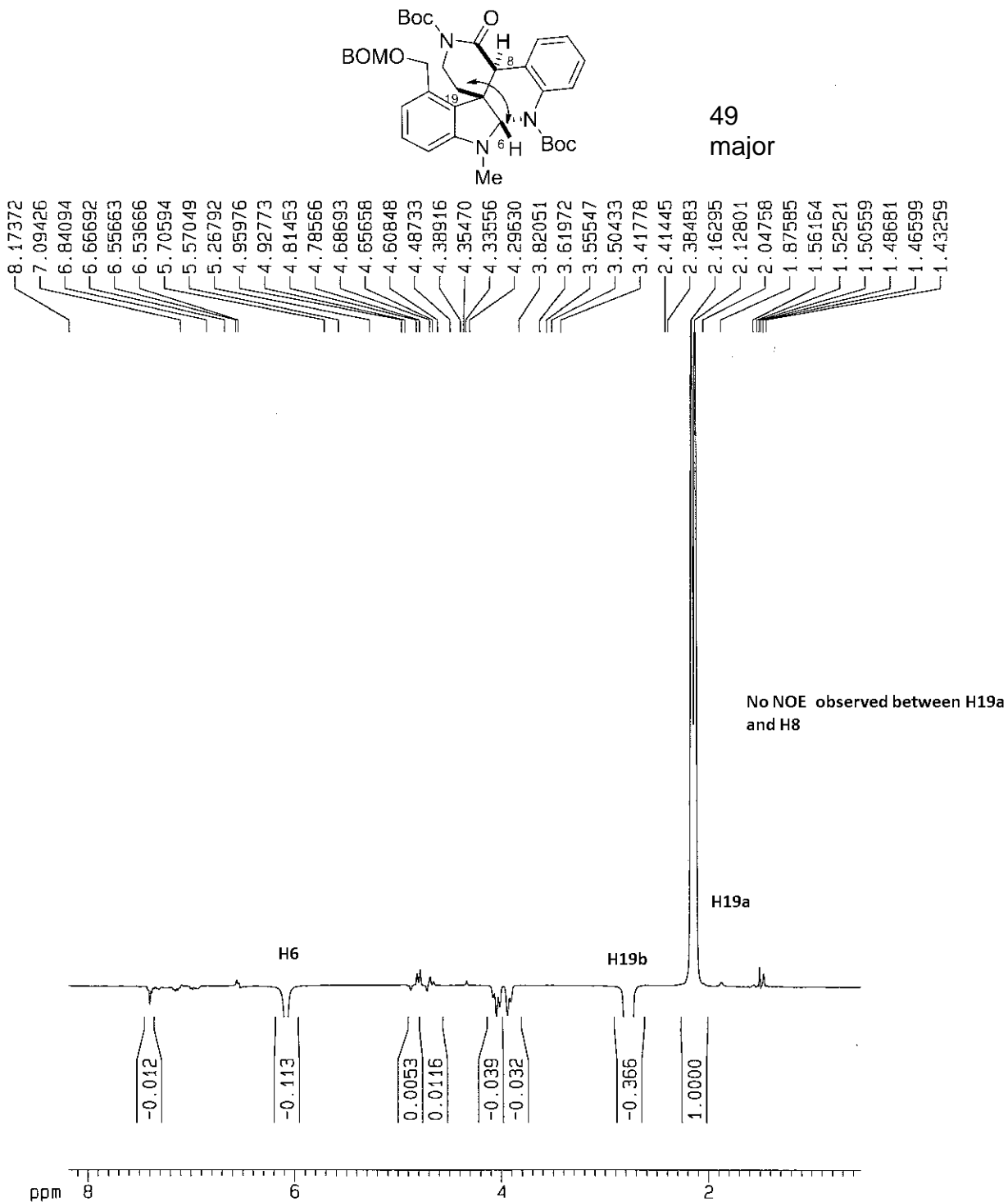
F2 - Processing parameters  
SI 2048  
SF 400.1300000 MHz  
WDW SINE  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.40

F1 - Processing parameters  
SI 1024  
MC2 TPPI  
SF 400.1300000 MHz  
WDW SINE  
SSB 0  
LB 0.00 Hz  
GB 0

CX2 15.00 cm  
CX1 15.00 cm  
F2PLO 8.065 ppm  
F2LO 3226.89 Hz  
F2PHI 0.565 ppm  
F2H1 225.98 Hz  
F1PLO 8.997 ppm  
F1LO 3600.07 Hz  
F1PHI -0.663 ppm  
F1H1 -265.49 Hz  
F2PPMCM 0.49939 ppm/cm  
F2HZCM 200.05969 Hz/cm  
F1PPMCM 0.64405 ppm/cm  
F1HZCM 257.70398 Hz/cm

ppm

Integral



Current Data Parameters  
NAME PL-Ju130-09  
EXPNO 5  
PROCNO 3

F2 - Acquisition Parameters  
Date\_ 20090801  
Time 2.06  
INSTRUM spect  
PROBHD 5 mm BBI 1H-0  
PULPROG noesygptp  
TD 2048  
SOLVENT CDCl3  
NS 8  
DS 8  
SWH 3063.726 Hz  
FIDRES 1.495960 Hz  
AQ 0.3342636 sec  
RG 40.3  
DM 163.200 usec  
DE 6.00 usec  
TE 300.0 K  
d0 0.0000300 sec  
d1 4.0000000 sec  
d8 0.80000001 sec  
d16 0.00020000 sec  
d20 0.39880002 sec  
IND 0.00012496 sec

===== CHANNEL f1 =====  
NUC1 1H  
P1 7.39 usec  
P2 14.78 usec  
PL1 0.00 dB  
SF01 400.1317534 MHz

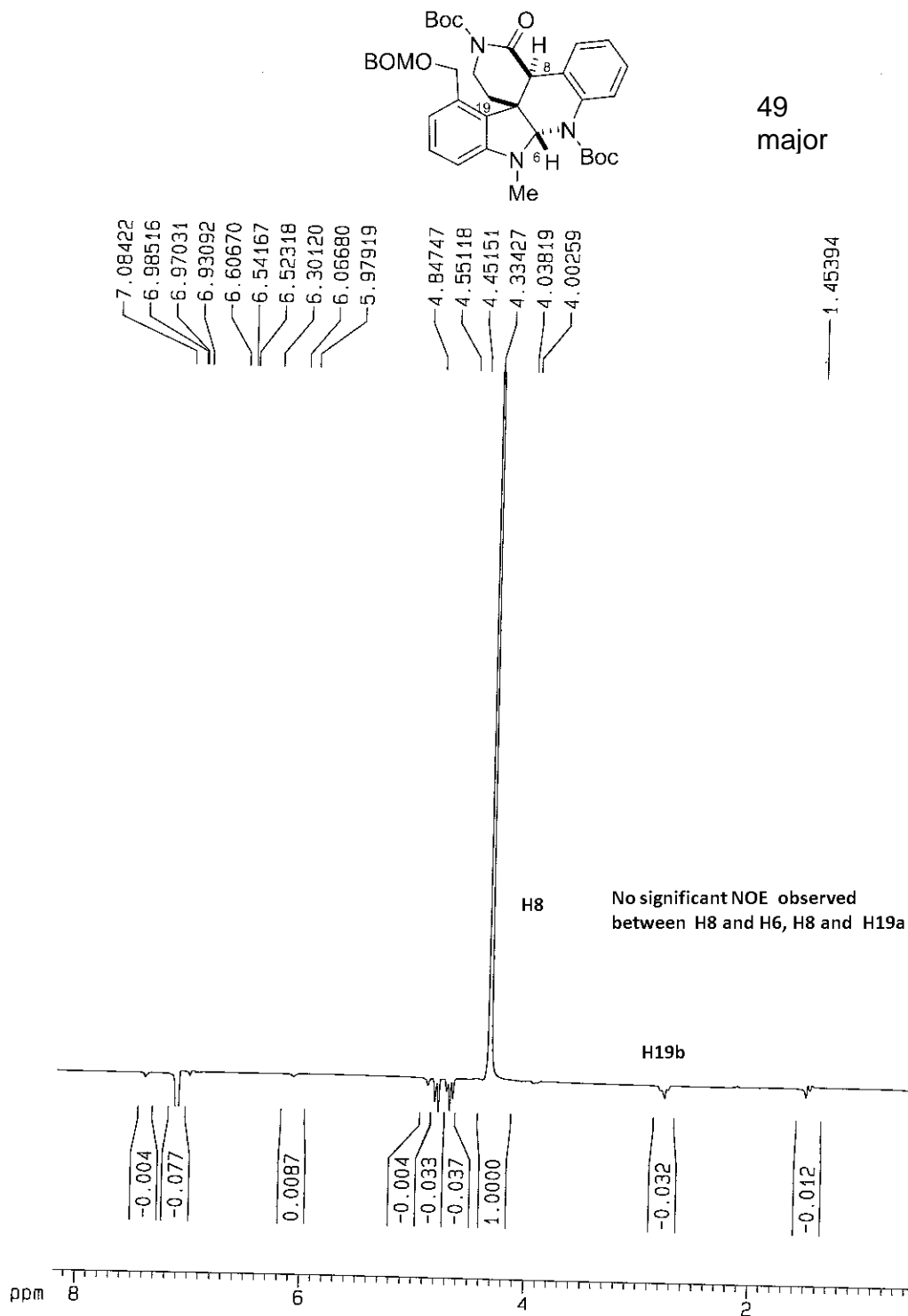
===== GRADIENT CHANNEL =====  
GPNAM1 sine.100  
GPNAM2 sine.100  
GPX1 0.00 %  
GPX2 0.00 %  
GPY1 0.00 %  
GPY2 0.00 %  
GPZ1 40.00 %  
GPZ2 -40.00 %  
P16 1000.00 usec

F2 - Processing parameters  
SI 2048  
SF 400.1300092 MHz  
WDW QSINE  
SSB 2  
LB 0.00 Hz  
GB 0  
PC 1.00

1D NMR plot parameters  
CX 20.00 cm  
F1P 11.000 ppm  
F1 4401.43 Hz  
F2P -1.000 ppm  
F2 -400.13 Hz  
PPMCM 0.68000 ppm/cm  
HZCM 240.07800 Hz/cm

Integral

ppm

49  
major

Current Data Parameters  
 NAME PL-Ju130-09  
 EXPNO 5  
 PROCNO 2

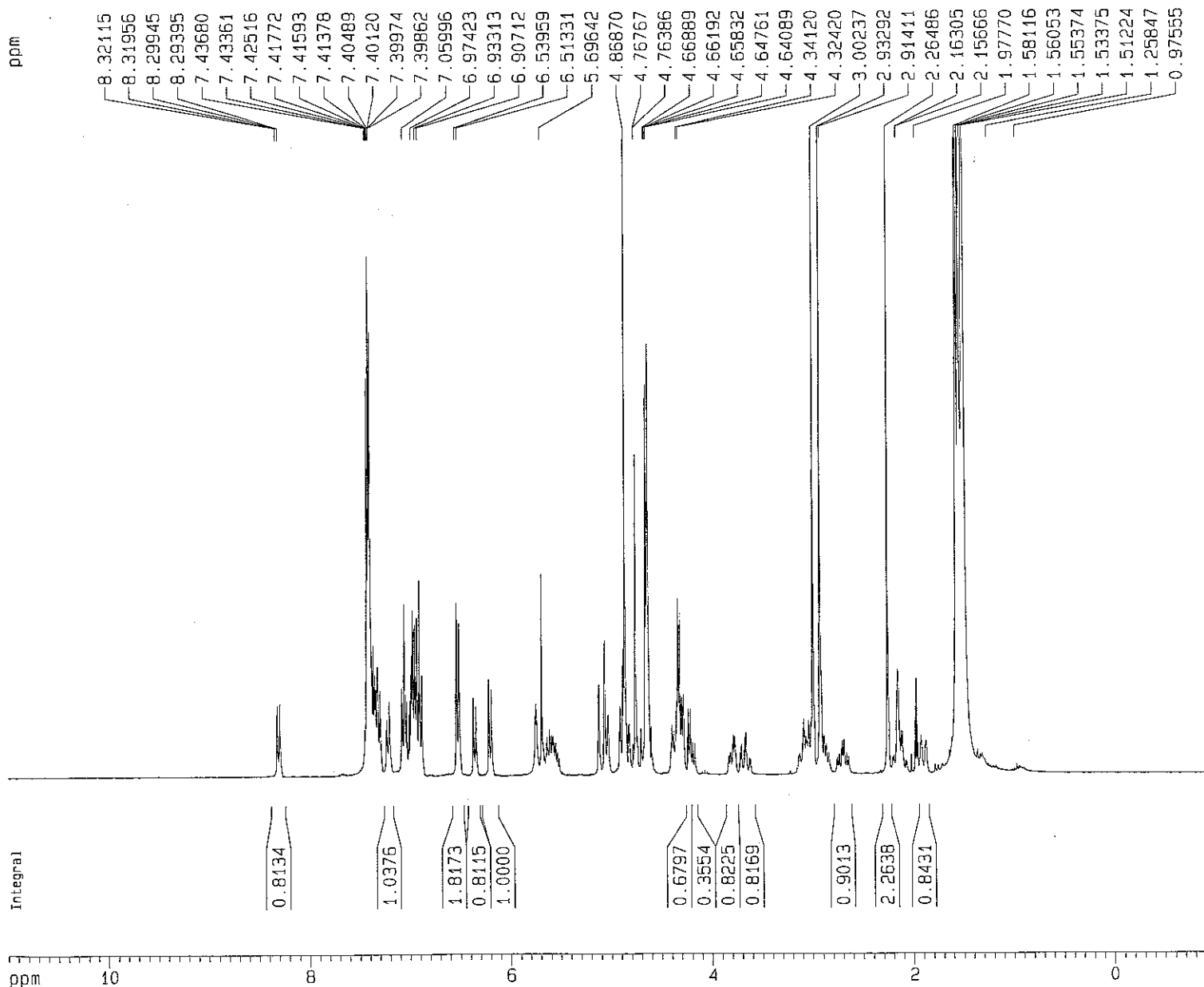
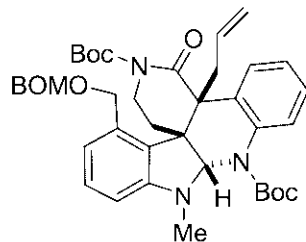
F2 - Acquisition Parameters  
 Date\_ 20090801  
 Time 2.06  
 INSTRUM spect  
 PROBHD 5 mm BBI 1H-8  
 PULPROG noesygptp  
 TD 2048  
 SOLVENT CDCl3  
 NS 8  
 DS 8  
 SWH 3063.726 Hz  
 FIDRES 1.495960 Hz  
 AQ 0.3342836 sec  
 RG 40.3  
 DW 163.200 usec  
 DE 6.00 usec  
 TE 300.0 K  
 d0 0.00000300 sec  
 D1 4.00000000 sec  
 D8 0.80000001 sec  
 D16 0.00020000 sec  
 D20 0.39880002 sec  
 INO 0.00012496 sec

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 7.39 usec  
 P2 14.78 usec  
 PL1 0.00 dB  
 SFO1 400.1317534 MHz

===== GRADIENT CHANNEL =====  
 GPNAM1 sine.100  
 GPNAM2 sine.100  
 GPX1 0.00 %  
 GPX2 0.00 %  
 GPY1 0.00 %  
 GPY2 0.00 %  
 GPZ1 40.00 %  
 GPZ2 -40.00 %  
 P16 1000.00 usec

F2 - Processing parameters  
 SI 2048  
 SF 400.1300092 MHz  
 WDW GSINE  
 SSB 2  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 11.000 ppm  
 F1 4401.43 Hz  
 F2P -1.000 ppm  
 F2 -400.13 Hz  
 PPMCM 0.60000 ppm/cm  
 HZCM 240.07800 Hz/cm



## Current Data Parameters

NAME PL-Aug20-09  
EXPNO 7  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20090820  
Time 20.10  
INSTRUM spect  
PROBHD 5 mm QNP 1H/1  
PULPROG zg30  
TD 65536  
SOLVENT CD3CN  
NS 16  
DS 2  
SWH 6172.839 Hz  
FIDRES 0.094190 Hz  
AQ 5.3084660 sec  
RG 50.8  
DW 81.000 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec

## ===== CHANNEL f1 =====

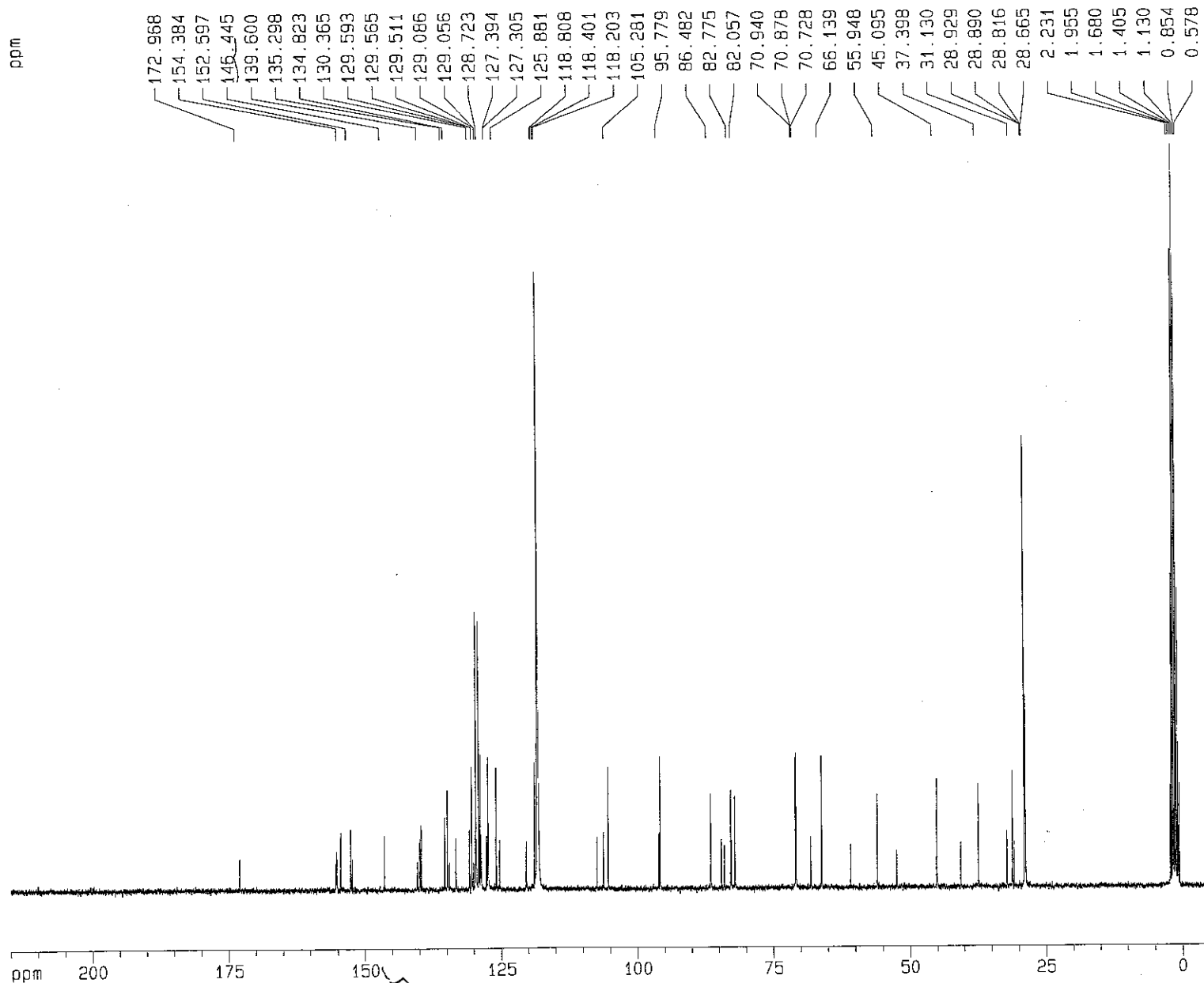
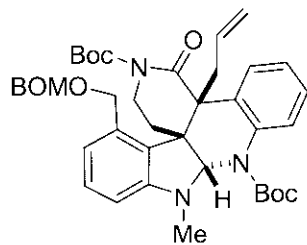
NUC1 1H  
P1 11.70 usec  
PL1 0.00 dB  
SFO1 299.8718518 MHz

## F2 - Processing parameters

SI 32768  
SF 299.8700000 MHz  
WDW no  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

## 1D NMR plot parameters

CX 20.00 cm  
F1P 11.000 ppm  
F1 3298.57 Hz  
F2P -1.000 ppm  
F2 -299.87 Hz  
PPMCM 0.60000 ppm/cm  
HZCM 179.92200 Hz/cm



Current Data Parameters  
NAME PL-Aug20-09  
EXPNO 3  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20090820  
Time 15.43  
INSTRUM spect  
PROBHD 5 mm GNP 1H/1  
PULPROG zgpg30  
TD 65536  
SOLVENT CD3CN  
NS 1673  
DS 4  
SWH 18796.992 Hz  
FIDRES 0.286619 Hz  
AQ 1.7433076 sec  
RG 512  
DW 26.600 usec  
DE 6.00 usec  
TE 300.0 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
D12 0.00002000 sec

===== CHANNEL f1 =====  
NUC1 13C  
P1 5.40 usec  
PL1 -6.00 dB  
SFO1 75.4106357 MHz

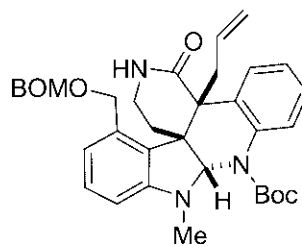
===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 115.00 usec  
PL2 0.00 dB  
PL12 20.00 dB  
PL13 20.00 dB  
SFO2 299.8711995 MHz

## F2 - Processing parameters

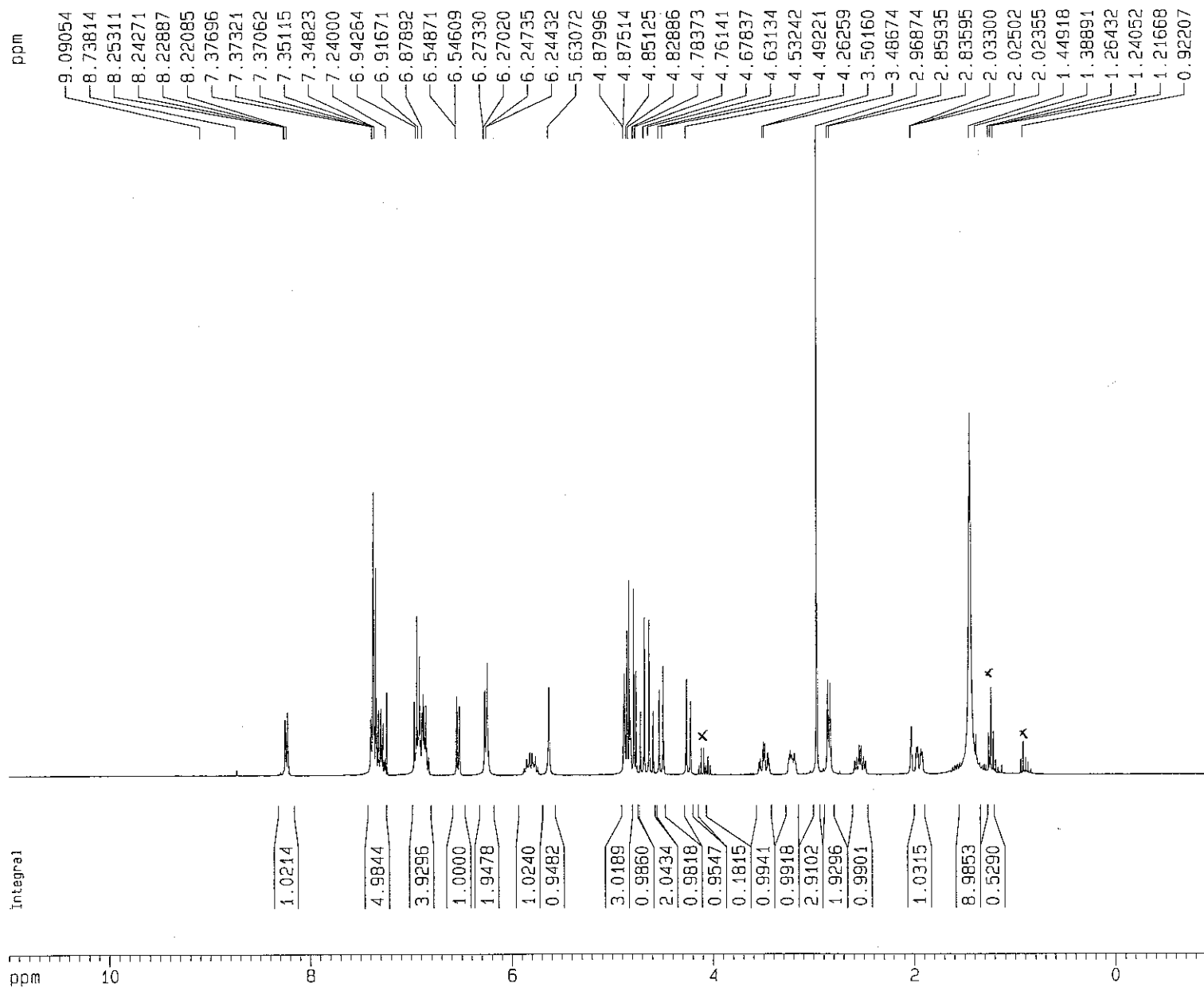
SI 32768  
SF 75.4022745 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

## 1D NMR plot parameters

CX 20.00 cm  
F1P 215.000 ppm  
F1 16211.49 Hz  
F2P -5.000 ppm  
F2 -377.01 Hz  
PPMCM 11.00000 ppm/cm  
HZCM 829.42505 Hz/cm



52



## Current Data Parameters

NAME PL-Aug22-09  
EXPNO 2  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20090822  
Time 12.26  
INSTRUM spect  
PROBHD 5 mm GNP 1H/1  
PULPROG zg30  
TD 65536  
SOLVENT CDC13  
NS 16  
DS 2  
SWH 6172.839 Hz  
FIDRES 0.094190 Hz  
AQ 5.3084660 sec  
RG 80.6  
DW 81.000 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec

## ===== CHANNEL f1 =====

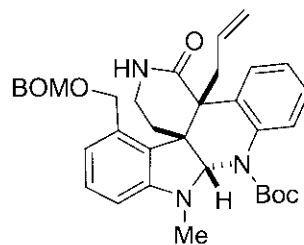
NUC1 1H  
P1 11.70 usec  
PL1 0.00 dB  
SFO1 299.8718518 MHz

## F2 - Processing parameters

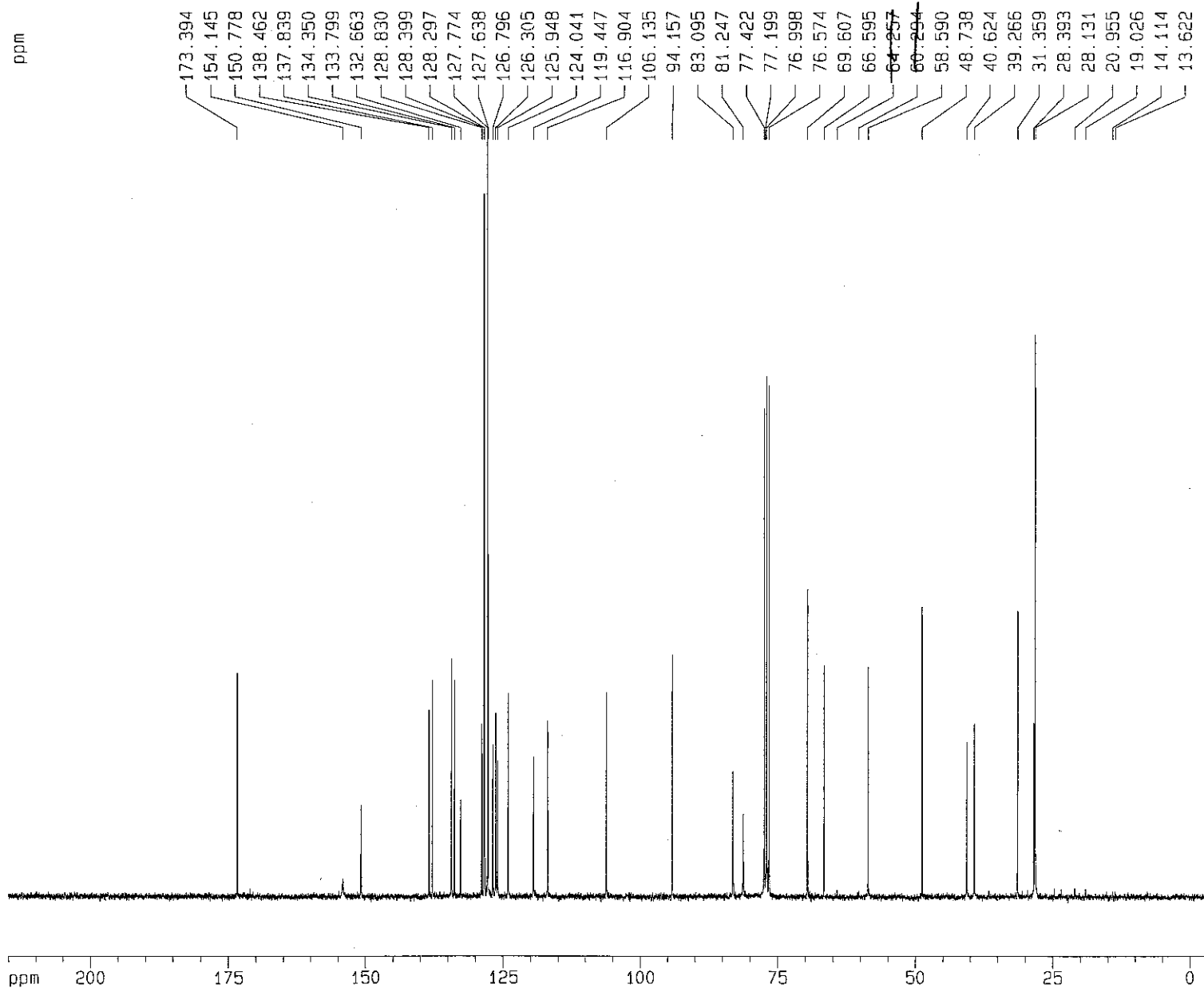
SI 32768  
SF 299.8700159 MHz  
WDW no  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

## 1D NMR plot parameters

CX 20.00 cm  
F1P 11.000 ppm  
F1 3298.57 Hz  
F2P -1.000 ppm  
F2 -299.87 Hz  
PPMCM 0.60000 ppm/cm  
HZCM 179.92201 Hz/cm



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## Current Data Parameters

NAME PL-Aug22-09  
EXPNO 3  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20090822  
Time 13.56  
INSTRUM spect  
PROBHD 5 mm GNP 1H/1  
PULPROG zgpg30  
TD 65536  
SOLVENT CDC13  
NS 1393  
DS 4  
SWH 18796.992 Hz  
FIDRES 0.286619 Hz  
AQ 1.7433076 sec  
RG 1024  
DW 26.600 usec  
DE 6.00 usec  
TE 300.0 K  
D1 2.0000000 sec  
D11 0.0300000 sec  
D12 0.0000200 sec

## ===== CHANNEL f1 =====

NUC1 13C  
P1 5.40 usec  
PL1 -6.00 dB  
SFO1 75.4106357 MHz

## ===== CHANNEL f2 =====

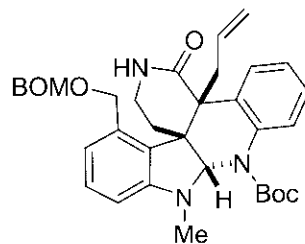
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 115.00 usec  
PL2 0.00 dB  
PL12 20.00 dB  
PL13 20.00 dB  
SFO2 299.8711995 MHz

## F2 - Processing parameters

SI 32768  
SF 75.4023815 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

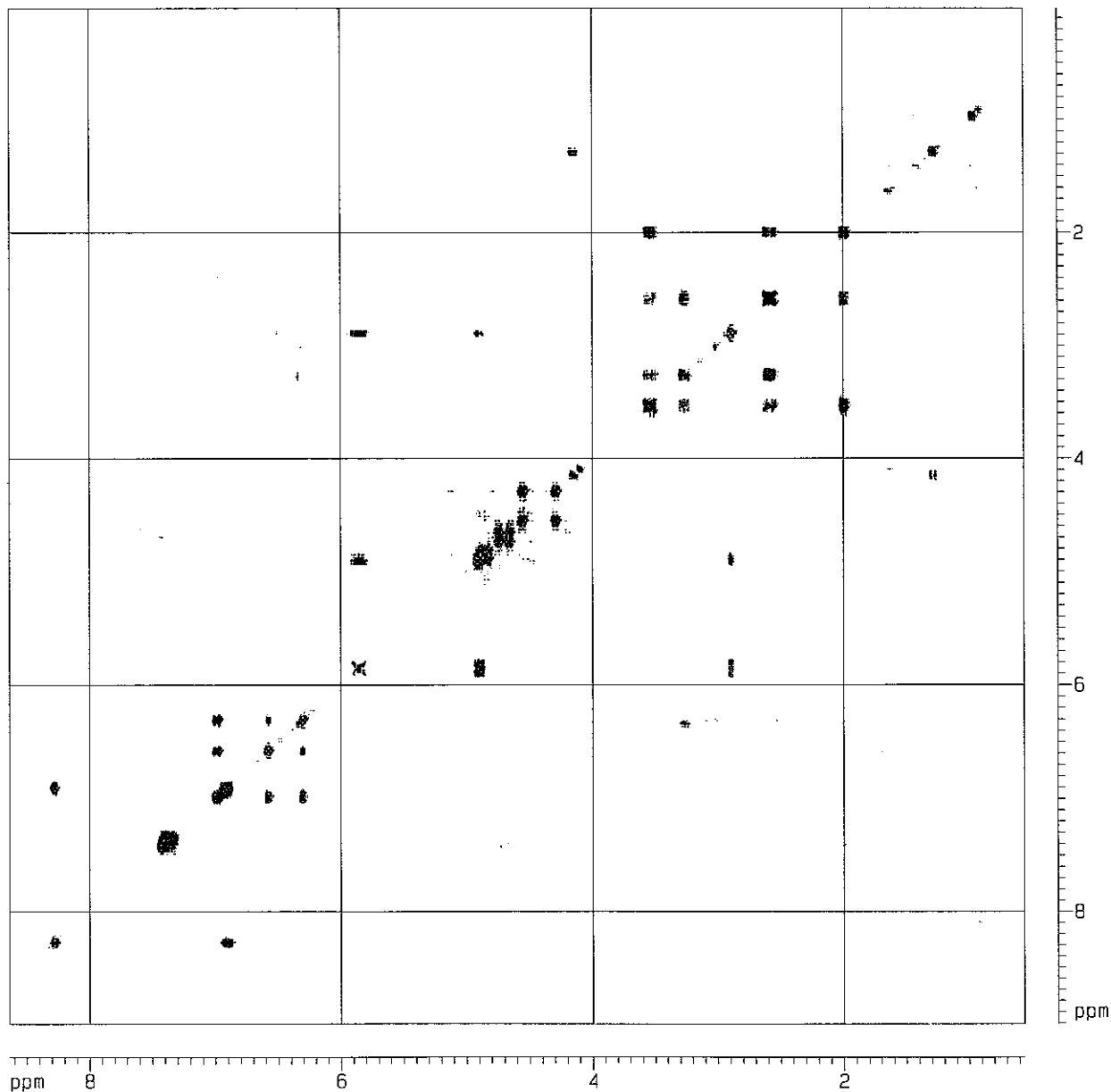
## 1D NMR plot parameters

CX 20.00 cm  
F1P 215.000 ppm  
F1 16211.51 Hz  
F2P -5.000 ppm  
F2 -377.01 Hz  
PPMCM 11.00000 ppm/cm  
HZCM 829.42621 Hz/cm



52

COSY



Current Data Parameters  
 NAME PL-Aug22-09  
 EXPNO 4  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20090823  
 Time 0.15  
 INSTRUM spect  
 PROBHD 5 mm BBI 1H-6  
 PULPROG ajb.cosygmftp  
 TD 2048  
 SOLVENT CDCl3  
 NS 4  
 DS 8  
 SWH 3324.468 Hz  
 FIDRES 1.623275 Hz  
 AQ 0.3080692 sec  
 RG 3549.1  
 DW 150.400 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D0 0.00000000 sec  
 D1 4.00000000 sec  
 d13 0.00000000 sec  
 D16 0.00020000 sec  
 d20 0.00120000 sec  
 INO 0.0009360 sec

----- CHANNEL f1 -----  
 NUC1 1H  
 P1 7.00 usec  
 P2 14.00 usec  
 PL1 0.00 dB  
 SFO1 400.1318736 MHz  
 ----- GRADIENT CHANNEL -----  
 P16 1000.00 usec

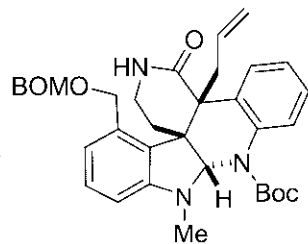
F1 - Acquisition parameters  
 NDO 2  
 TD 650  
 SFO1 400.1319 MHz  
 FIDRES 8.216277 Hz  
 SW 13.350 ppm

F2 - Processing parameters  
 SI 2048  
 SF 400.1300000 MHz  
 WDW SINE  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.40

F1 - Processing parameters  
 SI 1024  
 MC2 TPPI  
 SF 400.1300000 MHz  
 WDW SINE  
 SSB 0  
 LB 0.00 Hz  
 GB 0

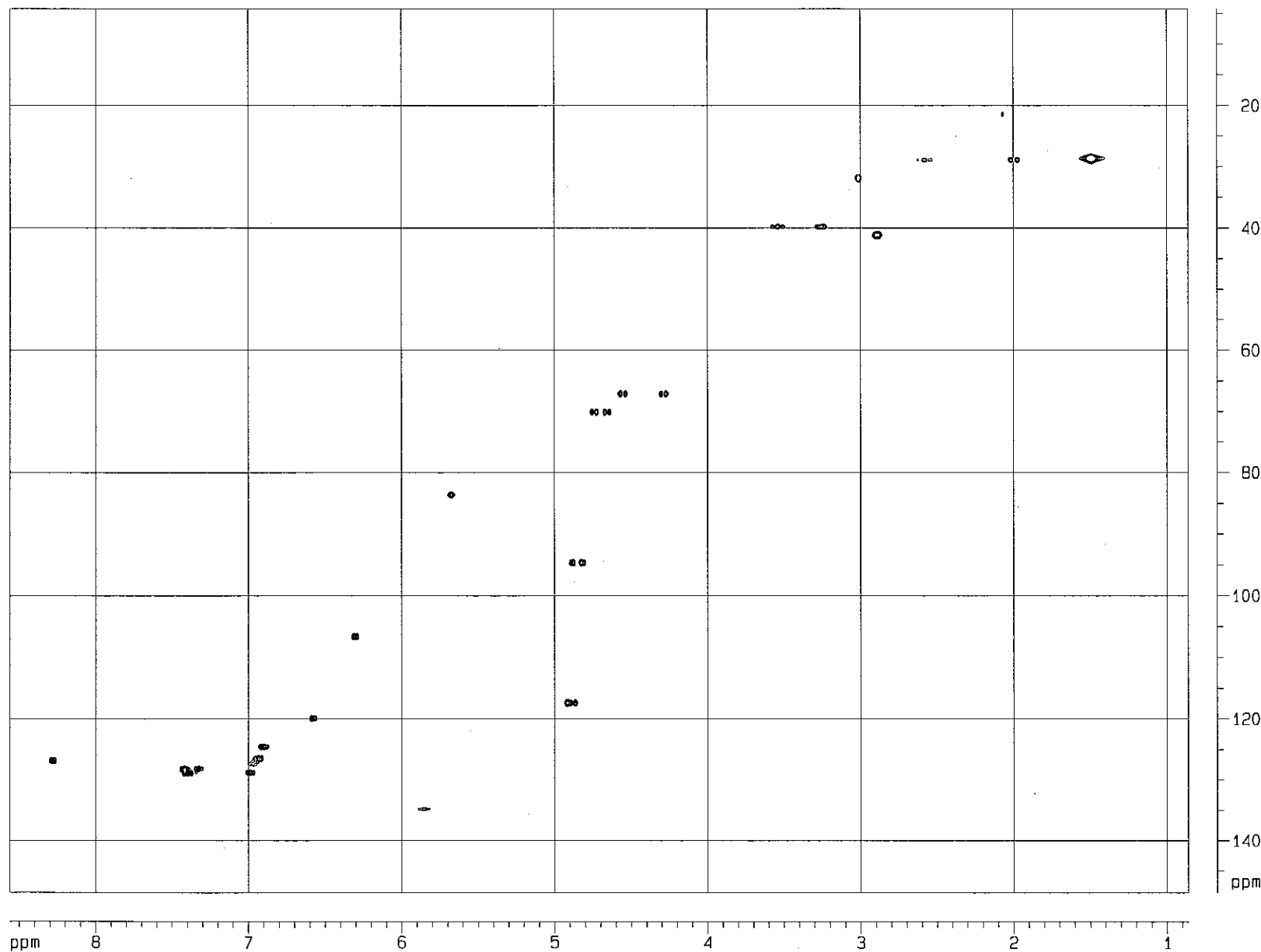
2D NMR plot parameters  
 CX2 15.00 cm  
 CX1 15.00 cm  
 F2PLO 8.638 ppm  
 F2LO 3458.50 Hz  
 F2PHI 0.561 ppm  
 F2HI 224.96 Hz  
 F1PLO 9.011 ppm  
 F1LO 3605.75 Hz  
 F1PHI 0.016 ppm  
 F1HI 6.24 Hz  
 F2PPMCM 0.53846 ppm/cm  
 F2HZCM 215.46277 Hz/cm  
 F1PPMCM 0.59972 ppm/cm  
 F1HZCM 239.96729 Hz/cm





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HMQC



```

Current Data Parameters
NAME      PL-Aug22-09
EXPNO    2
PROCNO    1

F2 - Acquisition Parameters
Date_     20090822
Time      21.25
INSTRUM   spect
PROBHD    5 mm BBI 1H-2
PULPROG   mw49p1p
TD         2645
SOLVENT   CDCl3
NS         4
DS         80
SWH        3324.465 Hz
FIDRES     1.623275 Hz
AQ         0.308092 sec
RG         7298.2
DK         150.400 usec
DE         6.00 usec
TE         300.0 K
CNS12     145.000000
d0         0.0000000 sec
d1         1.00031290 sec
d2         0.00344326 sec
d4         0.00172414 sec
d11        0.03000000 sec
d13        0.00000000 sec
d16        0.00000000 sec
d20        0.00052414 sec
d21        0.00224428 sec
IND        0.00001450 sec

----- CHANNEL f1 -----
NUC1       1H
P1         7.00 usec
PE         14.00 usec
PL1        0.00 dB
SFO1       400.1316738 MHz

----- CHANNEL f2 -----
CPDPRG2   90T2
NUC2       13C
P2         17.00 usec
P4         34.00 usec
PCPD2     56.00 usec
PL2        0.00 dB
PL12       6.30 dB
SFO2       100.6203332 MHz

----- GRADIENT CHANNEL -----
GPNAM1    SINE.100
GPNAM2    SINE.100
GPNAM3    SINE.100
GFX1      17.00 X
GFX2      20.00 X
GFX3      25.00 X
GPY1      17.00 X
GPY2      20.00 X
GPY3      25.00 X
GZ1       17.00 X
GZ2       20.00 X
GZ3       25.00 X
P10       1000.00 usec

F1 - Acquisition parameters
ND0        4
TD         655
SFO1       100.6203332 MHz
FIDRES     65.541107 Hz
SM         166.751 ppm

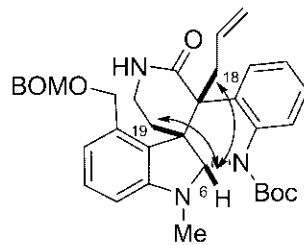
F2 - Processing parameters
SI         2048
SF         400.1300600 MHz
WDW        05SINE
SSB        2
LB         0.00 Hz
GB         0
PC         1.00

F1 - Processing parameters
SI         1024
MC2        TPPI
SF         100.6127280 MHz
WDW        05SINE
SSB        2
LB         0.00 Hz
GB         0

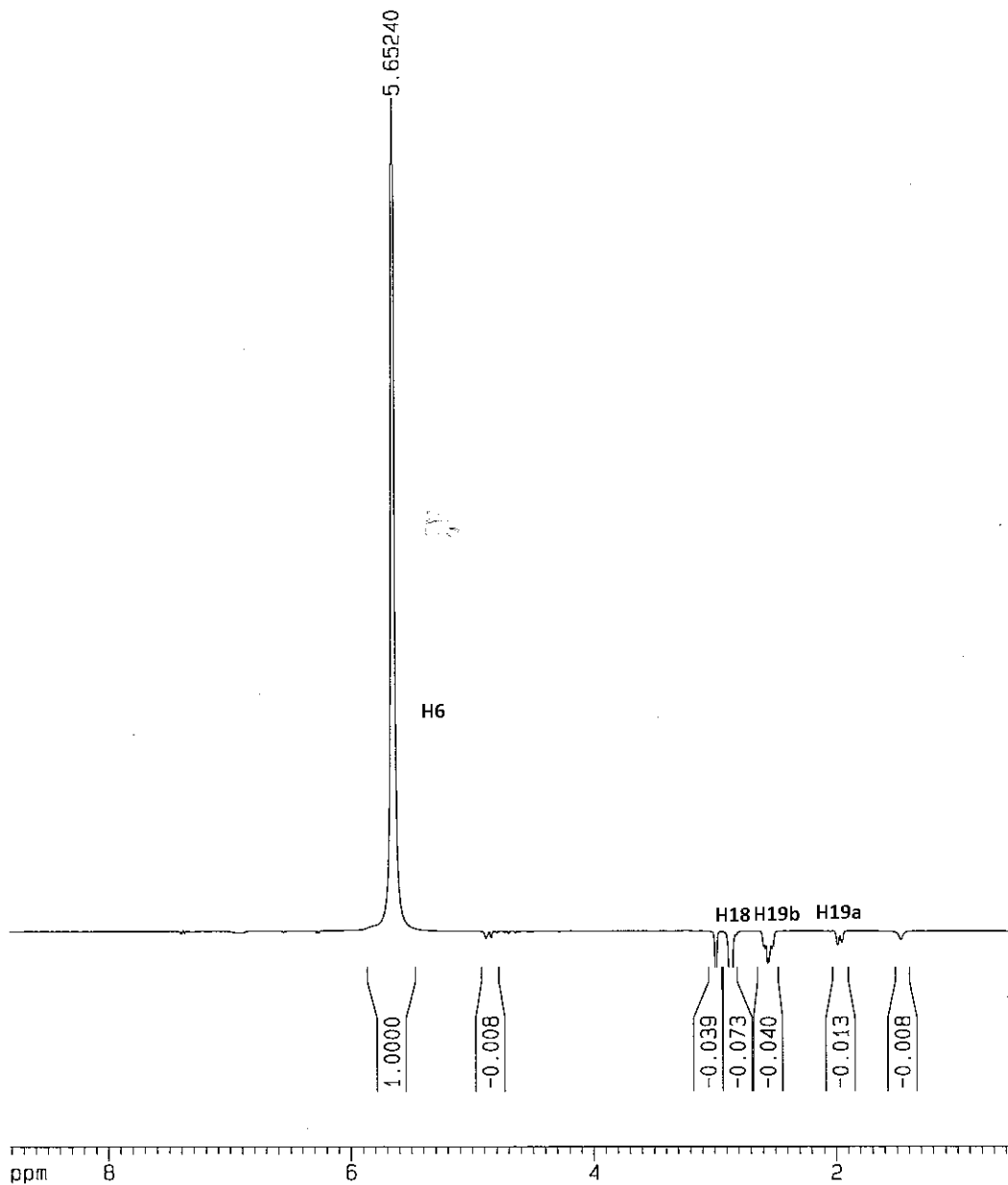
2D NMR plot parameters
CX2        20.00 cm
CX1         5.00 cm
F2PLD      0.561 ppm
F2LD       3425.66 Hz
F2PHC      0.861 ppm
F2H1       344.58 Hz
F1PLD      148.064 ppm
F1LD       14959.49 Hz
F1PH1     4.834 ppm
F1H1       425.75 Hz
F2PPMCH    0.39500 ppm/cm
F2RZCH     154.04694 Hz/cm
F1PPMCH    0.63215 ppm/cm
F1RZCH     998.51614 Hz/cm

```

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ppm



Integral

Current Data Parameters  
 NAME PL-Aug22-09  
 EXPNO 5  
 PROCNO 5

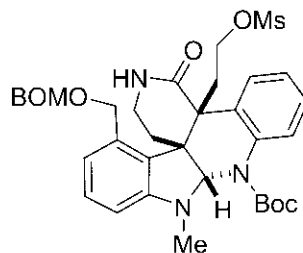
F2 - Acquisition Parameters  
 Date\_ 20090823  
 Time 3.25  
 INSTRUM spect  
 PROBHD 5 mm BBI 1H-B  
 PULPROG noesygtp  
 TD 2048  
 SOLVENT CDC13  
 NS 8  
 DS 8  
 SWH 3324.468 Hz  
 FIDRES 1.623275 Hz  
 AQ 0.3080692 sec  
 RG 64  
 DW 150.400 usec  
 DE 6.00 usec  
 TE 300.0 K  
 d0 0.00000300 sec  
 D1 4.00000000 sec  
 D8 0.80000001 sec  
 D16 0.00020000 sec  
 d20 0.39880002 sec  
 IN0 0.00012496 sec

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 7.00 usec  
 P2 14.00 usec  
 PL1 0.00 dB  
 SF01 400.1318738 MHz

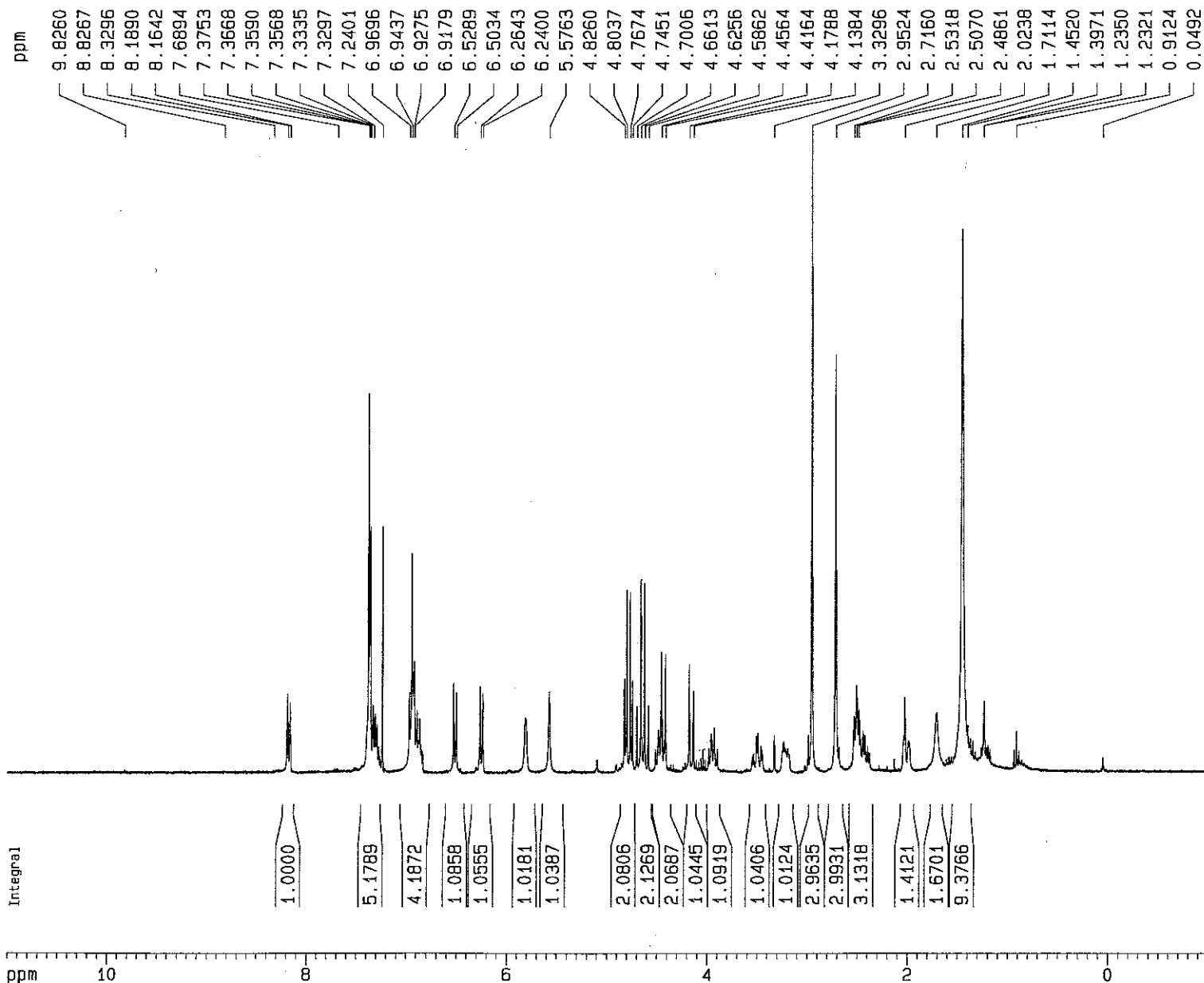
===== GRADIENT CHANNEL =====  
 GPNAM1 sine.100  
 GPNAM2 sine.100  
 GPX1 0.00 %  
 GPX2 0.00 %  
 GPY1 0.00 %  
 GPY2 0.00 %  
 GPZ1 40.00 %  
 GPZ2 -40.00 %  
 P16 1000.00 usec

F2 - Processing parameters  
 SI 2048  
 SF 400.130092 MHz  
 WDW QSINE  
 SSB 2  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 11.000 ppm  
 F1 4401.43 Hz  
 F2P -1.000 ppm  
 F2 -400.13 Hz  
 PPMCM 0.60000 ppm/cm  
 HZCM 240.07800 Hz/cm



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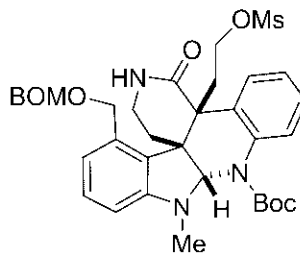
Current Data Parameters  
 NAME PL-Aug26-2009  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20090826  
 Time 9.57  
 INSTRUM spect  
 PROBHD 5 mm Multinu  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 16  
 DS 2  
 SWH 6172.639 Hz  
 FIDRES 0.094190 Hz  
 AQ 5.3084660 sec  
 RG 362  
 DW 81.000 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 1.00000000 sec

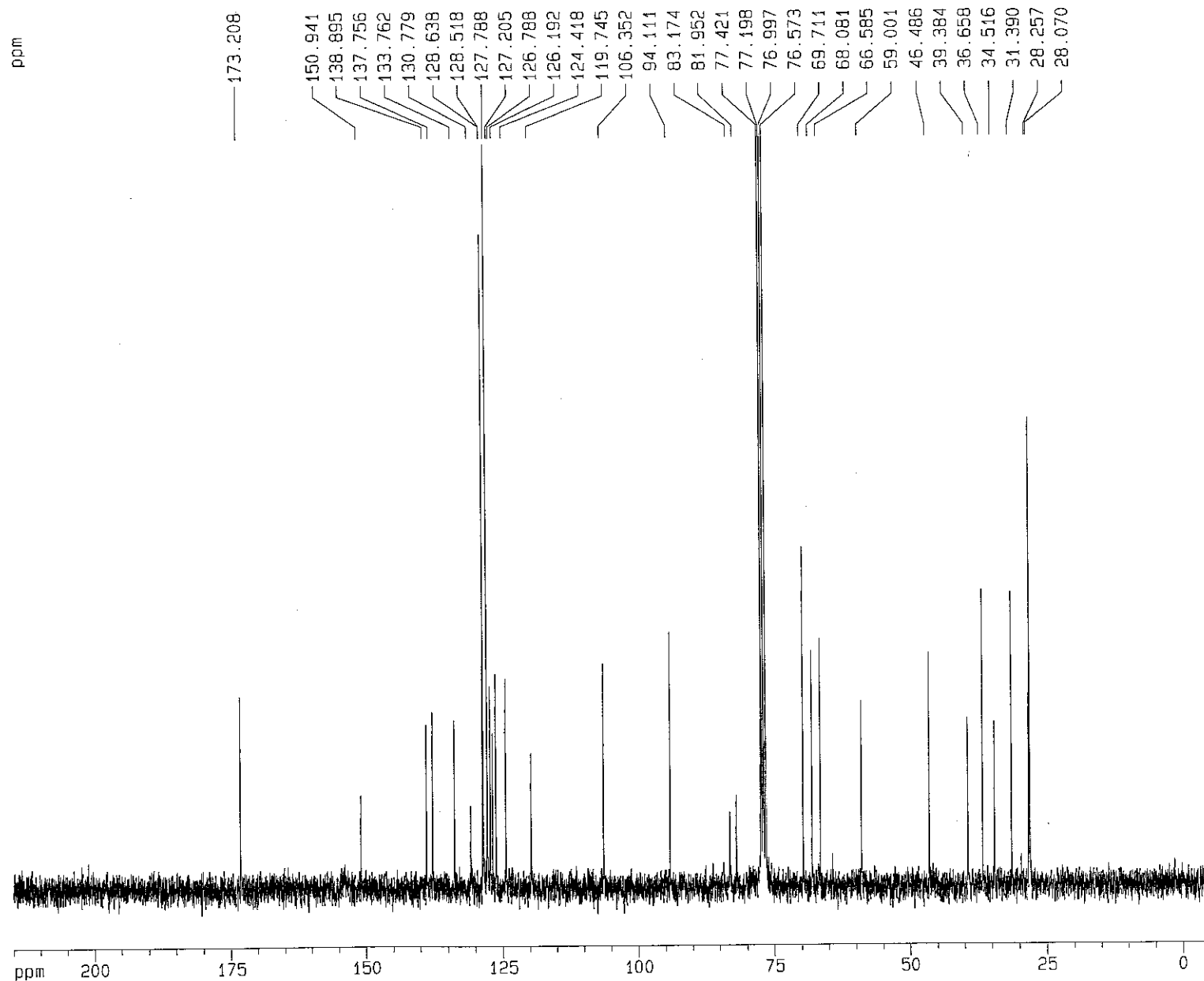
----- CHANNEL f1 -----  
 NUC1 1H  
 P1 9.60 usec  
 PL1 -6.00 dB  
 SF01 300.1318534 MHz

F2 - Processing parameters  
 SI 32768  
 SF 300.1300124 MHz  
 WDW no  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 11.000 ppm  
 F1 3301.43 Hz  
 F2P -1.000 ppm  
 F2 -300.13 Hz  
 PPMCM 0.60000 ppm/cm  
 HZCM 180.07800 Hz/cm



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Current Data Parameters  
NAME PL-Aug26-09  
EXPNO 2  
PROCNO 1

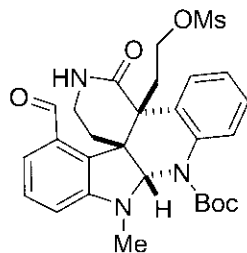
F2 - Acquisition Parameters  
Date\_ 20090826  
Time 10.51  
INSTRUM spect  
PROBHD 5 mm GNP 1H/1  
PULPROG zgpg30  
TD 65536  
SOLVENT CDC13  
NS 1405  
DS 4  
SWH 18796.992 Hz  
FIDRES 0.286819 Hz  
AQ 1.7433076 sec  
RG 512  
DW 26.600 usec  
DE 6.00 usec  
TE 300.0 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
D12 0.00002000 sec

===== CHANNEL f1 =====  
NUC1 13C  
P1 5.40 usec  
PL1 -6.00 dB  
SFO1 75.4106357 MHz

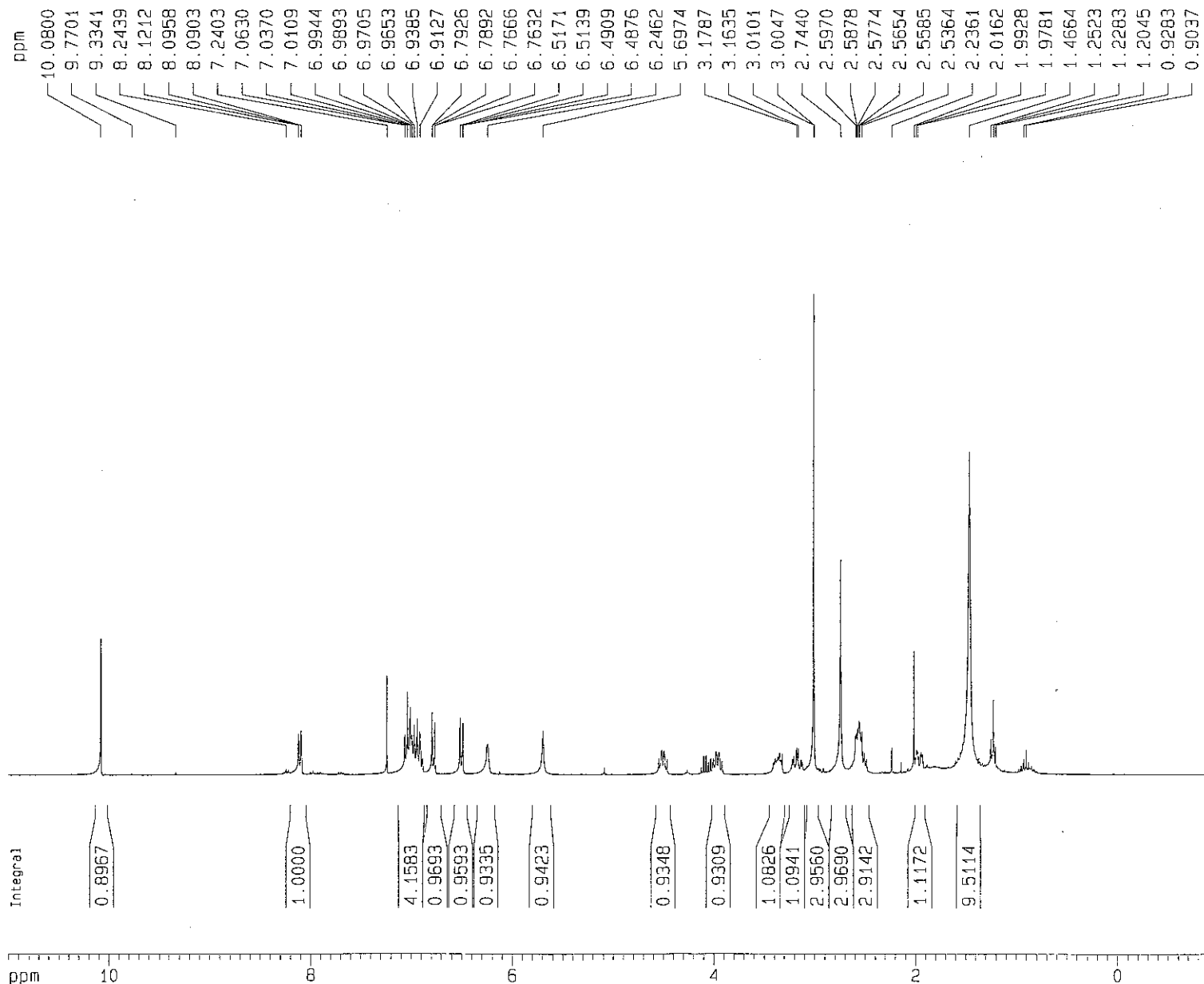
===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 115.00 usec  
PL2 0.00 dB  
PL12 20.00 dB  
PL13 20.00 dB  
SFO2 299.8711995 MHz

F2 - Processing parameters  
SI 32768  
SF 75.4023769 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

1D NMR plot parameters  
CX 20.00 cm  
F1P 215.000 ppm  
F1 16211.51 Hz  
F2P -5.000 ppm  
F2 -377.01 Hz  
PPMCM 11.00000 ppm/cm  
HZCM 829.42615 Hz/cm



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## Current Data Parameters

NAME PL-Oct24-09  
EXPNO 1  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20091024  
Time 12.37  
INSTRUM spect  
PROBHD 5 mm GNP 1H/1  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6172.839 Hz  
FIDRES 0.094190 Hz  
AQ 5.3084660 sec  
RG 181  
DW 81.000 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec

## ===== CHANNEL f1 =====

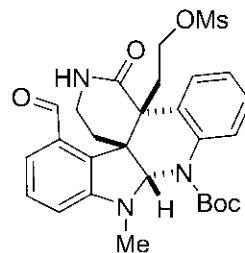
NUC1 1H  
P1 11.70 usec  
PL1 0.00 dB  
SF01 299.8718518 MHz

## F2 - Processing parameters

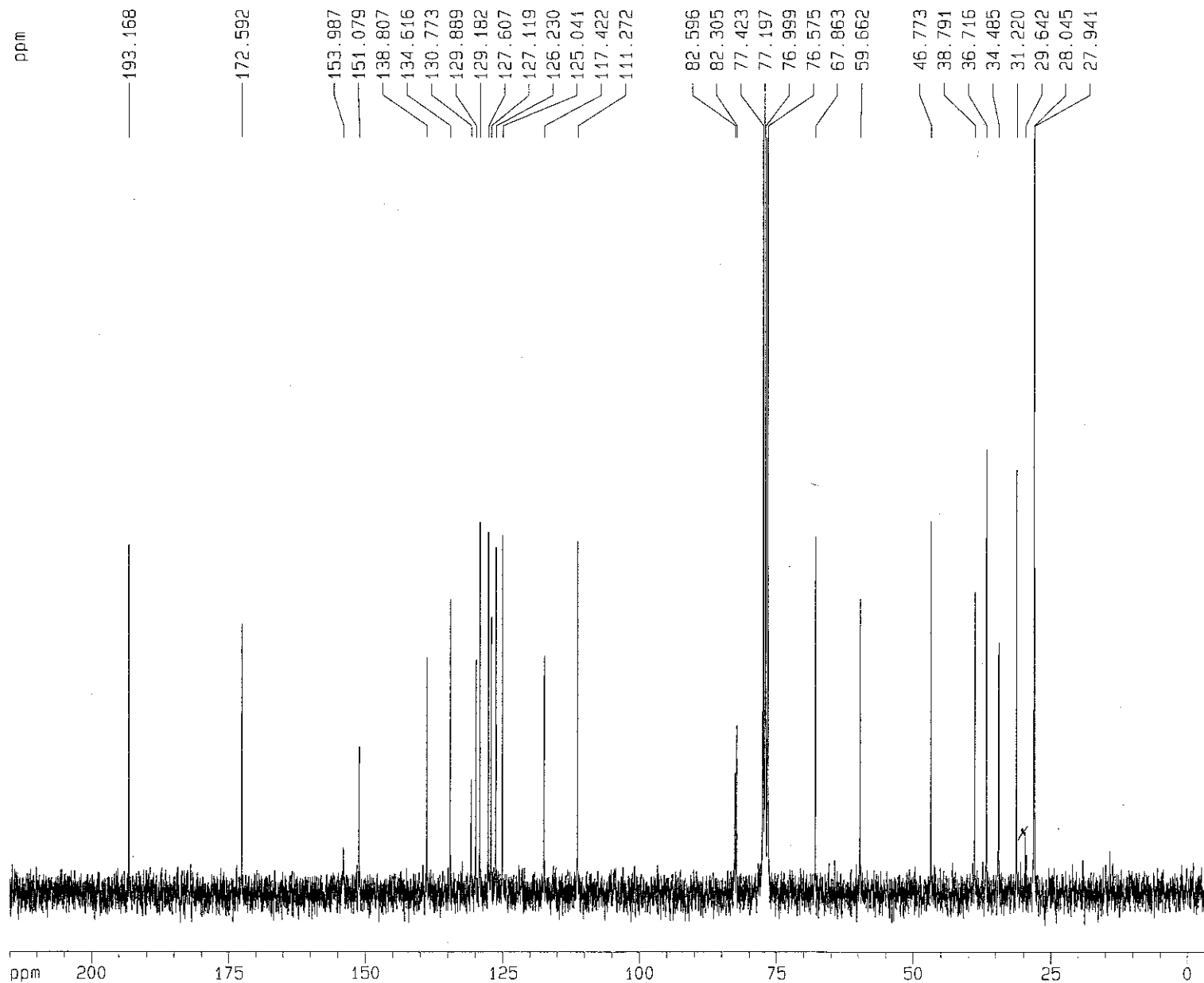
SI 32768  
SF 299.8700157 MHz  
WDW no  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

## 1D NMR plot parameters

CX 20.00 cm  
F1P 11.000 ppm  
F1 3298.57 Hz  
F2P -1.000 ppm  
F2 -299.87 Hz  
PPMCM 0.60000 ppm/cm  
HZCM 179.92201 Hz/cm



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Current Data Parameters  
 NAME PL-Oct24-09  
 EXPNO 2  
 PROCNO 1

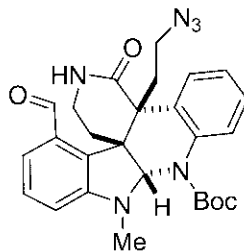
F2 - Acquisition Parameters  
 Date\_ 20091024  
 Time 13.43  
 INSTRUM spect  
 PROBHD 5 mm GNP 1H/1  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDC13  
 NS 991  
 DS 4  
 SWH 18796.992 Hz  
 FIDRES 0.286619 Hz  
 AQ 1.7433076 sec  
 RG 4096  
 DW 26.600 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 2.0000000 sec  
 D11 0.0300000 sec  
 D12 0.0000200 sec

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 5.40 usec  
 PL1 -6.00 dB  
 SF01 75.4106357 MHz

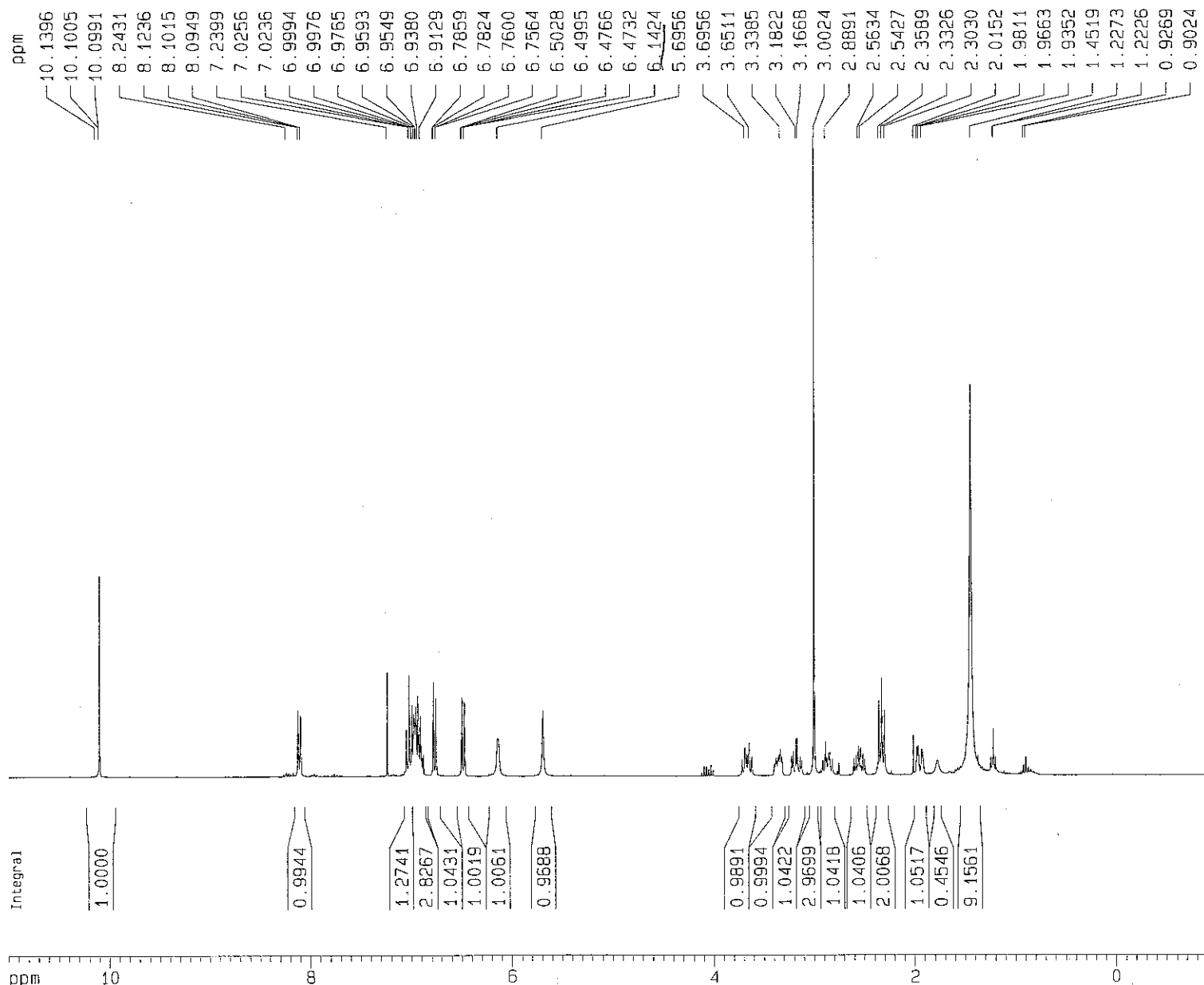
===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 115.00 usec  
 PL2 0.00 dB  
 PL12 20.00 dB  
 PL13 20.00 dB  
 SF02 299.8711995 MHz

F2 - Processing parameters  
 SI 32768  
 SF 75.4023775 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 215.000 ppm  
 F1 16211.51 Hz  
 F2P -5.000 ppm  
 F2 -377.01 Hz  
 PPMCM 11.00000 ppm/cm  
 HZCM 829.42615 Hz/cm



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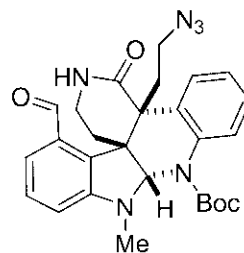
Current Data Parameters  
 NAME PL-Oct28-09  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20091028  
 Time 12.23  
 INSTRUM spect  
 PROBHD 5 mm GNP 1H/1  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 16  
 DS 2  
 SWH 6172.839 Hz  
 FIDRES 0.094190 Hz  
 AQ 5.3084660 sec  
 RG 181  
 DW 81.000 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 1.00000000 sec

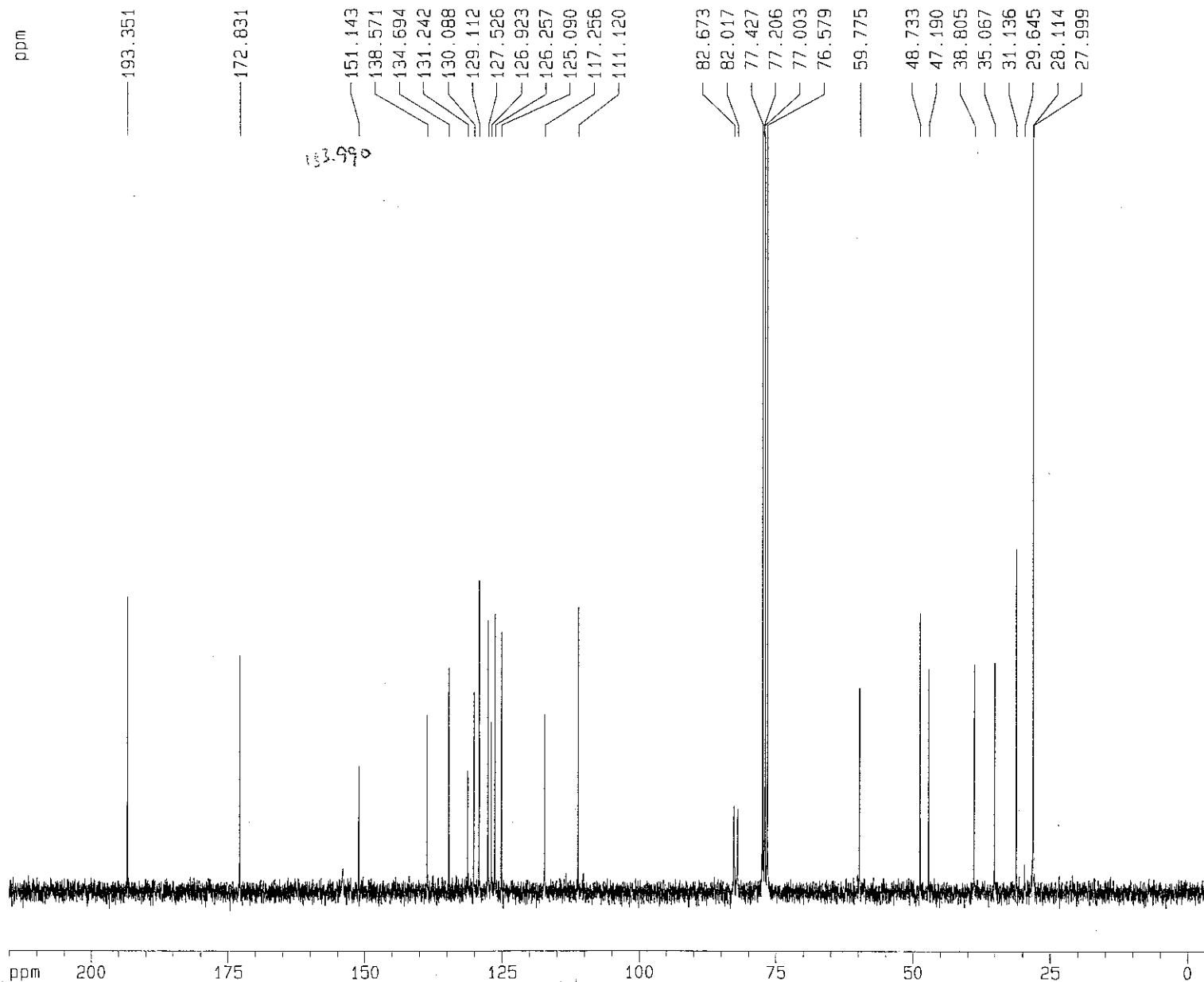
===== CHANNEL f1 =====  
 NUC1 1H  
 P1 11.70 usec  
 PL1 0.00 dB  
 SF01 299.8718518 MHz

F2 - Processing parameters  
 SI 32768  
 SF 299.8700161 MHz  
 WDW no  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 11.000 ppm  
 F1 3298.57 Hz  
 F2P -1.000 ppm  
 F2 -299.87 Hz  
 PPMCM 0.60000 ppm/cm  
 HZCM 179.92201 Hz/cm



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## Current Data Parameters

NAME PL-Oct28-09  
EXPNO 2  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20091028  
Time 12.31  
INSTRUM spect  
PROBHD 5 mm GNP 1H/1  
PULPROG zgpg30  
TD 65536  
SOLVENT CDC13  
NS 540  
DS 4  
SWH 18796.992 Hz  
FIDRES 0.286819 Hz  
AQ 1.7433076 sec  
RG 512  
DW 26.600 usec  
DE 6.00 usec  
TE 300.0 K  
D1 2.0000000 sec  
D11 0.0300000 sec  
D12 0.0000200 sec

## ===== CHANNEL f1 =====

NUC1 13C  
P1 5.40 usec  
PL1 -6.00 dB  
SF01 75.4106357 MHz

## ===== CHANNEL f2 =====

CPOPRG2 waltz16  
NUC2 1H  
PCPD2 115.00 usec  
PL2 0.00 dB  
PL12 20.00 dB  
PL13 20.00 dB  
SF02 299.8711995 MHz

## F2 - Processing parameters

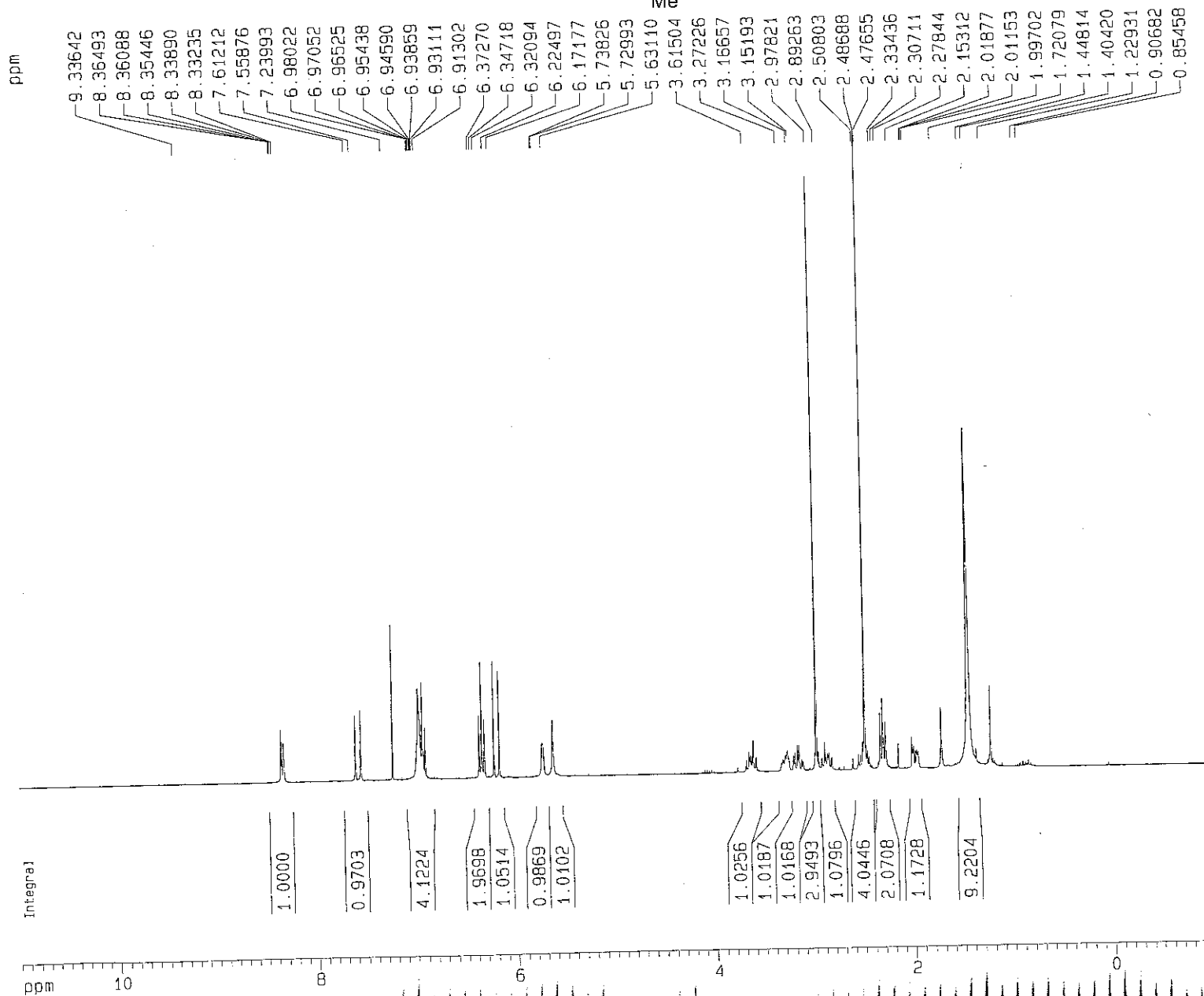
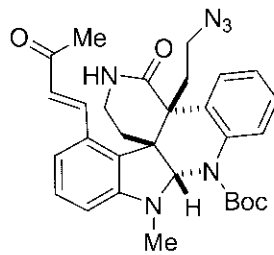
SI 32768  
SF 75.4023775 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

## 1D NMR plot parameters

CX 20.00 cm  
F1P 215.000 ppm  
F1 16211.51 Hz  
F2P -5.000 ppm  
F2 -377.01 Hz  
PPMCM 11.00000 ppm/cm  
HZCM 829.42615 Hz/cm



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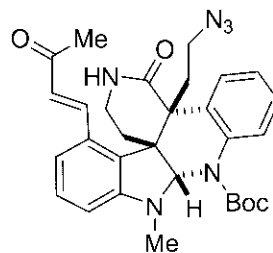
Current Data Parameters  
 NAME PL-Oct29-09  
 EXPNO 3  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20091029  
 Time 18.49  
 INSTRUM spect  
 PROBHD 5 mm QNP 1H/1  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 16  
 DS 2  
 SWH 6172.839 Hz  
 FIDRES 0.094190 Hz  
 AQ 5.3084660 sec  
 RG 203.2  
 DW 81.000 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 1.00000000 sec

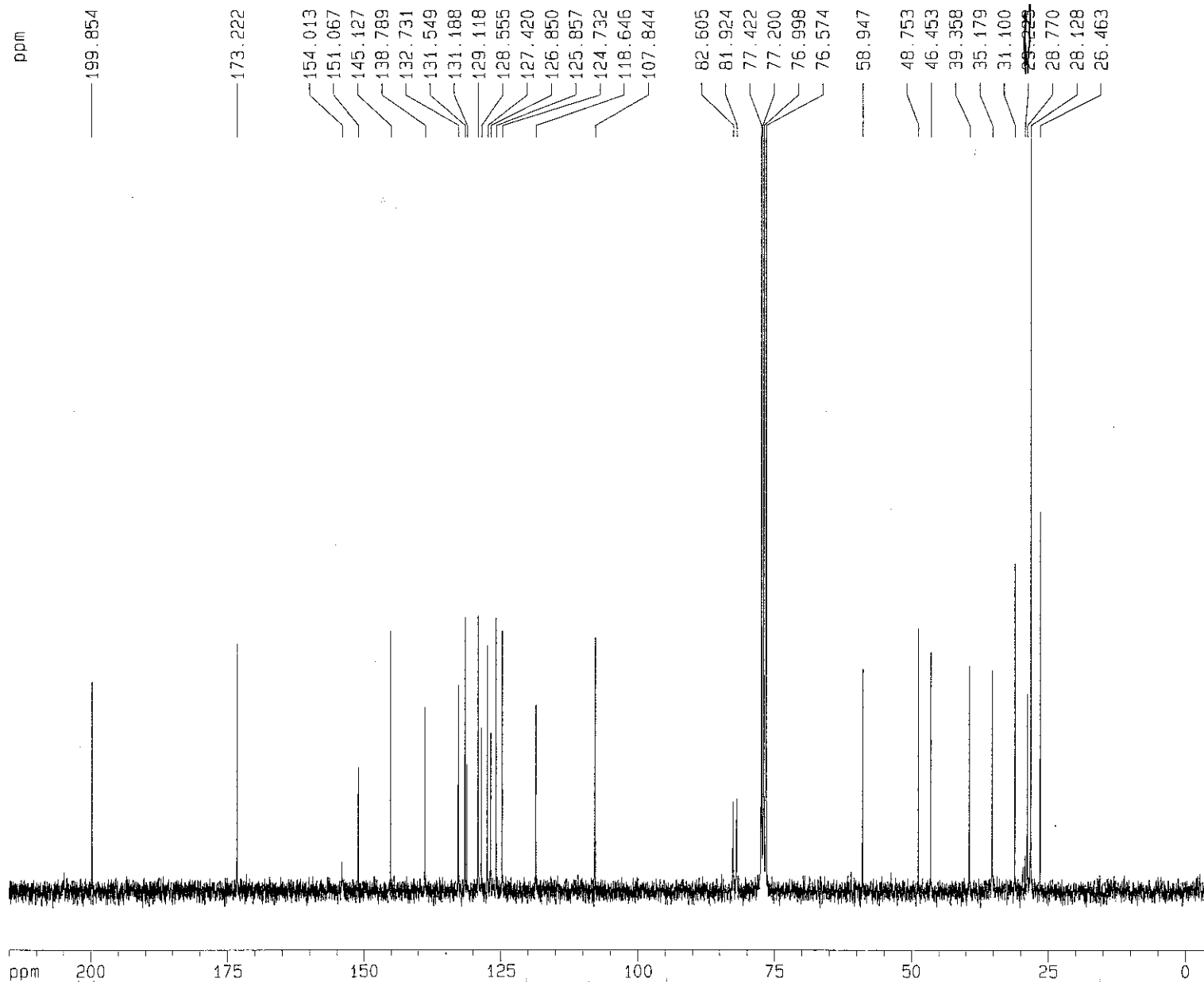
===== CHANNEL f1 =====  
 NUC1 1H  
 P1 11.70 usec  
 PL1 0.00 dB  
 SFO1 299.8718518 MHz

F2 - Processing parameters  
 SI 32768  
 SF 299.8700161 MHz  
 WDW no  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 11.000 ppm  
 F1 3298.57 Hz  
 F2P -1.000 ppm  
 F2 -299.87 Hz  
 PPMCM 0.60000 ppm/cm  
 HZCM 179.92201 Hz/cm



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## Current Data Parameters

NAME PL-Oct29-09  
EXPNO 4  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20091029  
Time 18.55  
INSTRUM spect  
PROBHD 5 mm GNP 1H/1  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 1230  
DS 4  
SWH 18796.992 Hz  
FIDRES 0.286819 Hz  
AQ 1.7433076 sec  
RG 4096  
DW 26.600 usec  
DE 6.00 usec  
TE 300.0 K  
D1 2.0000000 sec  
D11 0.0300000 sec  
D12 0.0002000 sec

## ===== CHANNEL f1 =====

NUC1 <sup>13</sup>C  
P1 5.40 usec  
PL1 -6.00 dB  
SFO1 75.4106357 MHz

## ===== CHANNEL f2 =====

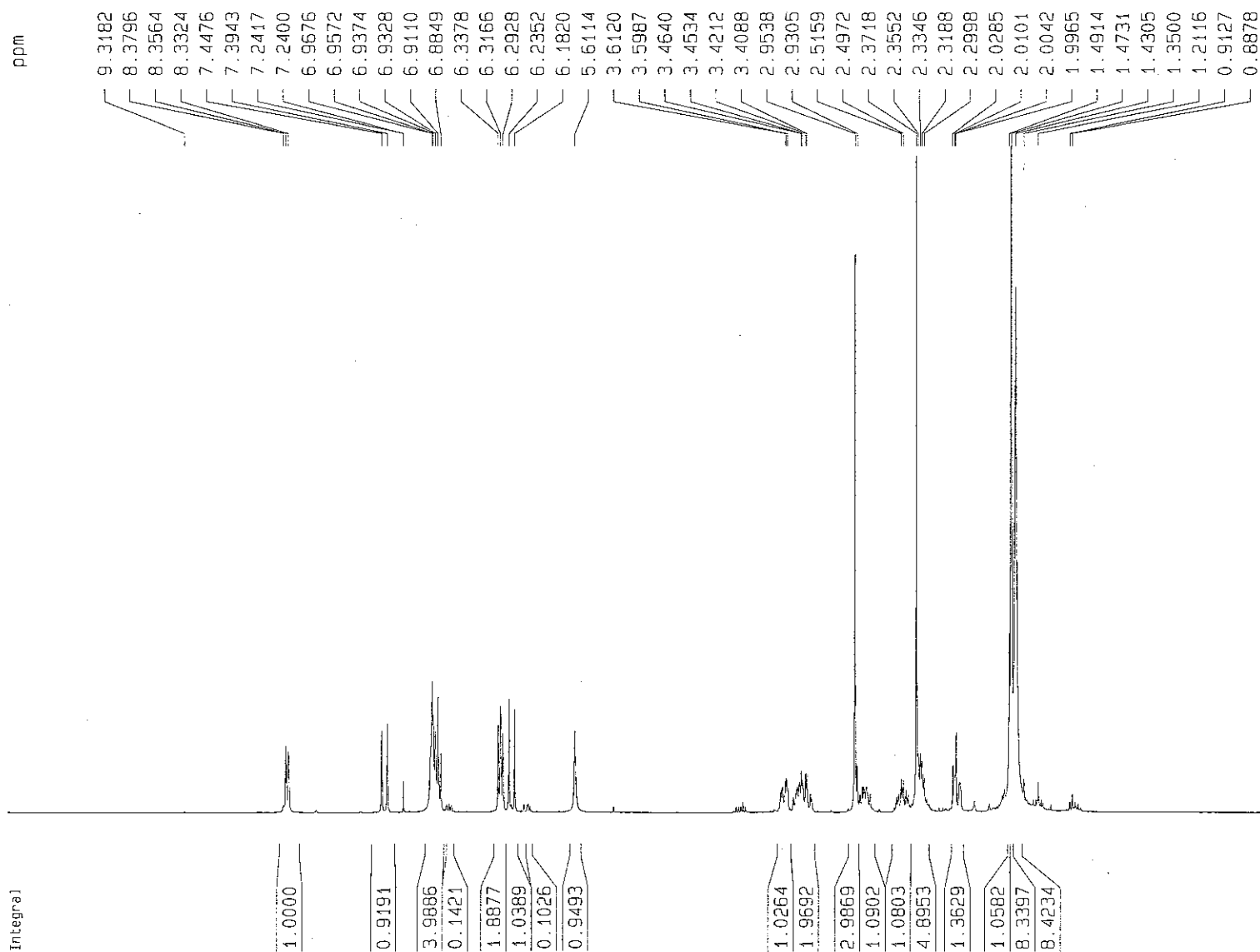
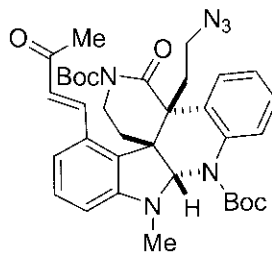
CPDPRG2 waltz16  
NUC2 <sup>1</sup>H  
PCPD2 115.00 usec  
PL2 0.00 dB  
PL12 20.00 dB  
PL13 20.00 dB  
SFO2 299.8711995 MHz

## F2 - Processing parameters

SF 32768  
SF 75.4023769 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

## 1D NMR plot parameters

CX 20.00 cm  
F1P 215.000 ppm  
F1 16211.51 Hz  
F2P -5.000 ppm  
F2 -377.01 Hz  
PPMCM 11.00000 ppm/cm  
HZCM 829.42615 Hz/cm



## Current Data Parameters

NAME PL-Nov01-09  
EXPNO 3  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20091101  
Time 12.50  
INSTRUM spect  
PROBHD 5 mm GNP 1H/1  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6172.839 Hz  
FIDRES 0.094190 Hz  
AQ 5.3084660 sec  
RG 80.6  
DW 81.000 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec

## ===== CHANNEL f1 =====

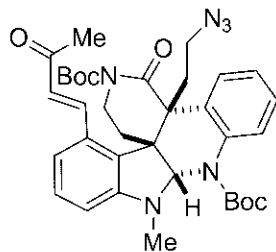
NUC1 1H  
P1 11.70 usec  
PL1 0.00 dB  
SFO1 299.8718518 MHz

## F2 - Processing parameters

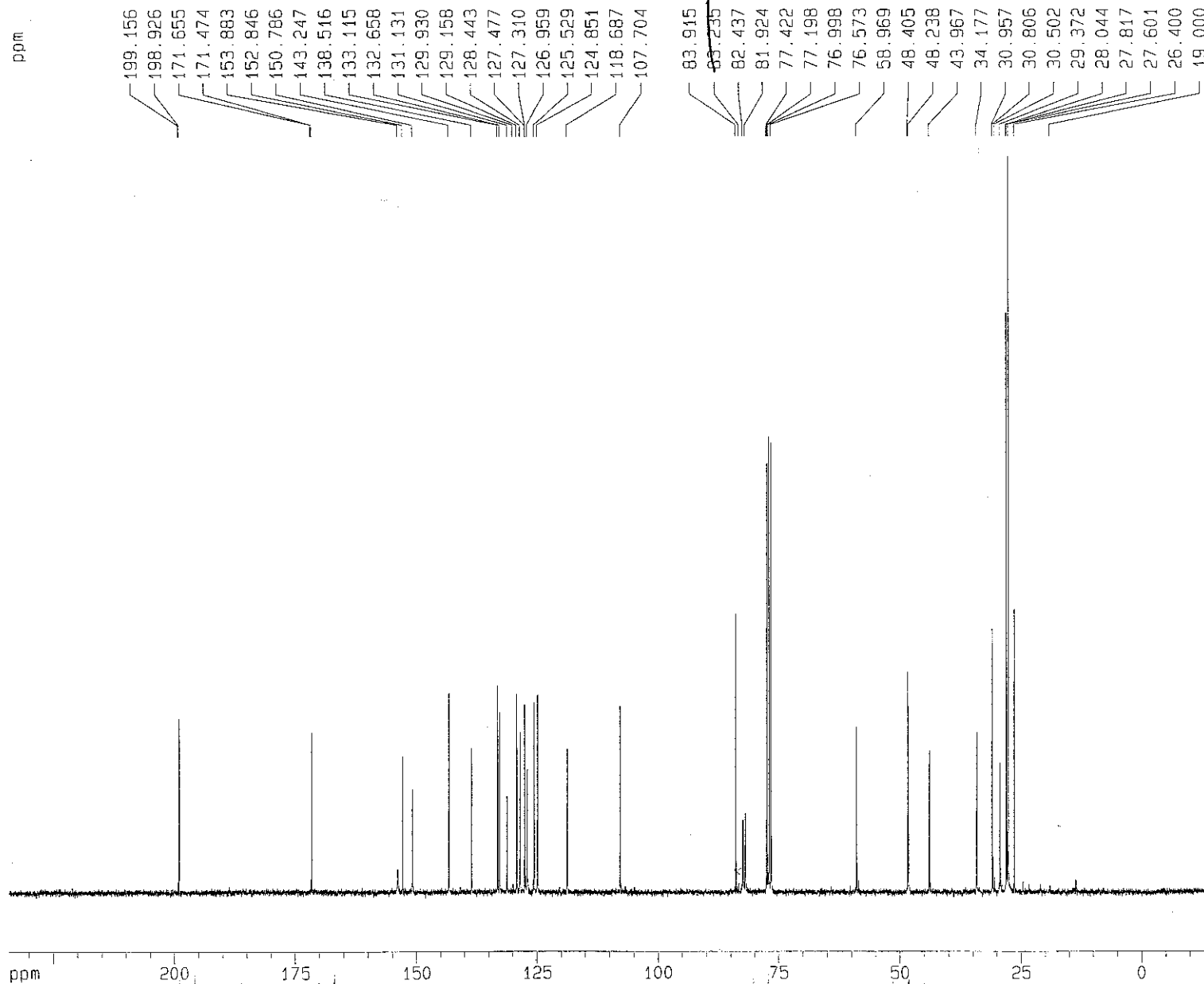
SI 32768  
SF 299.8700157 MHz  
WDW no  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

## 1D NMR plot parameters

CX 20.00 cm  
F1P 11.000 ppm  
F1 3298.57 Hz  
F2P -1.000 ppm  
F2 -299.87 Hz  
PPMCM 0.60000 ppm/cm  
HZCM 179.92201 Hz/cm



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Current Data Parameters  
 NAME PL-Nov01-09  
 EXPNO 4  
 PROCNO 1

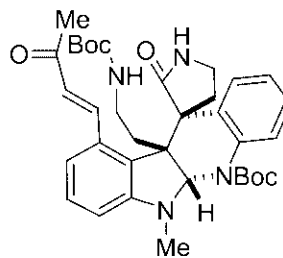
F2 - Acquisition Parameters  
 Date\_ 20091101  
 Time 12.57  
 INSTRUM spect  
 PROBHD 5 mm GNP 1H/1  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 1028  
 DS 4  
 SWH 18796.992 Hz  
 FIDRES 0.286819 Hz  
 AQ 1.7433076 sec  
 RG 4096  
 DW 26.600 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 2.0000000 sec  
 D11 0.0300000 sec  
 D12 0.0002000 sec

===== CHANNEL f1 =====  
 NUC1 <sup>13</sup>C  
 P1 5.40 usec  
 PL1 -6.00 dB  
 SFO1 75.4106357 MHz

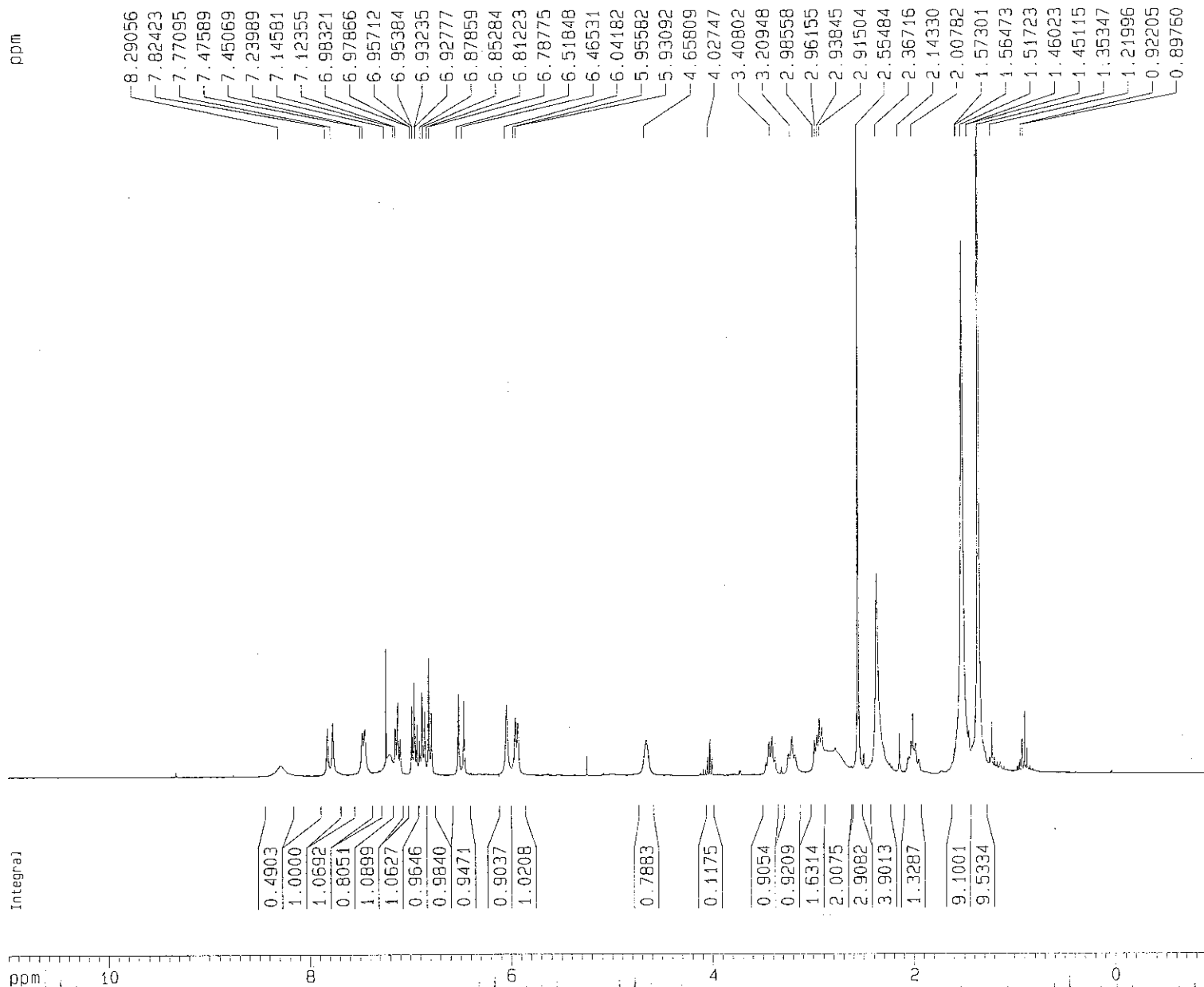
===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 <sup>1</sup>H  
 PCPD2 115.00 usec  
 PL2 0.00 dB  
 PL12 20.00 dB  
 PL13 20.00 dB  
 SFO2 299.8711995 MHz

F2 - Processing parameters  
 SI 32768  
 SF 75.4023615 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 234.114 ppm  
 F1 17652.77 Hz  
 F2P -15.175 ppm  
 F2 -1144.22 Hz  
 PPMCM 12.46445 ppm/cm  
 HZCM 939.84949 Hz/cm



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## Current Data Parameters

NAME PL-Nov03-09  
EXPNO 2  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20091103  
Time 17.33  
INSTRUM spect  
PROBHD 5 mm QNP 1H/1  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6172.839 Hz  
FIDRES 0.094190 Hz  
AQ 5.3064660 sec  
RG 80.6  
DW 81.000 usec  
DE 6.00 usec  
TE 300.0 K  
O1 1.00000000 sec

## ===== CHANNEL f1 =====

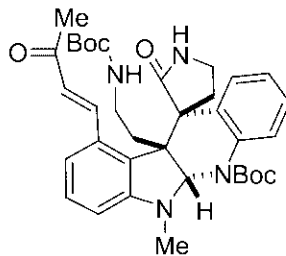
NUC1 1H  
P1 11.70 usec  
PL1 0.00 dB  
SFO1 299.8718516 MHz

## F2 - Processing parameters

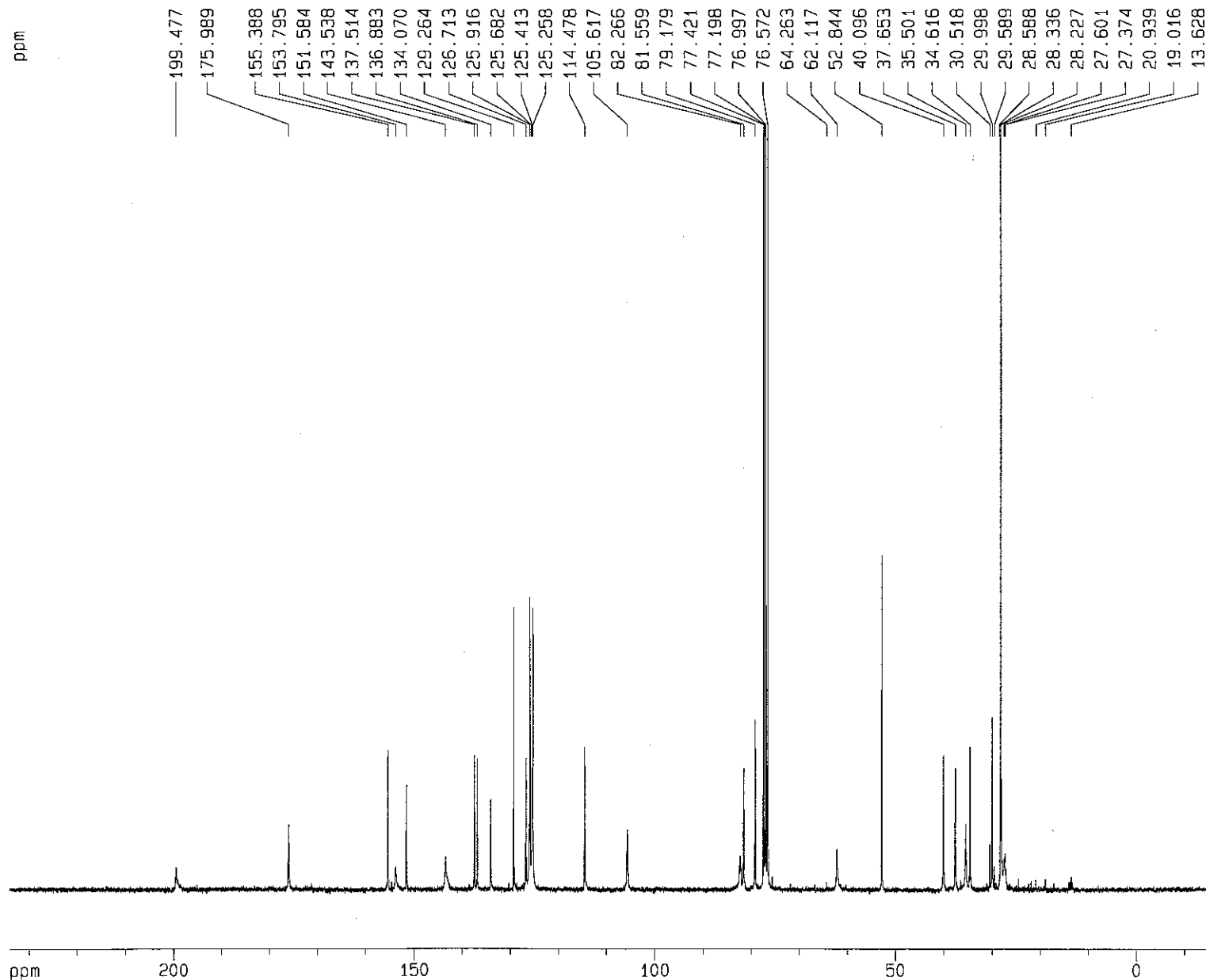
SF 32768  
SF 299.8700159 MHz  
WDW no  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

## 1D NMR plot parameters

CX 20.00 cm  
F1P 11.000 ppm  
F1 3298.57 Hz  
F2P -1.000 ppm  
F2 -299.87 Hz  
PPMCM 0.60000 ppm/cm  
HZCM 179.92201 Hz/cm



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## Current Data Parameters

NAME PL-Nov03-09  
EXPNO 5  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20091103  
Time 21.26  
INSTRUM spect  
PROBHD 5 mm GNP 1H/1  
PULPROG zgpg30  
TD 65536  
SOLVENT CDC13  
NS 12000  
DS 4  
SWH 16796.992 Hz  
FIDRES 0.286819 Hz  
AQ 1.7433076 sec  
RG 4096  
DW 26.600 usec  
DE 6.00 usec  
TE 300.0 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
D12 0.00002000 sec

## ===== CHANNEL f1 =====

NUC1 13C  
P1 5.40 usec  
PL1 -6.00 dB  
SF01 75.4106357 MHz

## ===== CHANNEL f2 =====

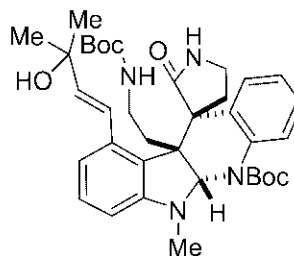
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 115.00 usec  
PL2 0.00 dB  
PL12 20.00 dB  
PL13 20.00 dB  
SF02 299.8711995 MHz

## F2 - Processing parameters

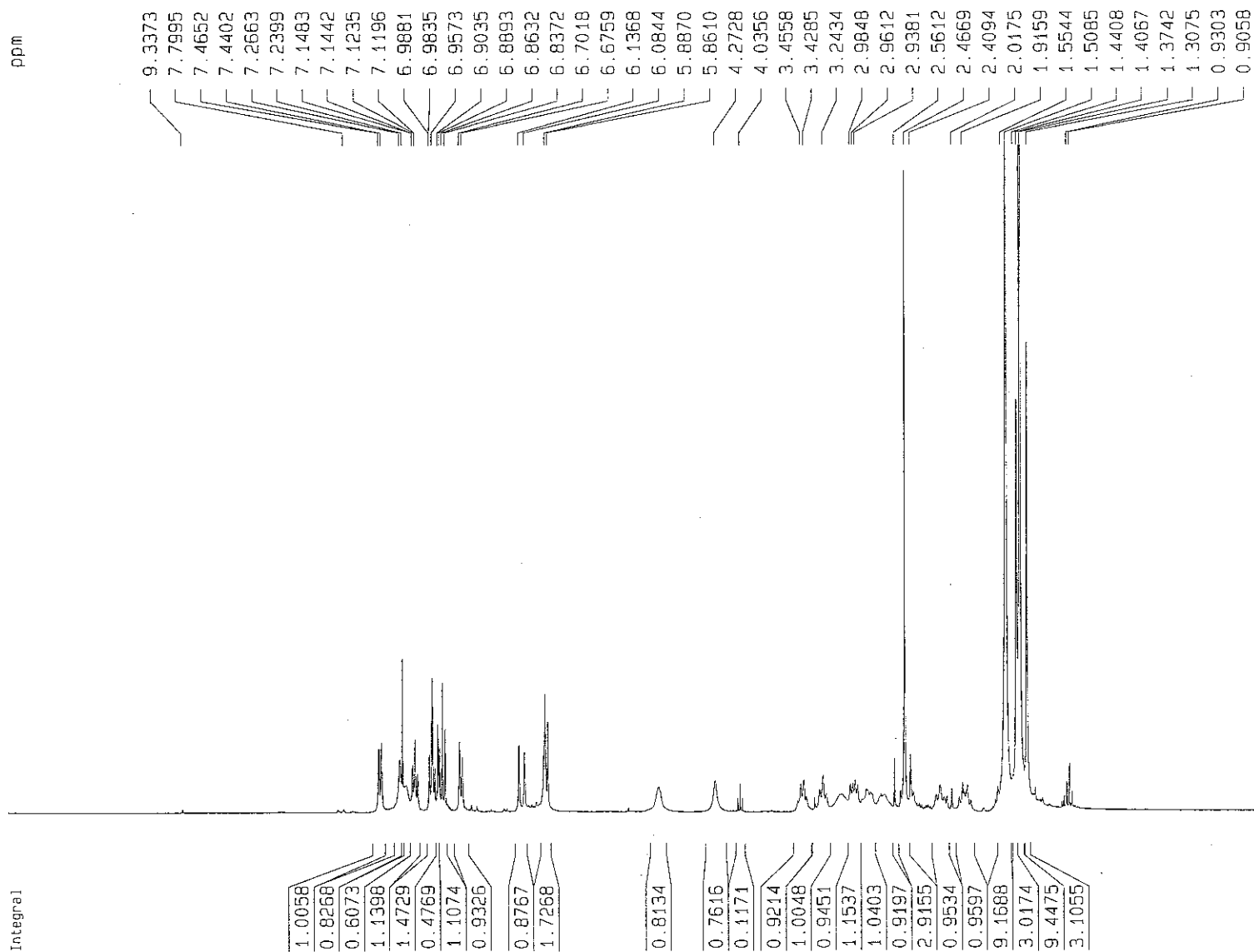
SI 32768  
SF 75.4023809 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

## 1D NMR plot parameters

CX 20.00 cm  
F1P 234.122 ppm  
F1 17653.34 Hz  
F2P -15.167 ppm  
F2 -1143.65 Hz  
PPMCM 12.46446 ppm/cm  
HZCM 939.84961 Hz/cm



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Current Data Parameters  
NAME PL-Nov06-09  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20091106  
Time 11.53  
INSTRUM spect  
PROBHD 5 mm QNP 1H/1  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6172.839 Hz  
FIDRES 0.094190 Hz  
AQ 5.3084660 sec  
RG 114  
DW 81.000 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec

===== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 0.00 dB  
SFO1 299.8718518 MHz

F2 - Processing parameters  
SI 32768  
SF 299.8700159 MHz  
WDW no  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

1D NMR plot parameters  
CX 20.00 cm  
F1P 11.000 ppm  
F1 3298.57 Hz  
F2P -1.000 ppm  
F2 -299.87 Hz  
PPMCM 0.60000 ppm/cm  
HZCM 179.92201 Hz/cm

ppm

10

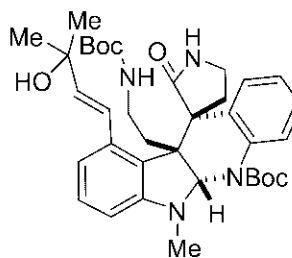
8

6

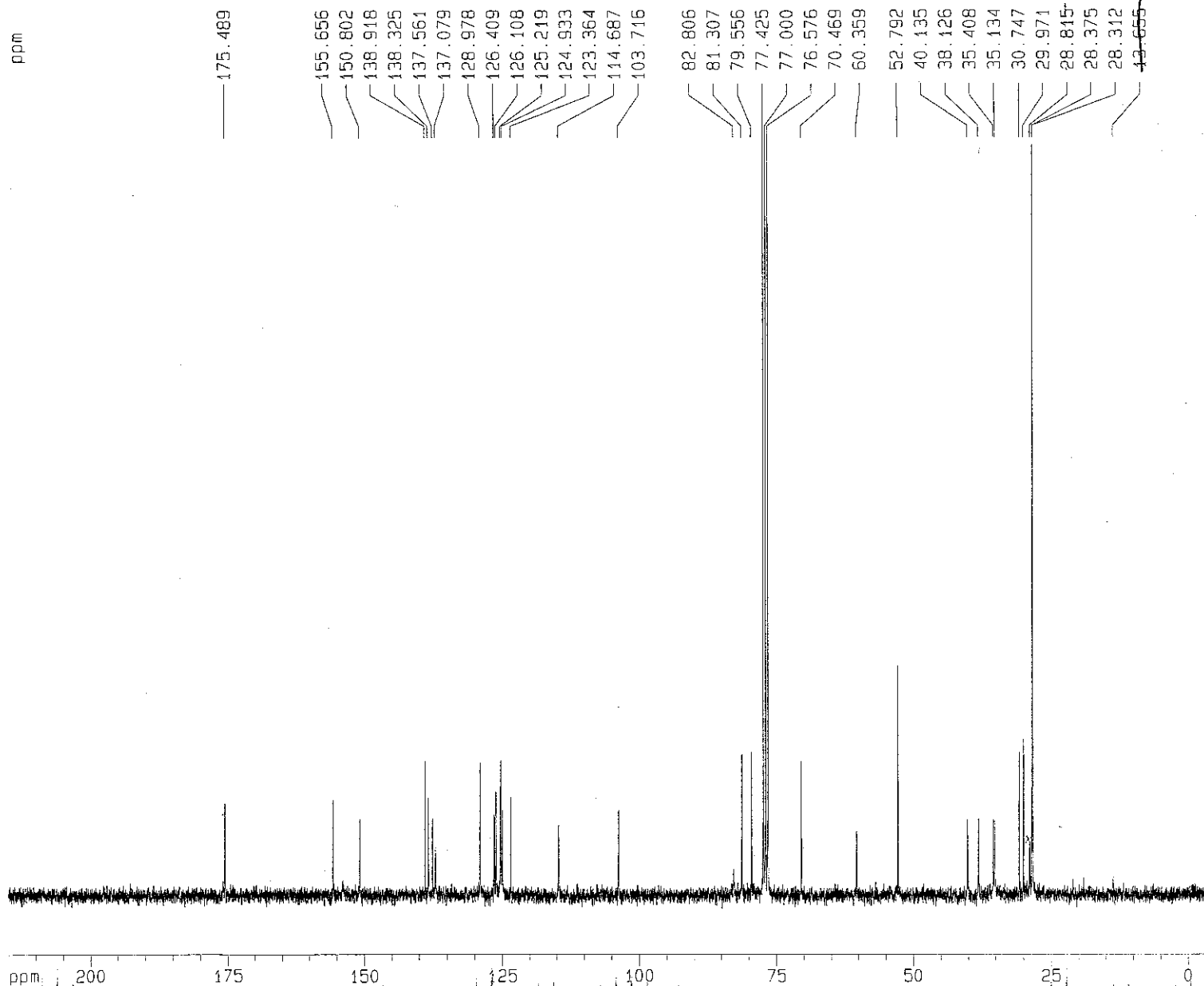
4

2

0



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## Current Data Parameters

NAME PL-Nov06-09  
EXPNO 2  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20091106  
Time 12.01  
INSTRUM spect  
PROBHD 5 mm QNP 1H/1  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 411  
DS 4  
SWH 18796.992 Hz  
FIDRES 0.286819 Hz  
AQ 1.7433076 sec  
RG 4096  
DW 26.600 usec  
DE 6.00 usec  
TE 300.0 K  
D1 2.0000000 sec  
D11 0.0300000 sec  
D12 0.0000200 sec

## ===== CHANNEL f1 =====

NUC1 <sup>13</sup>C  
P1 5.40 usec  
PL1 -6.00 dB  
SF01 75.4106357 MHz

## ===== CHANNEL f2 =====

CPDPRG2 waltz16  
NUC2 <sup>1</sup>H  
PCPD2 115.00 usec  
PL2 0.00 dB  
PL12 20.00 dB  
PL13 20.00 dB  
SF02 299.6711995 MHz

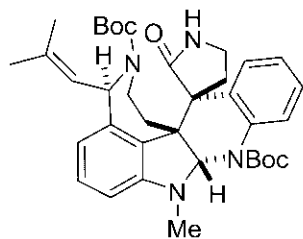
## F2 - Processing parameters

SI 32768  
SF 75.4023798 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
SB 0  
PC 1.40

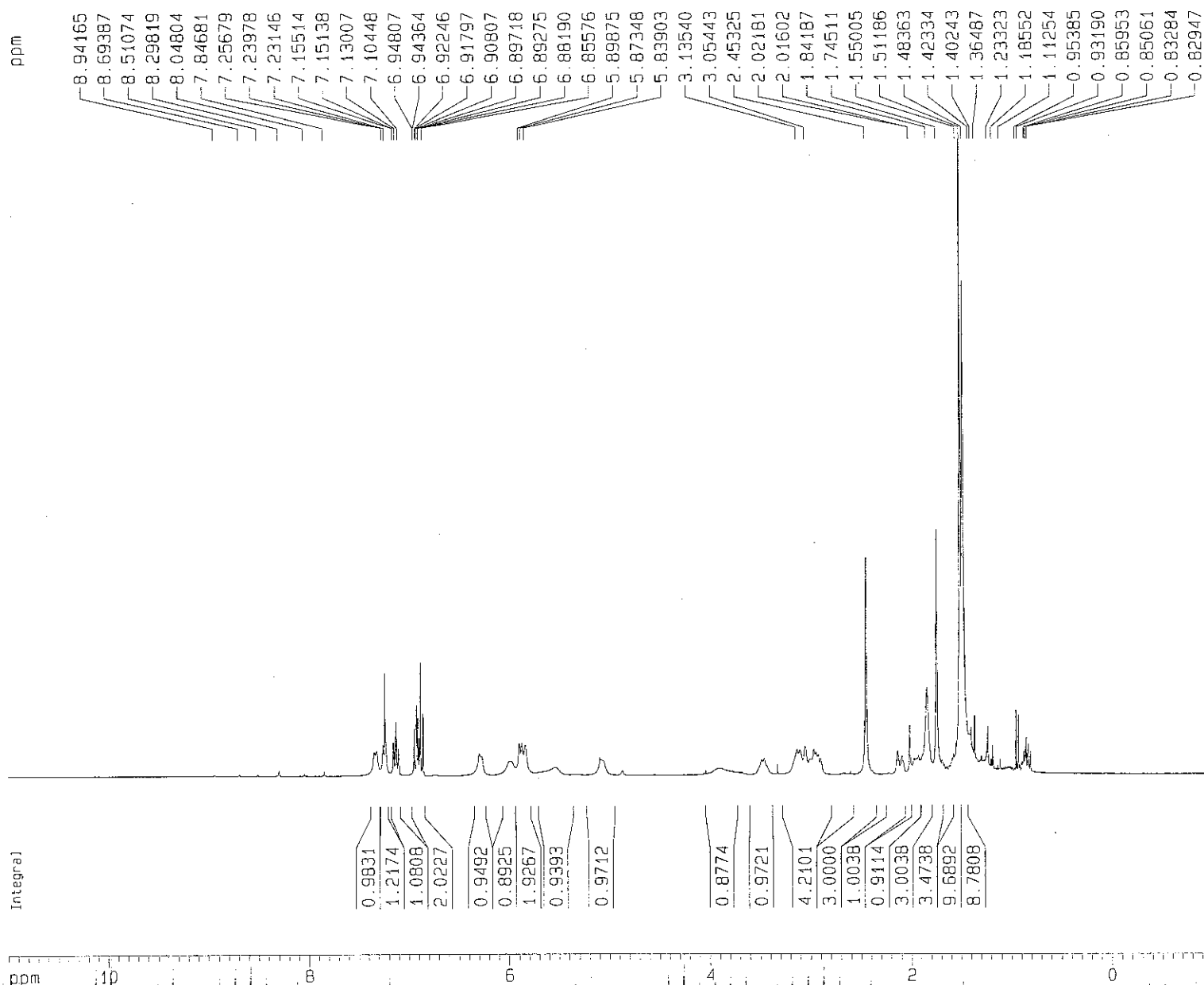
## 1D NMR plot parameters

CX 20.00 cm  
F1P 215.000 ppm  
F1 16211.51 Hz  
F2P -5.000 ppm  
F2 -377.01 Hz  
PPMCM 11.00000 ppm/cm  
HZCM 829.42621 Hz/cm





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## Current Data Parameters

NAME PL-Nov07-09  
 EXPNO 1  
 PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20091107  
 Time 11.50  
 INSTRUM spect  
 PROBHD 5 mm QNP 1H/1  
 PULPROG zg30  
 TD 65536  
 SOLVENT CDC13  
 NS 16  
 DS 2  
 SWH 6172.839 Hz  
 FIDRES 0.094190 Hz  
 AQ 5.3084660 sec  
 RG 128  
 QW 81.000 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 1.00000000 sec

## ===== CHANNEL f1 =====

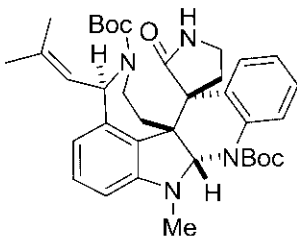
NUC1 1H  
 P1 11.70 usec  
 PL1 0.00 dB  
 SFO1 299.8718516 MHz

## F2 - Processing parameters

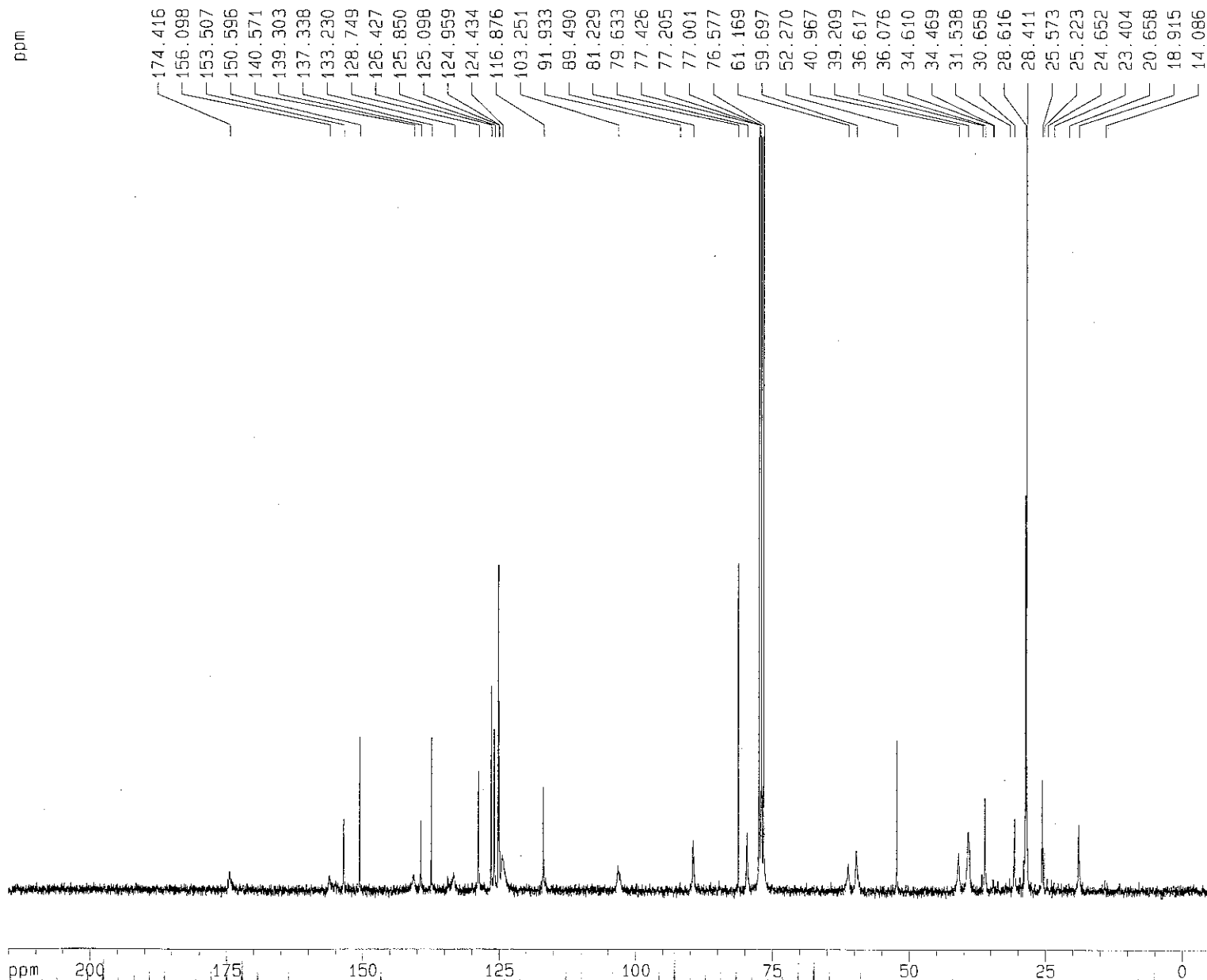
SI 32768  
 SF 299.8700161 MHz  
 WDW no  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

## 1D NMR plot parameters

CX 20.00 cm  
 F1P 11.000 ppm  
 F1 3298.57 Hz  
 F2P -1.000 ppm  
 F2 -299.87 Hz  
 PPMCM 0.60000 ppm/cm  
 HZCM 179.92201 Hz/cm



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## Current Data Parameters

NAME PL-Nov07-09  
EXPNO 3  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20091107  
Time 13.18  
INSTRUM spect  
PROBHD 5 mm GNP 1H/1  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 6686  
DS 4  
SWH 18796.992 Hz  
FIDRES 0.286819 Hz  
AQ 1.7433076 sec  
RG 1024  
DW 26.600 usec  
DE 6.00 usec  
TE 300.0 K  
D1 2.0000000 sec  
D11 0.0300000 sec  
D12 0.0000200 sec

## ===== CHANNEL f1 =====

NUC1 13C  
P1 5.40 usec  
PL1 -6.00 dB  
SF01 75.4106357 MHz

## ===== CHANNEL f2 =====

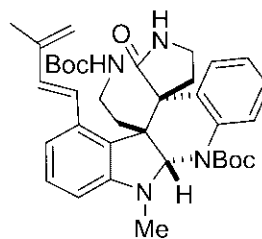
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 115.00 usec  
PL2 0.00 dB  
PL12 20.00 dB  
PL13 20.00 dB  
SF02 299.8711995 MHz

## F2 - Processing parameters

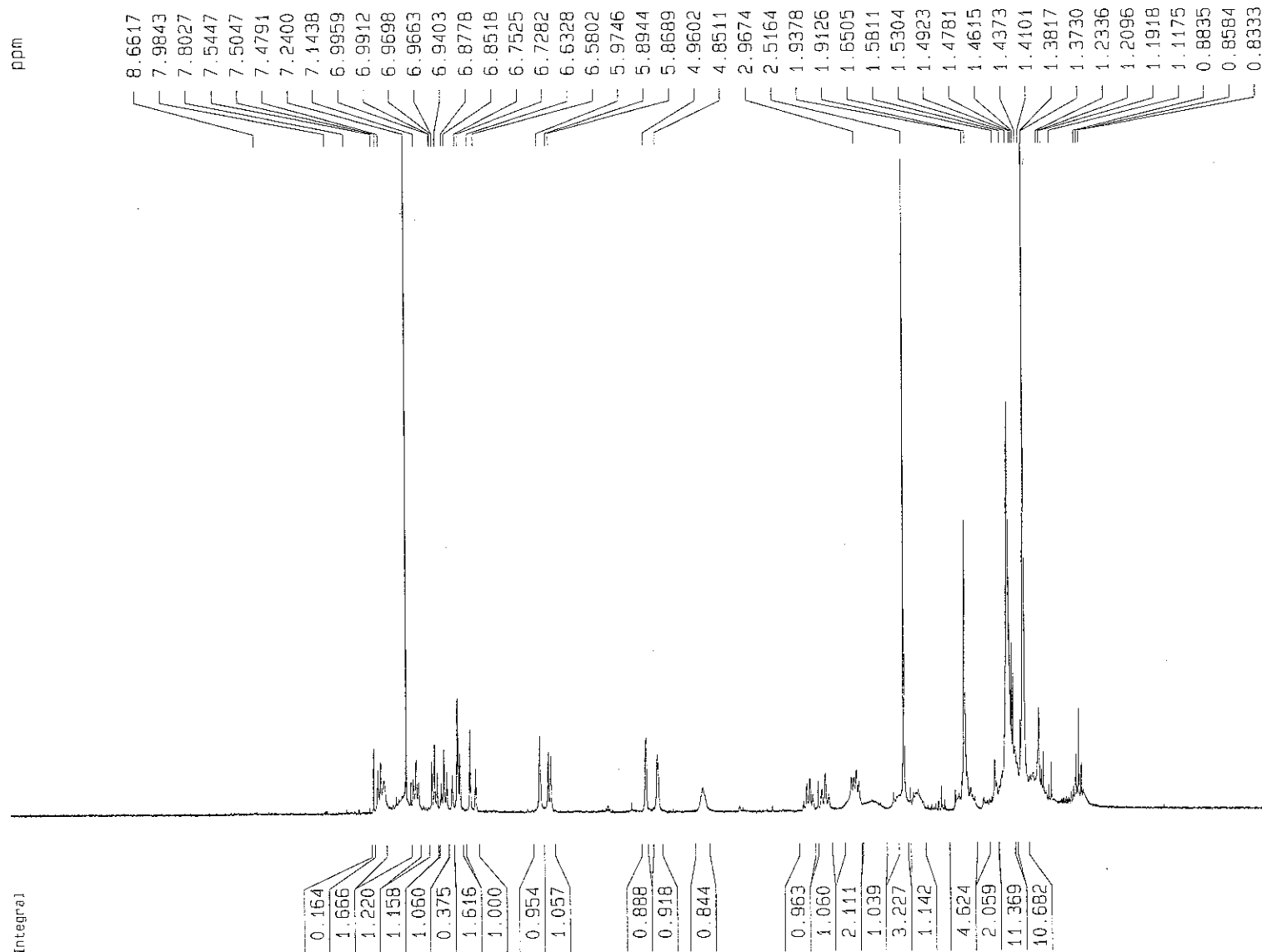
SI 32768  
SF 75.4023775 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

## 1D NMR plot parameters

CX 20.00 cm  
F1P 215.000 ppm  
F1 16211.51 Hz  
F2P -377.01 Hz  
F2 -377.01 Hz  
PPMCM 11.00000 ppm/cm  
HZCM 829142615 Hz/cm



60



## Current Data Parameters

NAME PL-Nov07-09  
EXPNO 5  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20091107  
Time 21.02  
INSTRUM spect  
PROBHD 5 mm QNP 1H/1  
PULPROG zg30  
TD 65536  
SOLVENT CDC13  
NS 16  
DS 2  
SWH 6172.839 Hz  
FIDRES 0.094190 Hz  
AQ 5.3084660 sec  
RG 406.4  
DW 81.000 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec

## ===== CHANNEL f1 =====

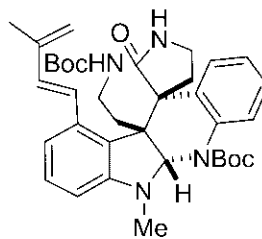
NUC1 1H  
P1 11.70 usec  
PL1 0.00 dB  
SFO1 299.8718518 MHz

## F2 - Processing parameters

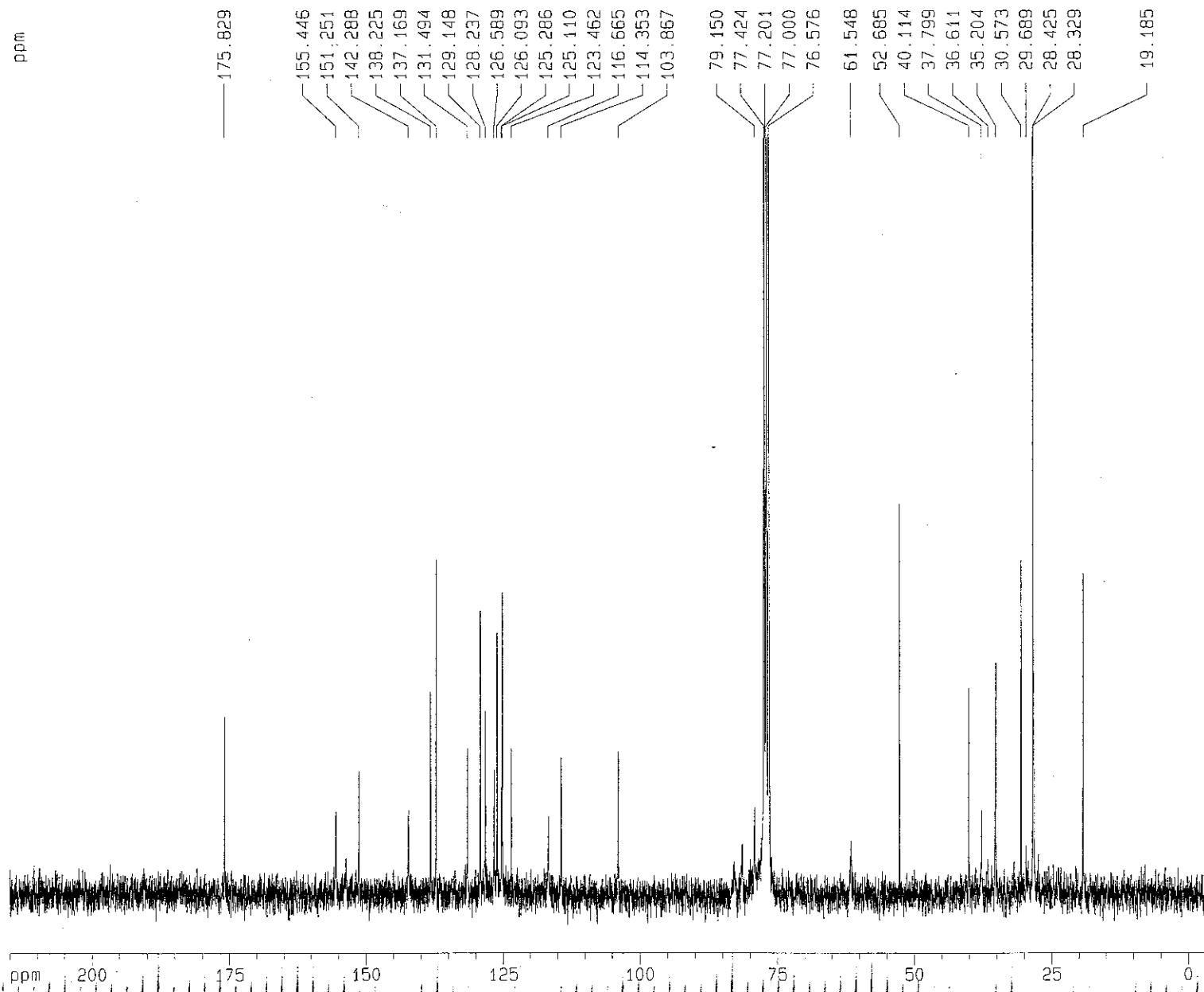
SI 32768  
SF 299.8700161 MHz  
WDW no  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

## 1D NMR plot parameters

CX 20.00 cm  
F1P 11.000 ppm  
F1 3298.57 Hz  
F2P -1.000 ppm  
F2 -299.87 Hz  
PPMCM 0.60000 ppm/cm  
HZCM 179.92201 Hz/cm



60



## Current Data Parameters

NAME PL-Nov07-09  
EXPNO 6  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20091107  
Time 21.12  
INSTRUM spect  
PROBHD 5 mm GNP 1H/1  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 12293  
DS 4  
SWH 16796.992 Hz  
FIDRES 0.286819 Hz  
AQ 1.7433076 sec  
RG 2048  
DW 26.600 usec  
DE 6.00 usec  
TE 300.0 K  
D1 2.0000000 sec  
D11 0.0300000 sec  
D12 0.00002000 sec

## ===== CHANNEL f1 =====

NUC1 13C  
P1 5.40 usec  
PL1 -6.00 dB  
SFO1 75.4106357 MHz

## ===== CHANNEL f2 =====

CPDPRG2 waltz16  
NUC2 1H  
PCPD2 115.00 usec  
PL2 0.00 dB  
PL12 20.00 dB  
PL13 20.00 dB  
SFO2 299.8711995 MHz

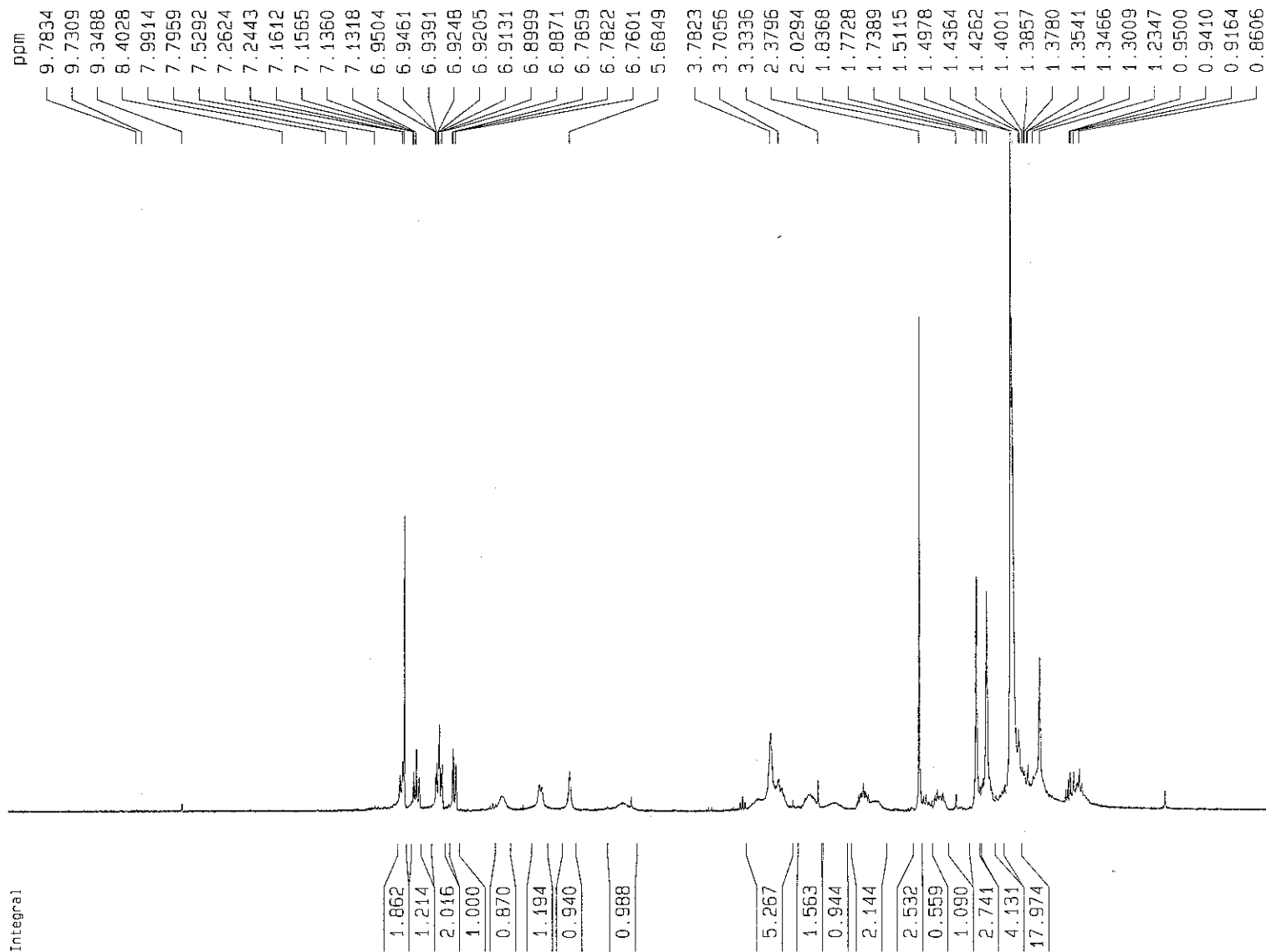
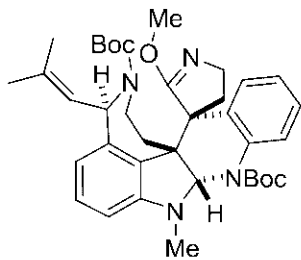
## F2 - Processing parameters

SI 32768  
SF 75.4023746 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

## 1D NMR plot parameters

CX 20.00 cm  
F1P 215.000 ppm  
F1 16211.51 Hz  
F2P -5.000 ppm  
F2 -377.01 Hz  
PPMCM 11.00000 ppm/cm  
HZCM 829.42611 Hz/cm

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## Current Data Parameters

NAME PL-Nov09-09  
EXPNO 1  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20091109  
Time 10.41  
INSTRUM spect  
PROBHD 5 mm QNP 1H/1  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6172.839 Hz  
FIDRES 0.094190 Hz  
AQ 5.3084660 sec  
RG 322.5  
DW 81.000 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec

## ----- CHANNEL f1 -----

NUC1 1H  
P1 11.70 usec  
PL1 0.00 dB  
SFO1 299.8718518 MHz

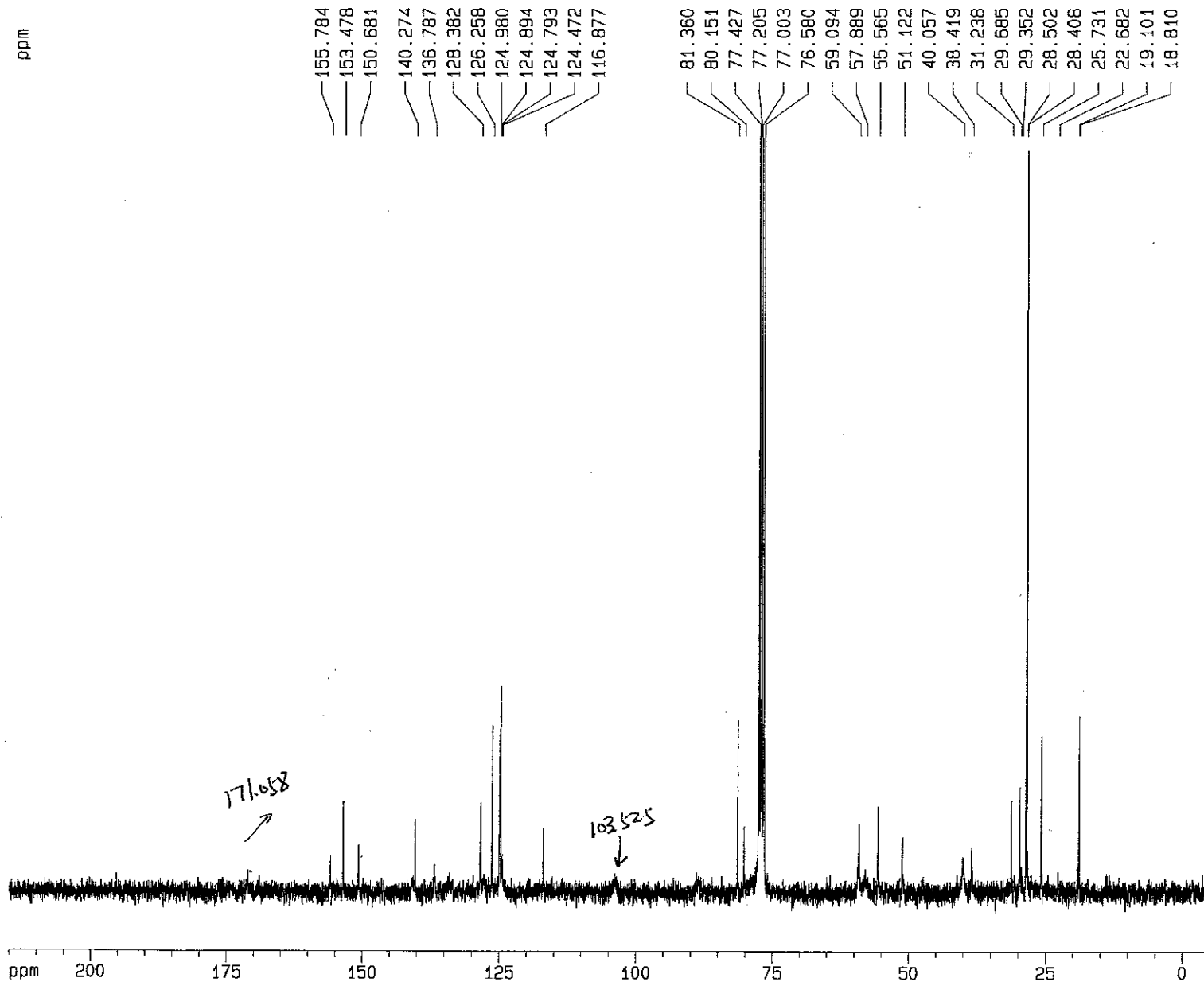
## F2 - Processing parameters

SI 32768  
SF 299.8700148 MHz  
WDW no  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

## 1D NMR plot parameters

CX 20.00 cm  
F1P 11.000 ppm  
F1 3298.57 Hz  
F2P -1.000 ppm  
F2 -299.87 Hz  
PPMCM 0.60000 ppm/cm  
HZCM 179.92201 Hz/cm

ppm



## Current Data Parameters

NAME PL-Nov09-09  
EXPNO 2  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20091108  
Time 22.43  
INSTRUM spect  
PROBHD 5 mm Multinu  
PULPROG zgpg30  
TD 65536  
SOLVENT CDC13  
NS 10949  
DS 4  
SWH 18832.393 Hz  
FIDRES 0.287360 Hz  
AQ 1.7400308 sec  
RG 16384  
DW 26.550 usec  
DE 6.00 usec  
TE 300.0 K  
D1 2.0000000 sec  
d11 0.0300000 sec  
d12 0.0002000 sec

===== CHANNEL f1 =====  
NUC1 13C  
P1 11.80 usec  
PL1 0.00 dB  
SFO1 75.4760200 MHz

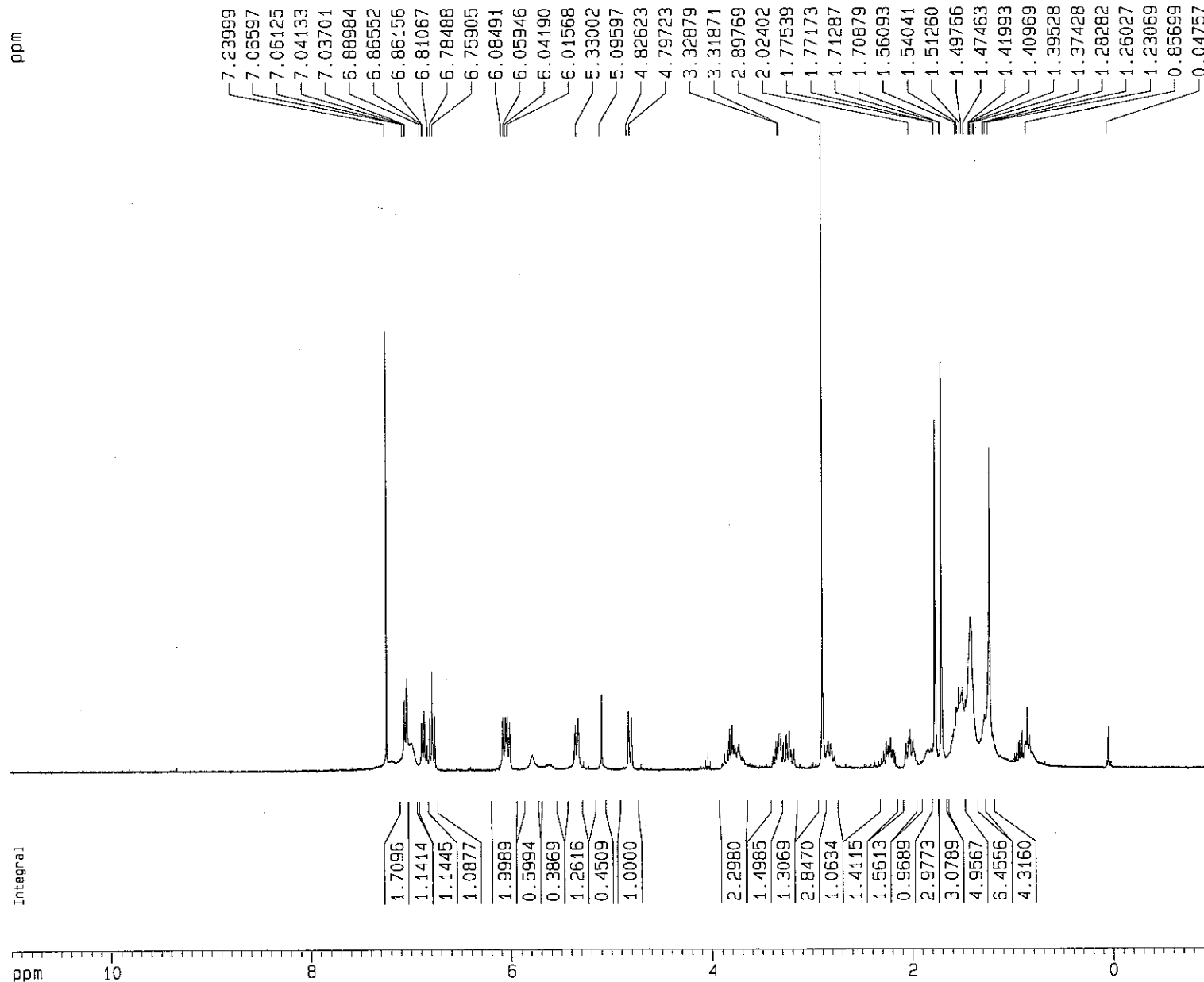
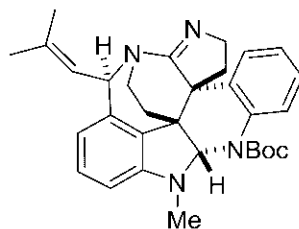
===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 110.00 usec  
PL2 0.00 dB  
PL12 17.50 dB  
PL13 17.50 dB  
SFO2 300.1312005 MHz

## F2 - Processing parameters

SI 32768  
SF 75.4677509 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

## 1D NMR plot parameters

CX 20.00 cm  
F1P 215.000 ppm  
F1 16225.57 Hz  
F2P -5.000 ppm  
F2 -377.34 Hz  
PPMCM 11.00000 ppm/cm  
HZCM 830.14525 Hz/cm



## Current Data Parameters

NAME PL-Nov12-09  
EXPNO 2  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20091112  
Time 20.18  
INSTRUM spect  
PROBHD 5 mm GNP 1H/1  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 6172.839 Hz  
FIDRES 0.094190 Hz  
AQ 5.3084660 sec  
RG 228.1  
DW 81.000 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec

## ===== CHANNEL f1 =====

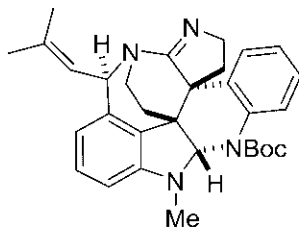
NUC1 1H  
P1 11.70 usec  
PL1 0.00 dB  
SF01 299.8718518 MHz

## F2 - Processing parameters

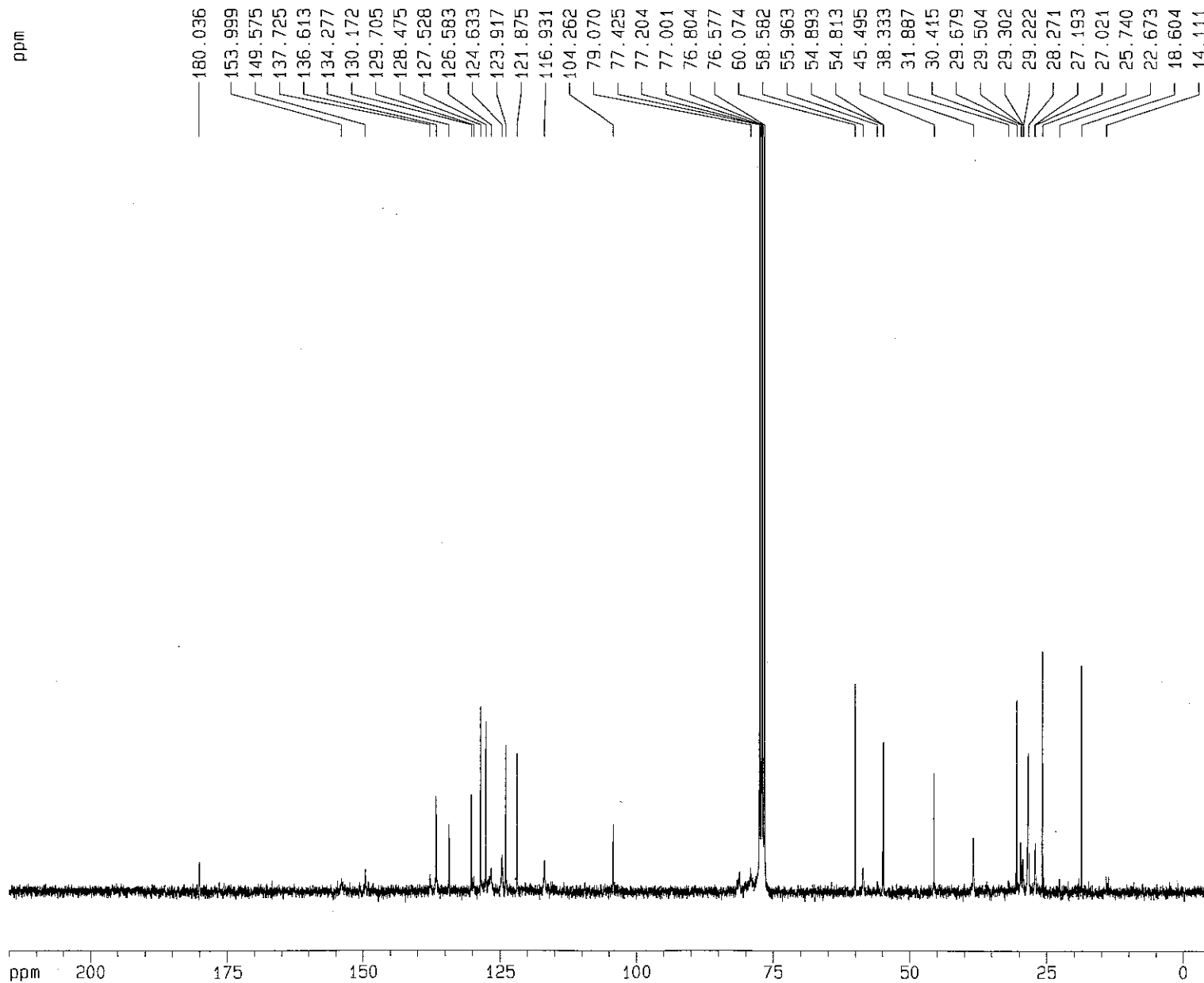
SI 32768  
SF 299.8700161 MHz  
WDW no  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

## 1D NMR plot parameters

CX 20.00 cm  
F1P 11.000 ppm  
F1 3298.57 Hz  
F2P -1.000 ppm  
F2 -299.87 Hz  
PPMCM 0.60000 ppm/cm  
HZCM 179.92201 Hz/cm



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Current Data Parameters  
NAME PL-Nov12-09  
EXPNO 4  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20091112  
Time 21.29  
INSTRUM spect  
PROBHD 5 mm QNP 1H/1  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 11367  
DS 4  
SWH 18796.992 Hz  
FIDRES 0.286819 Hz  
AQ 1.7433076 sec  
RG 1024  
DW 25.600 usec  
DE 6.00 usec  
TE 300.0 K  
D1 2.0000000 sec  
D11 0.0300000 sec  
D12 0.0002000 sec

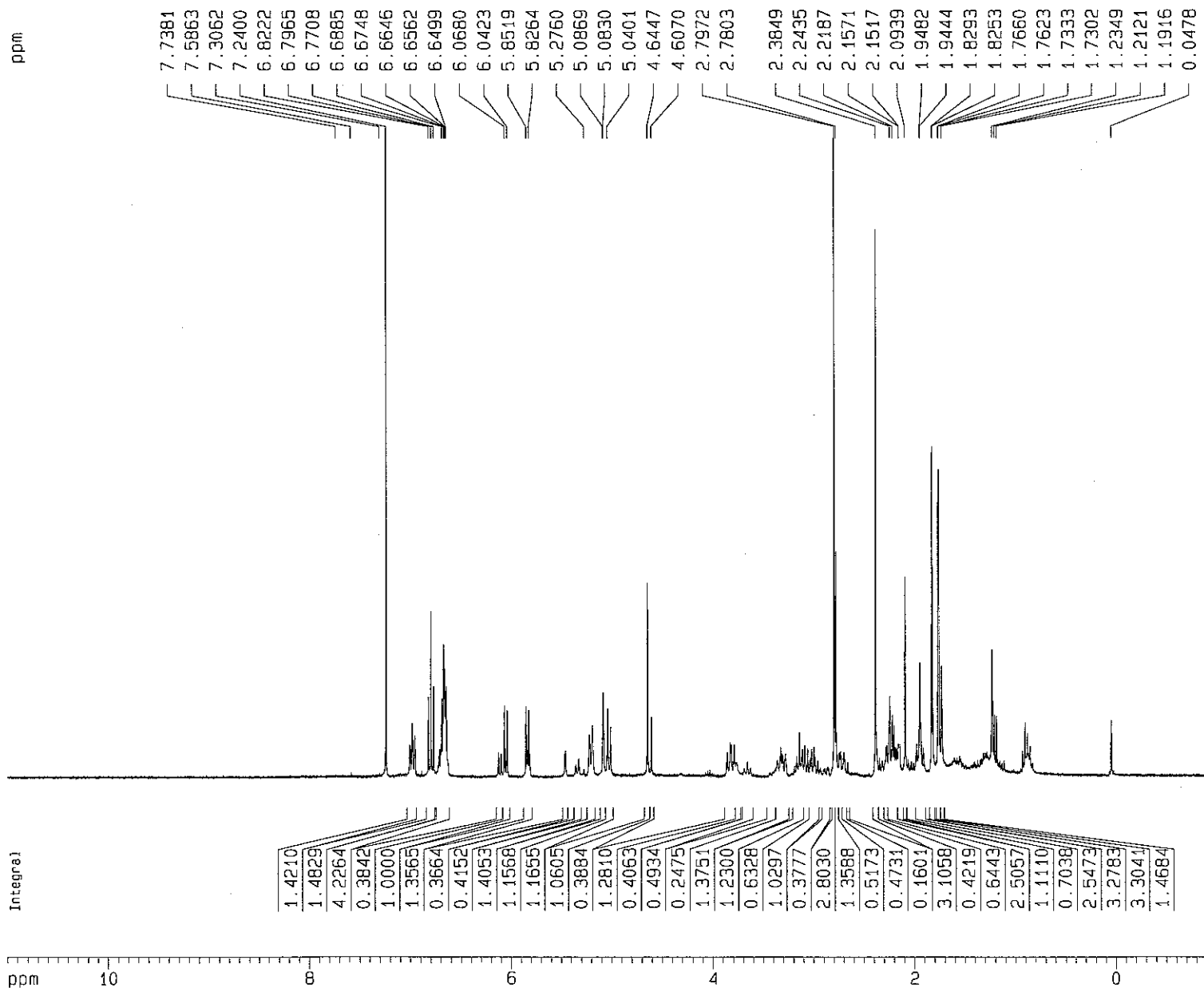
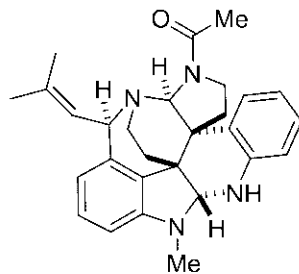
===== CHANNEL f1 =====  
NUC1 13C  
P1 5.40 usec  
PL1 -6.00 dB  
SF01 75.4106357 MHz

===== CHANNEL f2 =====  
CPOPRG2 waltz16  
NUC2 1H  
PCPD2 115.00 usec  
PL2 0.00 dB  
PL12 20.00 dB  
PL13 20.00 dB  
SF02 299.8711995 MHz

F2 - Processing parameters  
SI 32768  
SF 75.4023752 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

1D NMR plot parameters  
CX 20.00 cm  
F1P 215.000 ppm  
F1 16211.51 Hz  
F2P -5.000 ppm  
F2 -377.01 Hz  
PPMCM 11.00000 ppm/cm  
HZCM 829.42615 Hz/cm





## Current Data Parameters

NAME PL-Nov14-09  
EXPNO 1  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20091114  
Time 18.50  
INSTRUM spect  
PROBHD 5 mm GNP 1H/1  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 32  
DS 2  
SWH 6172.839 Hz  
FIDRES 0.094190 Hz  
AQ 5.3084660 sec  
RG 574.7  
DW 81.000 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.0000000 sec

## ===== CHANNEL f1 =====

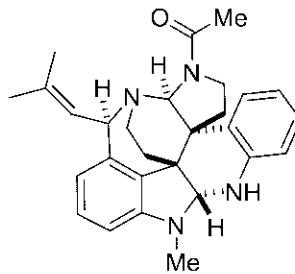
NUC1 1H  
P1 11.70 usec  
PL1 0.00 dB  
SFO1 299.8718518 MHz

## F2 - Processing parameters

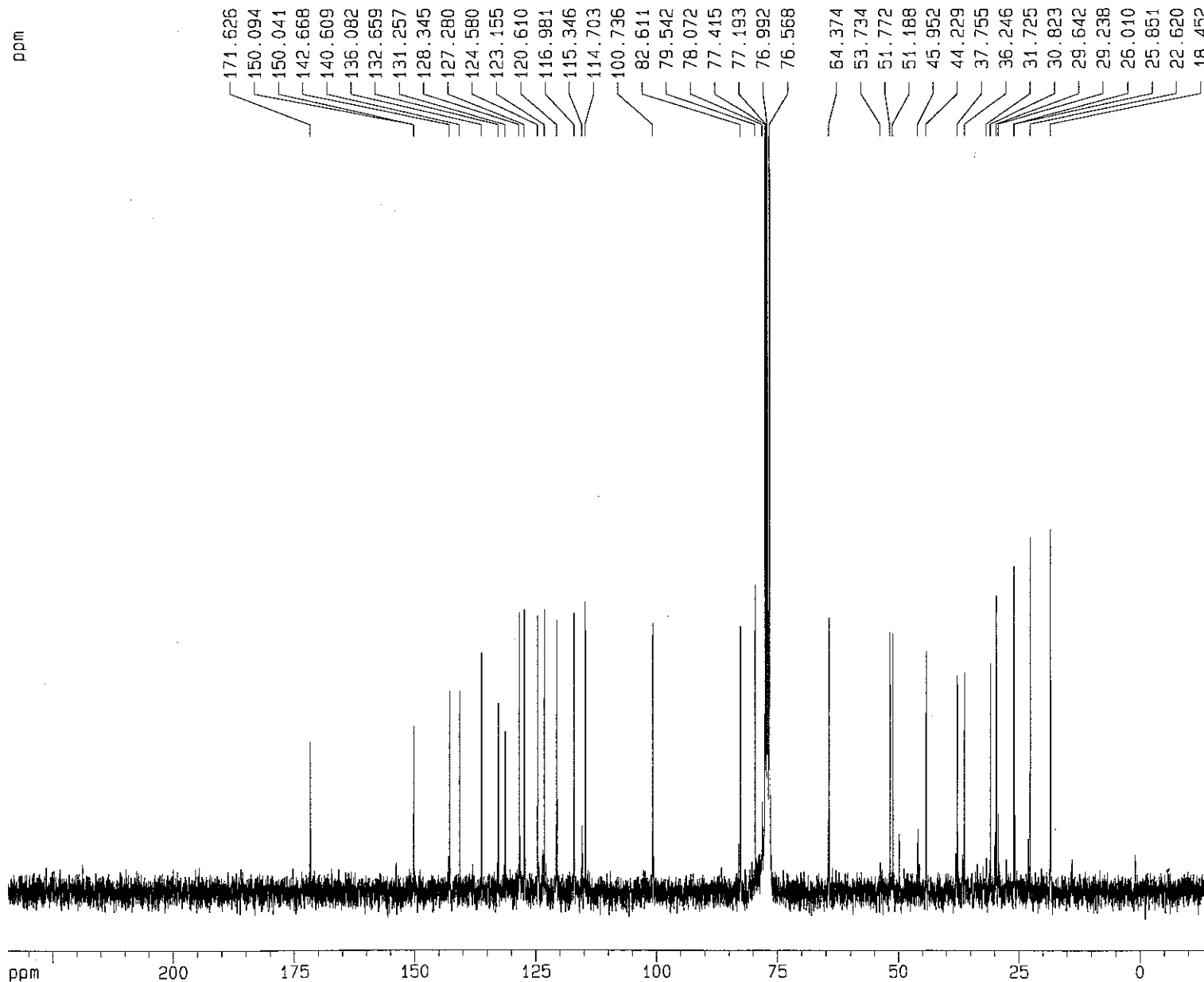
SI 32768  
SF 299.8700161 MHz  
WDW no  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

## 1D NMR plot parameters

CX 20.00 cm  
F1P 11.000 ppm  
F1 3298.57 Hz  
F2P -1.000 ppm  
F2 -299.87 Hz  
PPMCM 0.60000 ppm/cm  
HZCM 179.92201 Hz/cm



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## Current Data Parameters

NAME PL-Nov14-09  
 EXPNO 2  
 PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20091114  
 Time 19.05  
 INSTRUM spect  
 PROBHD 5 mm GNP 1H/1  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDC13  
 NS 15824  
 DS 4  
 SWH 18796.992 Hz  
 FIDRES 0.286819 Hz  
 AQ 1.7433076 sec  
 RG 2048  
 DW 26.600 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 2.0000000 sec  
 D11 0.0300000 sec  
 D12 0.00002000 sec

## ===== CHANNEL f1 =====

NUC1 13C  
 P1 5.40 usec  
 PL1 -6.00 dB  
 SFO1 75.4106357 MHz

## ===== CHANNEL f2 =====

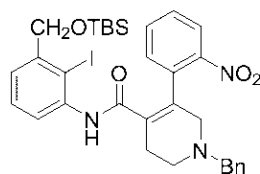
CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 115.00 usec  
 PL2 0.00 dB  
 PL12 20.00 dB  
 PL13 20.00 dB  
 SFO2 299.8711995 MHz

## F2 - Processing parameters

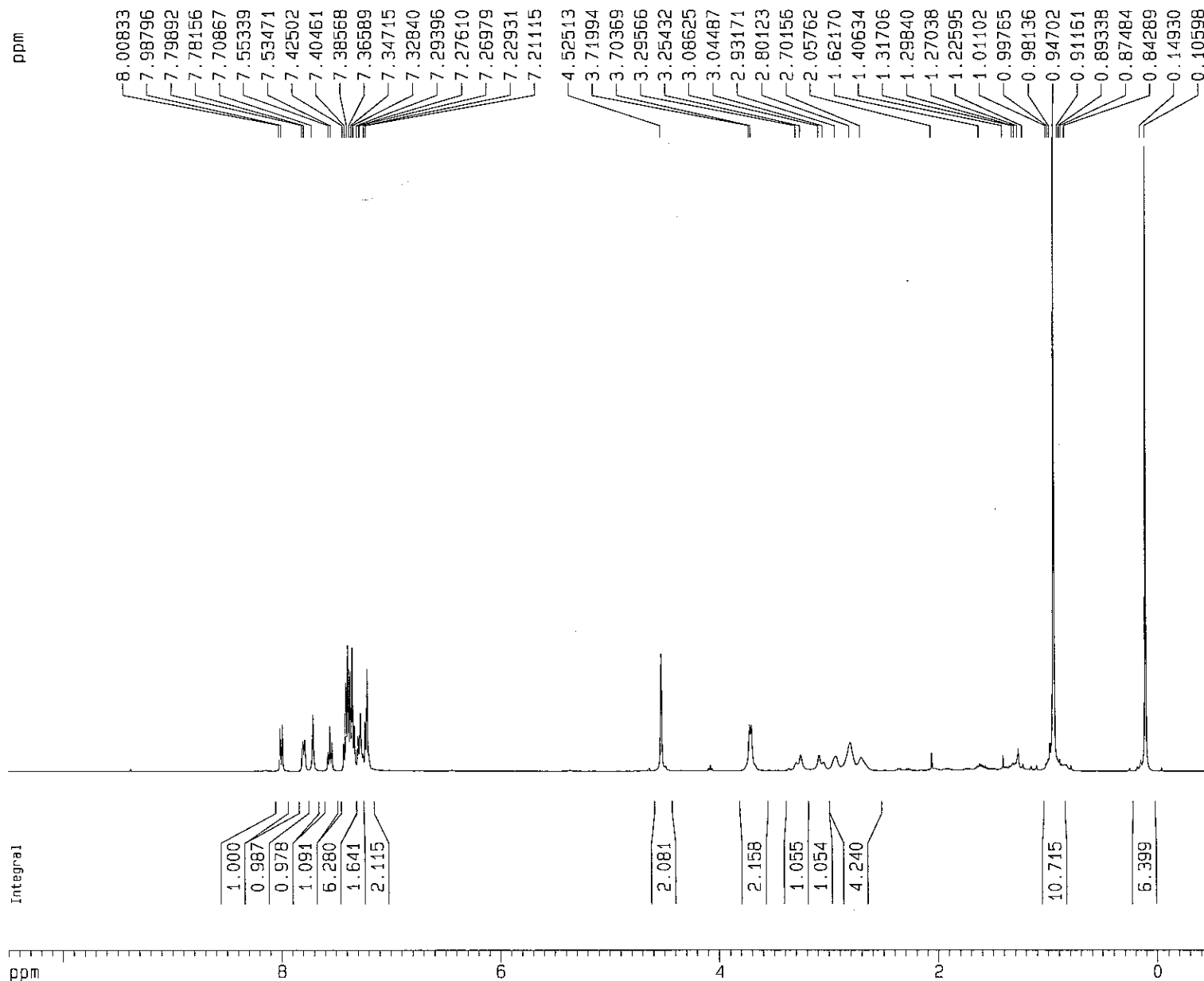
SI 32768  
 SF 75.4023752 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

## 1D NMR plot parameters

CX 20.00 cm  
 F1P 234.198 ppm  
 F1 17659.08 Hz  
 F2P -15.091 ppm  
 F2 -1137.91 Hz  
 PPMCM 12.46446 ppm/cm  
 HZCM 939.84961 Hz/cm



23b



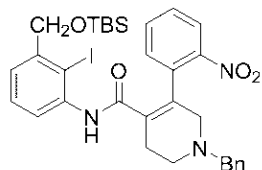
Current Data Parameters  
NAME Dec17-SJH  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20071217  
Time 13.04  
INSTRUM spect  
PROBHD 5 mm BBI 1H-B  
PULPROG zg30  
TD 49668  
SOLVENT CDCl3  
NS 14  
DS 2  
SWH 8278.146 Hz  
FIDRES 0.166670 Hz  
AQ 2.9999971 sec  
RG 128  
DW 60.400 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec

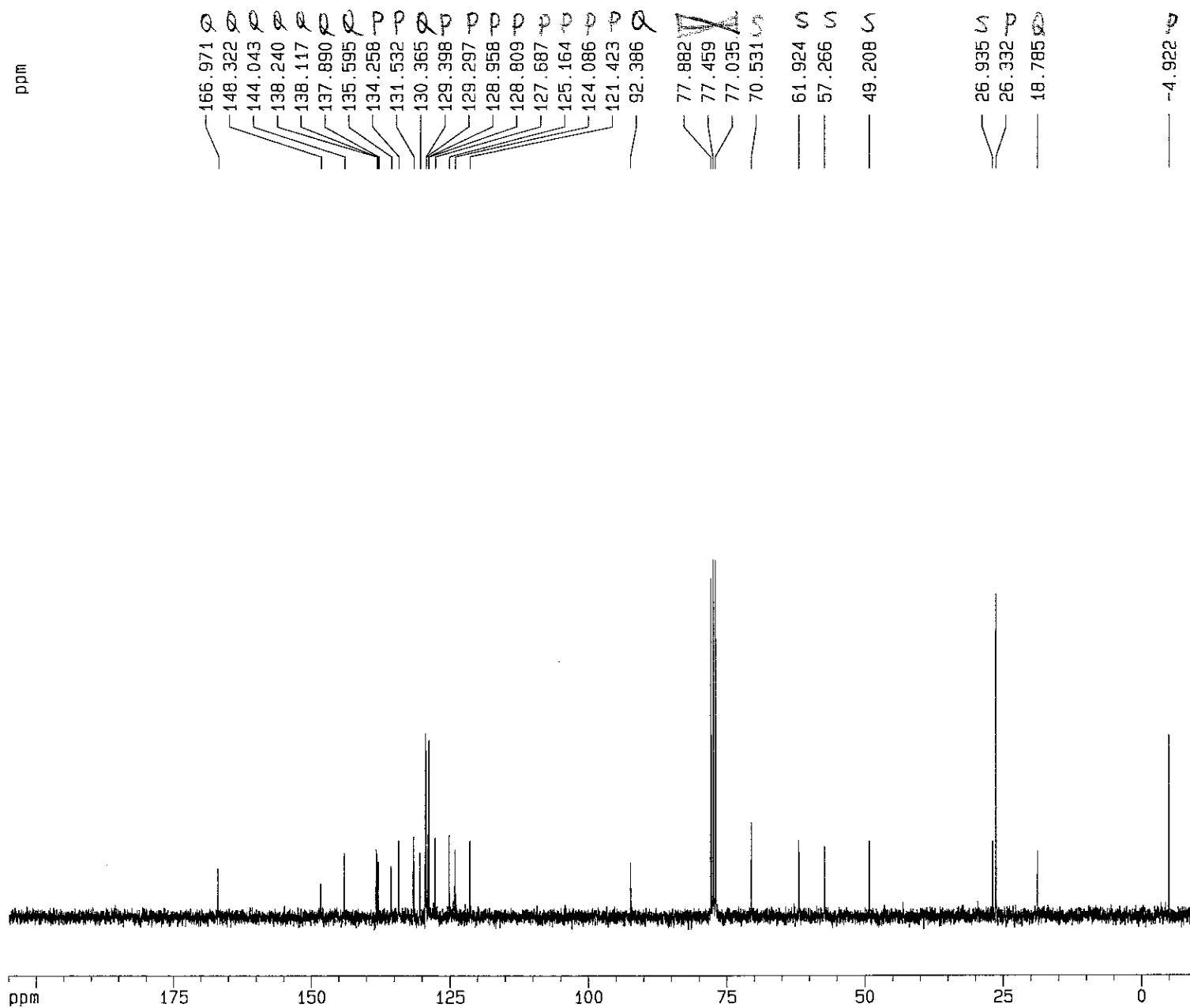
===== CHANNEL f1 =====  
NUC1 1H  
P1 6.45 usec  
PL1 0.00 dB  
SF01 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300054 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

1D NMR plot parameters  
CX 20.00 cm  
F1P 10.500 ppm  
F1 4201.37 Hz  
F2P -0.500 ppm  
F2 -200.07 Hz  
PPMCM 0.55000 ppm/cm  
HZCM 220.07150 Hz/cm



23b



Current Data Parameters  
NAME 1029-SJH  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20071029  
Time 13.34  
INSTRUM spect  
PROBHD 5 mm Multinu  
PULPROG zgpg30  
TD 65536  
SOLVENT CDC13  
NS 196  
DS 2  
SWH 18632.393 Hz  
FIDRES 0.287360 Hz  
AQ 1.7400308 sec  
RG 13004  
DW 26.550 usec  
DE 6.00 usec  
TE 300.0 K  
D1 2.00000000 sec  
d11 0.03000000 sec  
d12 0.00002000 sec

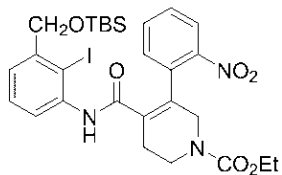
===== CHANNEL f1 =====  
NUC1 13C  
P1 11.80 usec  
PL1 0.00 dB  
SF01 75.4760200 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 110.00 usec  
PL2 0.00 dB  
PL12 17.50 dB  
PL13 17.50 dB  
SF02 300.1312005 MHz

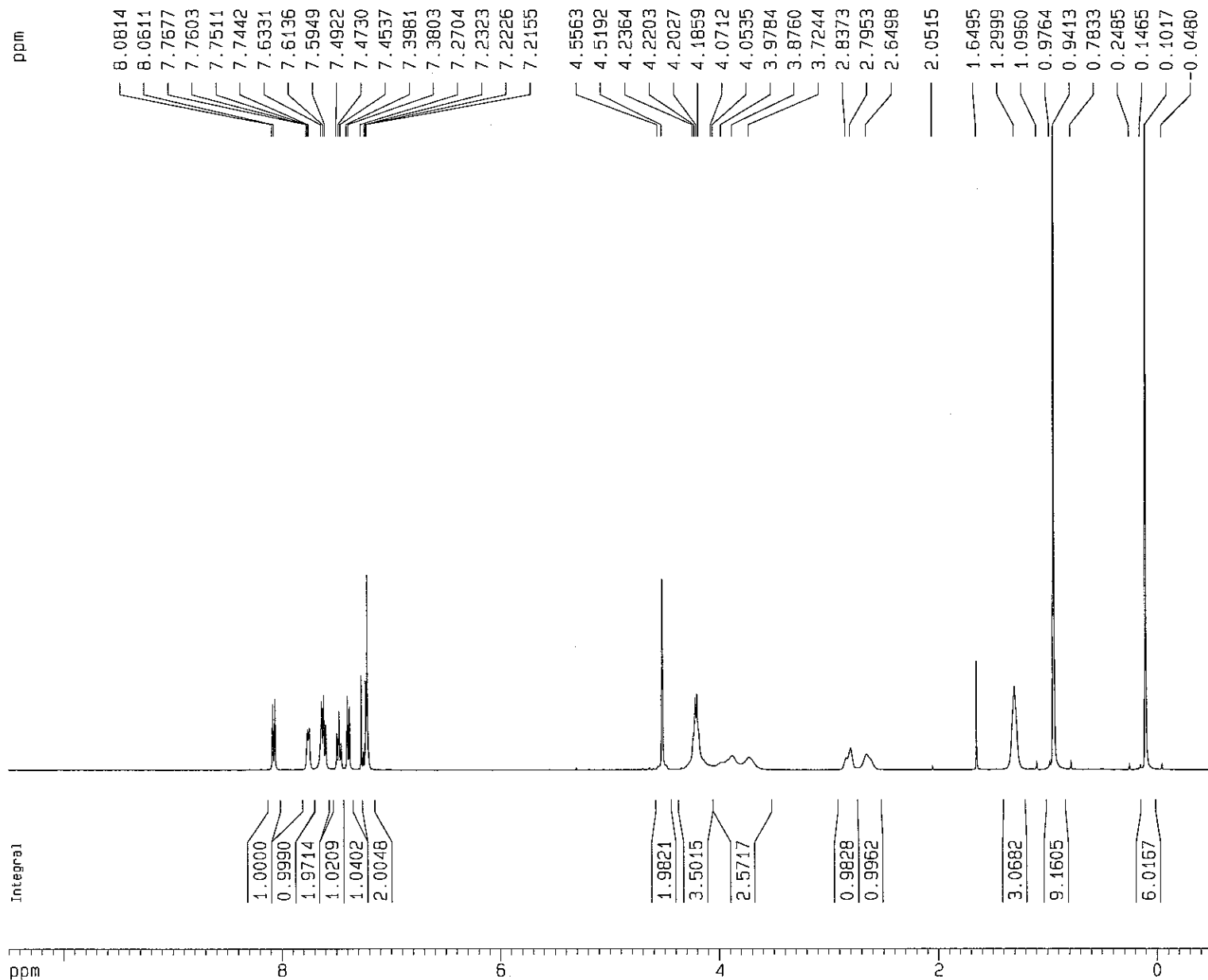
F2 - Processing parameters  
SI 32768  
SF 75.4677190 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

1D NMR plot parameters  
CX 20.00 cm  
F1P 205.000 ppm  
F1 15470.88 Hz  
F2P -10.000 ppm  
F2 -754.68 Hz  
PPMCM 10.75000 ppm/cm  
HZCM 811.27802 Hz/cm

2-H-V1-74



24b



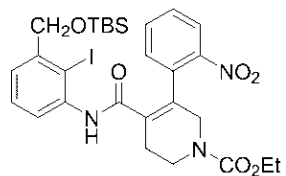
Current Data Parameters  
NAME Jan17-SJH  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20080117  
Time 14.21  
INSTRUM spect  
PROBHD 5 mm BBI 1H-B  
PULPROG zg30  
TD 49668  
SOLVENT CDC13  
NS 21  
DS 2  
SWH 8278.146 Hz  
FIDRES 0.166670 Hz  
AQ 2.9999971 sec  
RG 812.7  
DW 60.400 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec

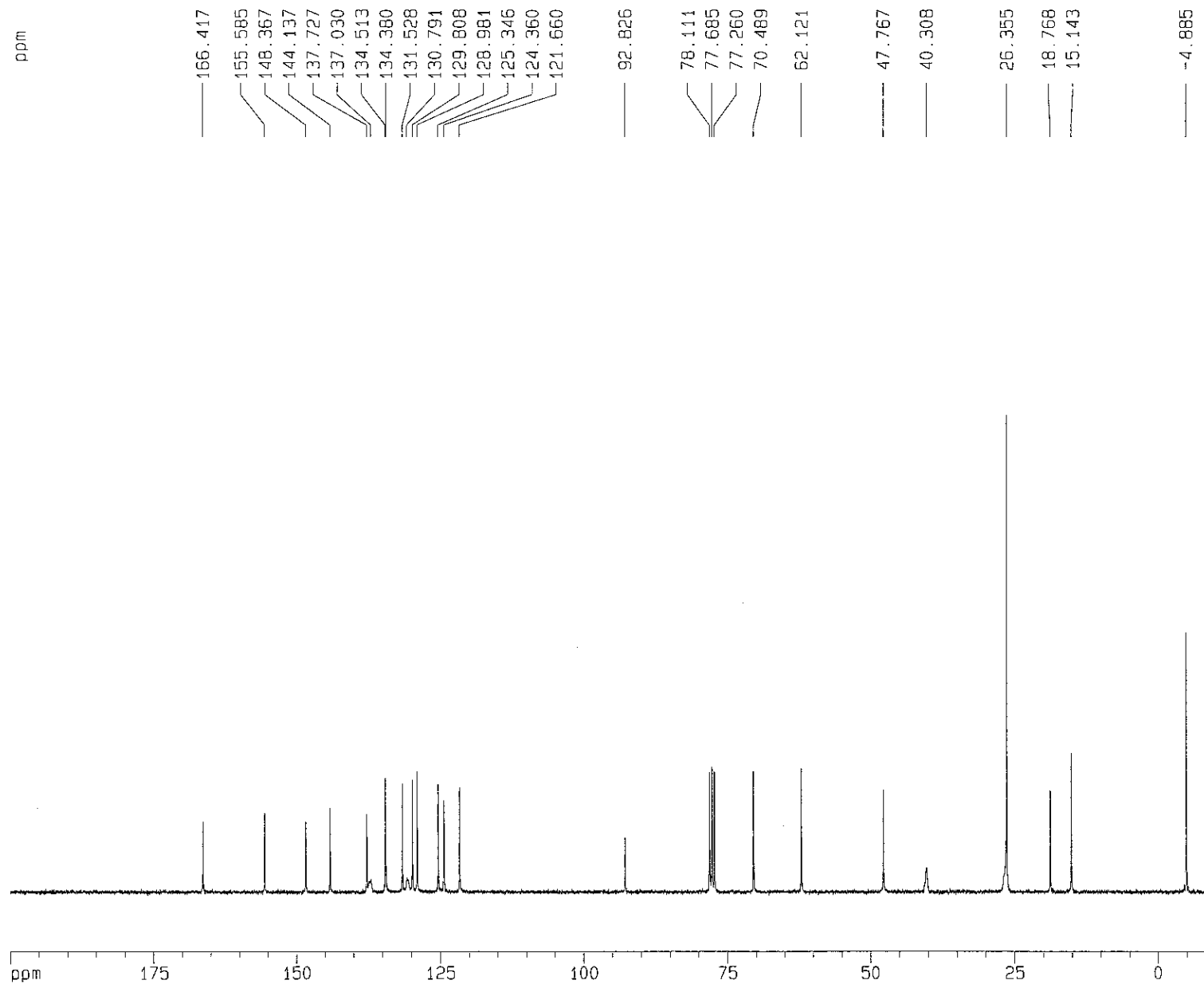
===== CHANNEL f1 =====  
NUC1 1H  
P1 6.45 usec  
PL1 0.00 dB  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300051 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

1D NMR plot parameters  
CX 20.00 cm  
F1P 10.500 ppm  
F1 4201.37 Hz  
F2P -0.500 ppm  
F2 -200.07 Hz  
PPMCM 0.55000 ppm/cm  
HZCM 220.07150 Hz/cm



24b



Current Data Parameters  
 NAME Jan17-SJH  
 EXPNO 4  
 PROCNO 1

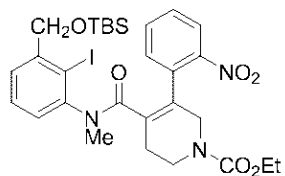
F2 - Acquisition Parameters  
 Date\_ 20080117  
 Time 19.45  
 INSTRUM spect  
 PROBHD 5 mm QNP 1H/1  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 512  
 DS 2  
 SWH 18796.992 Hz  
 FIDRES 0.286819 Hz  
 AQ 1.7433076 sec  
 RG 1024  
 DW 26.600 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 2.0000000 sec  
 D11 0.0300000 sec  
 D12 0.0000200 sec

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 5.40 usec  
 PL1 -6.00 dB  
 SF01 75.4106357 MHz

===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 115.00 usec  
 PL2 0.00 dB  
 PL12 20.00 dB  
 PL13 20.00 dB  
 SF02 299.8711995 MHz

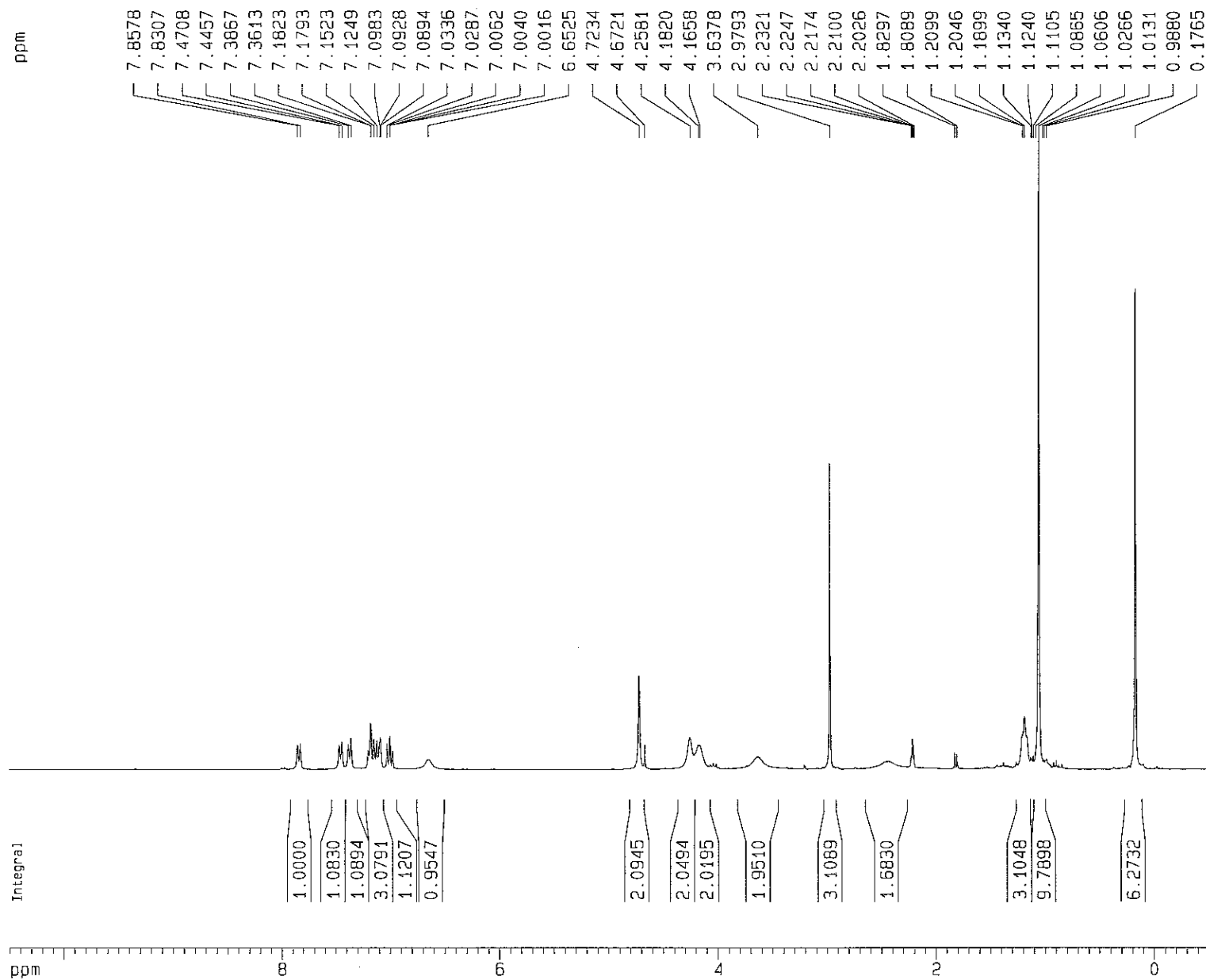
F2 - Processing parameters  
 SI 32768  
 SF 75.4023410 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 200.000 ppm  
 F1 15080.47 Hz  
 F2P -10.000 ppm  
 F2 -754.02 Hz  
 PPMCM 10.50000 ppm/cm  
 HZCM 791.72461 Hz/cm



in tol at 90 oC

25b



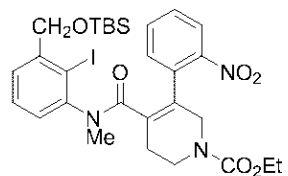
Current Data Parameters  
NAME 1031-SJH  
EXPNO 3  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20071031  
Time 12.31  
INSTRUM spect  
PROBHD 5 mm QNP 1H/1  
PULPROG zg30  
TD 37036  
SOLVENT Tol  
NS 10  
DS 2  
SWH 6172.839 Hz  
FIDRES 0.166671 Hz  
AQ 2.9999659 sec  
RG 161.3  
DW 81.000 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.0000000 sec

==== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 0.00 dB  
SFO1 299.8702999 MHz

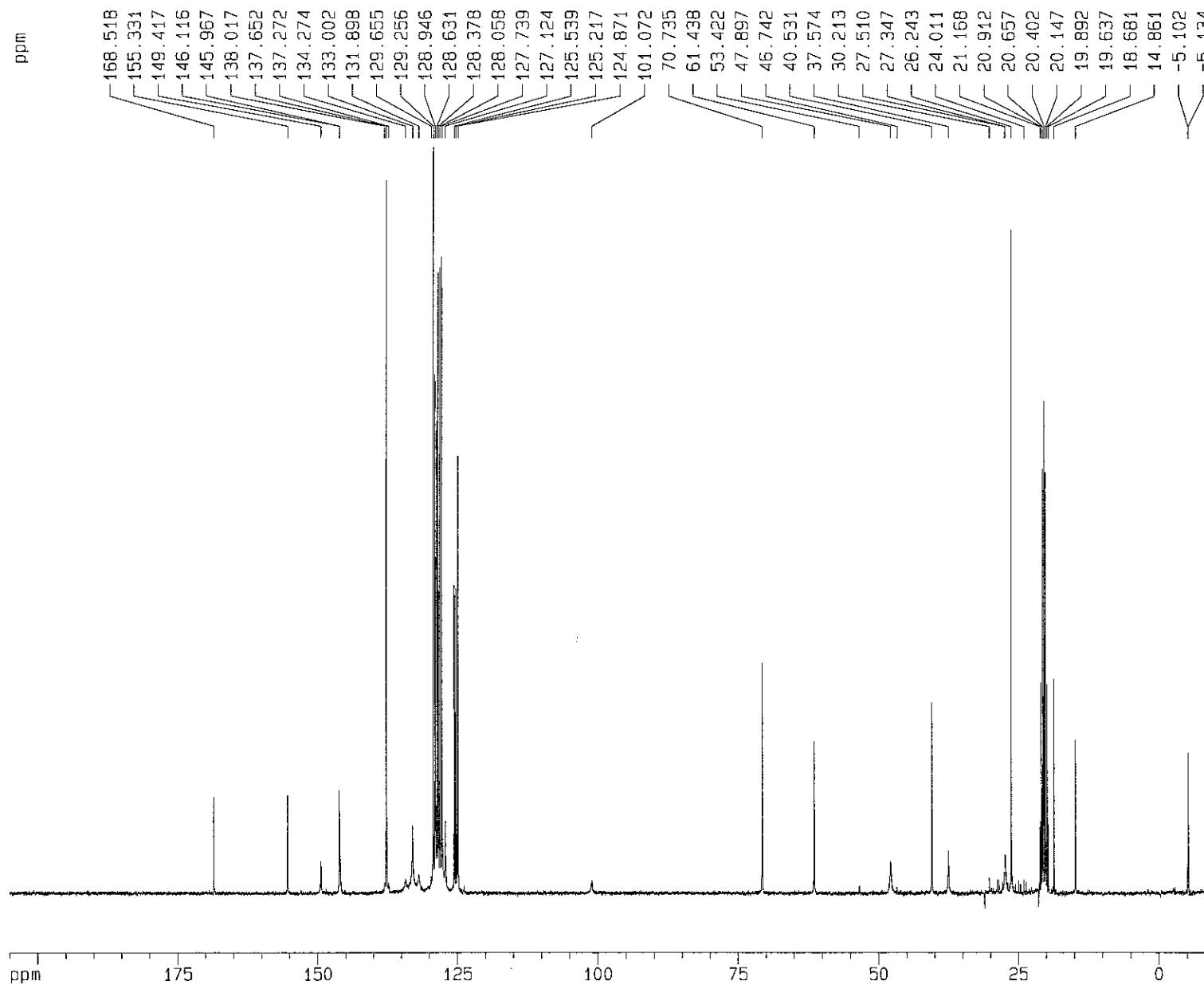
F2 - Processing parameters  
SI 32768  
SF 299.8684997 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

1D NMR plot parameters  
CX 20.00 cm  
F1P 10.500 ppm  
F1 3148.62 Hz  
F2P -0.500 ppm  
F2 -149.93 Hz  
PPMCM 0.55000 ppm/cm  
HZCM 164.92767 Hz/cm



in tol at 90 oC

25b



## Current Data Parameters

NAME 1101-SJH  
EXPNO 6  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20071101  
Time 19.28  
INSTRUM spect  
PROBHD 5 mm GNP 1H/1  
PULPROG zgpg30  
TD 65536  
SOLVENT Tol  
NS 10240  
DS 2  
SWH 18796.992 Hz  
FIDRES 0.286819 Hz  
AQ 1.7433076 sec  
RG 2048  
DW 26.600 usec  
DE 6.00 usec  
TE 300.0 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
D12 0.00002000 sec

## ===== CHANNEL f1 =====

NUC1 13C  
P1 5.40 usec  
PL1 -6.00 dB  
SF01 75.4106357 MHz

## ===== CHANNEL f2 =====

CPDPRG2 waltz16  
NUC2 1H  
PCPD2 115.00 usec  
PL2 0.00 dB  
PL12 20.00 dB  
PL13 20.00 dB  
SF02 299.8711995 MHz

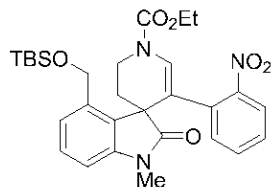
## F2 - Processing parameters

SI 32768  
SF 75.4019494 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

## 1D NMR plot parameters

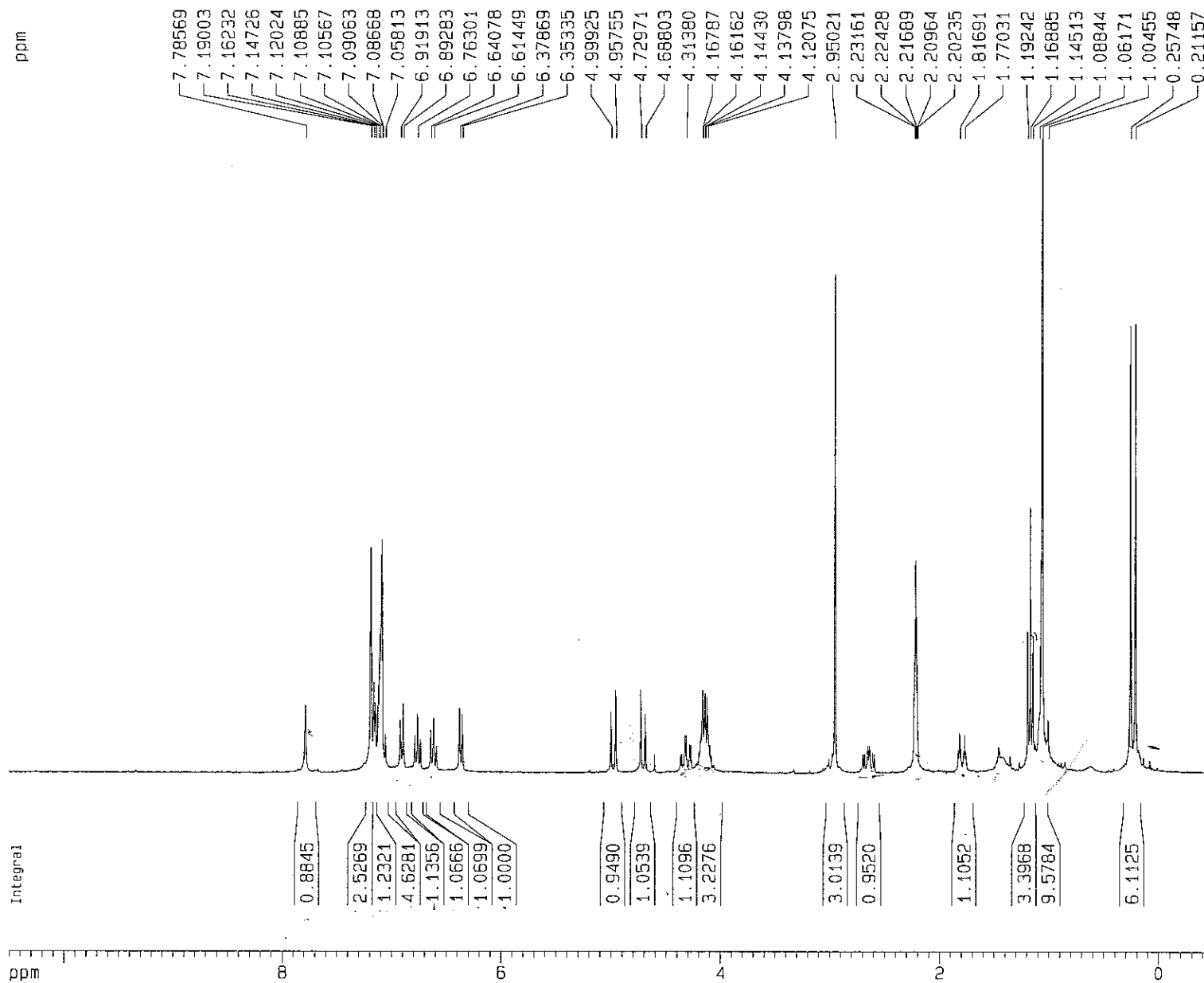
CX 20.00 cm  
F1P 205.000 ppm  
F1 15457.40 Hz  
F2P -10.000 ppm  
F2 -754.02 Hz  
PPMCM 10.75000 ppm/cm  
HZCM 810.57092 Hz/cm





in tol at 90 oC

26b



## Current Data Parameters

NAME 1101-SJH  
EXPNO 2  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20071101  
Time 13.32  
INSTRUM spect  
PROBHD 5 mm QNP 1H/1  
PULPROG zg30  
TD 37036  
SOLVENT Tol  
NS 16  
DS 2  
SWH 6172.839 Hz  
FIDRES 0.166671 Hz  
AQ 2.9999659 sec  
RG 812.7  
DW 81.000 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec

## ===== CHANNEL f1 =====

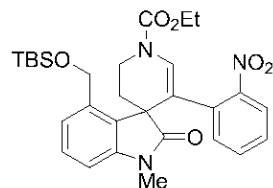
NUC1 1H  
P1 11.70 usec  
PL1 0.00 dB  
SF01 299.8702999 MHz

## F2 - Processing parameters

SI 32768  
SF 299.8684992 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

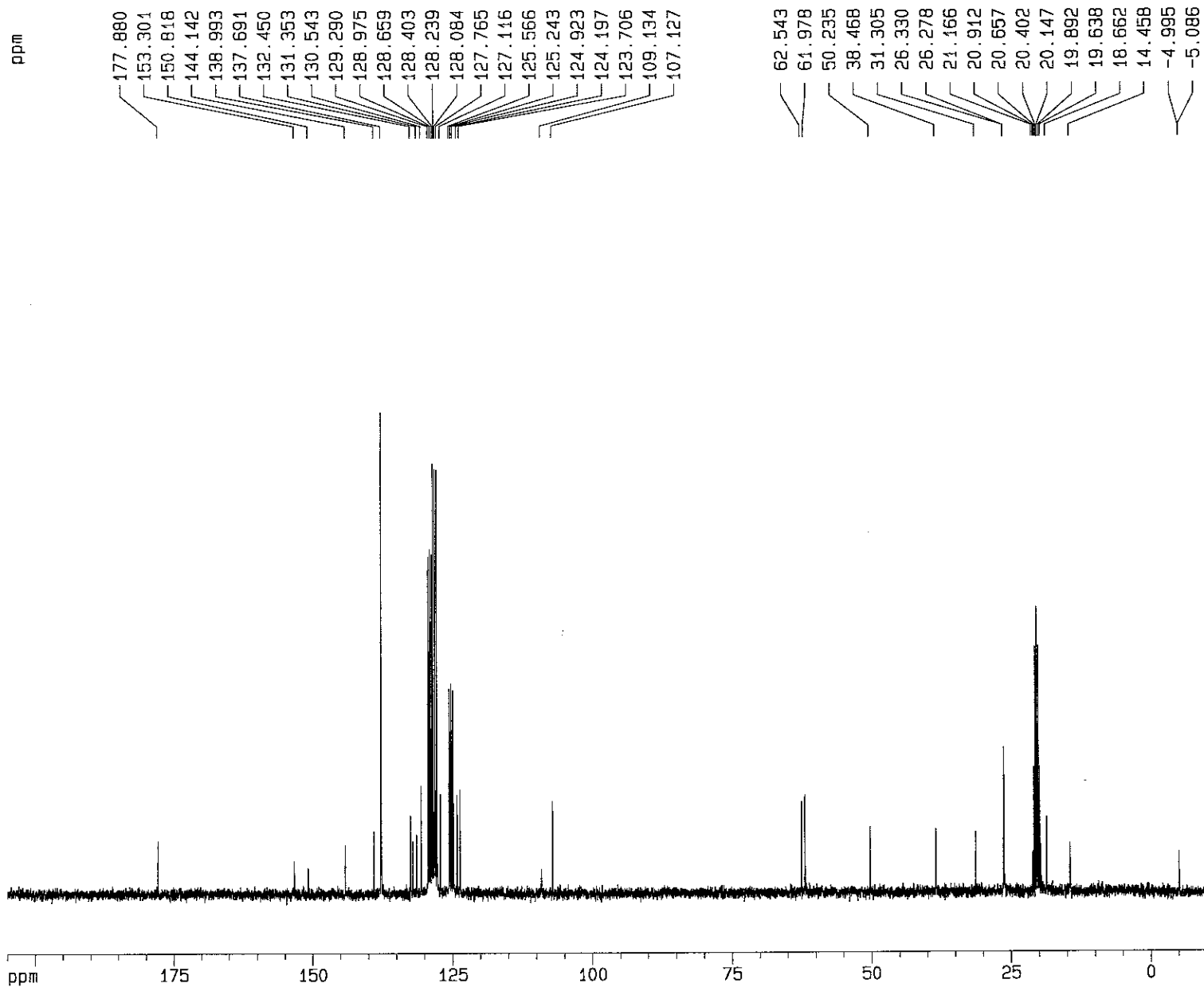
## 1D NMR plot parameters

CX 20.00 cm  
F1P 10.500 ppm  
F1 3148.62 Hz  
F2P -0.500 ppm  
F2 -149.93 Hz  
PPMCM 0.55000 ppm/cm  
HZCM 164.92767 Hz/cm



in tol at 90 oC 13C

26b



Current Data Parameters  
NAME 1102-SJH  
EXPNO 3  
PROCNO 1

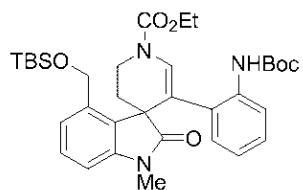
F2 - Acquisition Parameters  
Date\_ 20071102  
Time 14.59  
INSTRUM spect  
PROBHD 5 mm Multinu  
PULPROG zgpg30  
TD 65536  
SOLVENT Tol  
NS 545  
DS 2  
SWH 18832.393 Hz  
FIDRES 0.287360 Hz  
AQ 1.7400308 sec  
RG 11585.2  
DW 26.550 usec  
DE 6.00 usec  
TE 300.0 K  
D1 2.00000000 sec  
d11 0.03000000 sec  
d12 0.00002000 sec

===== CHANNEL f1 =====  
NUC1 13C  
P1 11.80 usec  
PL1 0.00 dB  
SFO1 75.4760200 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 110.00 usec  
PL2 0.00 dB  
PL12 17.50 dB  
PL13 17.50 dB  
SFO2 300.1312005 MHz

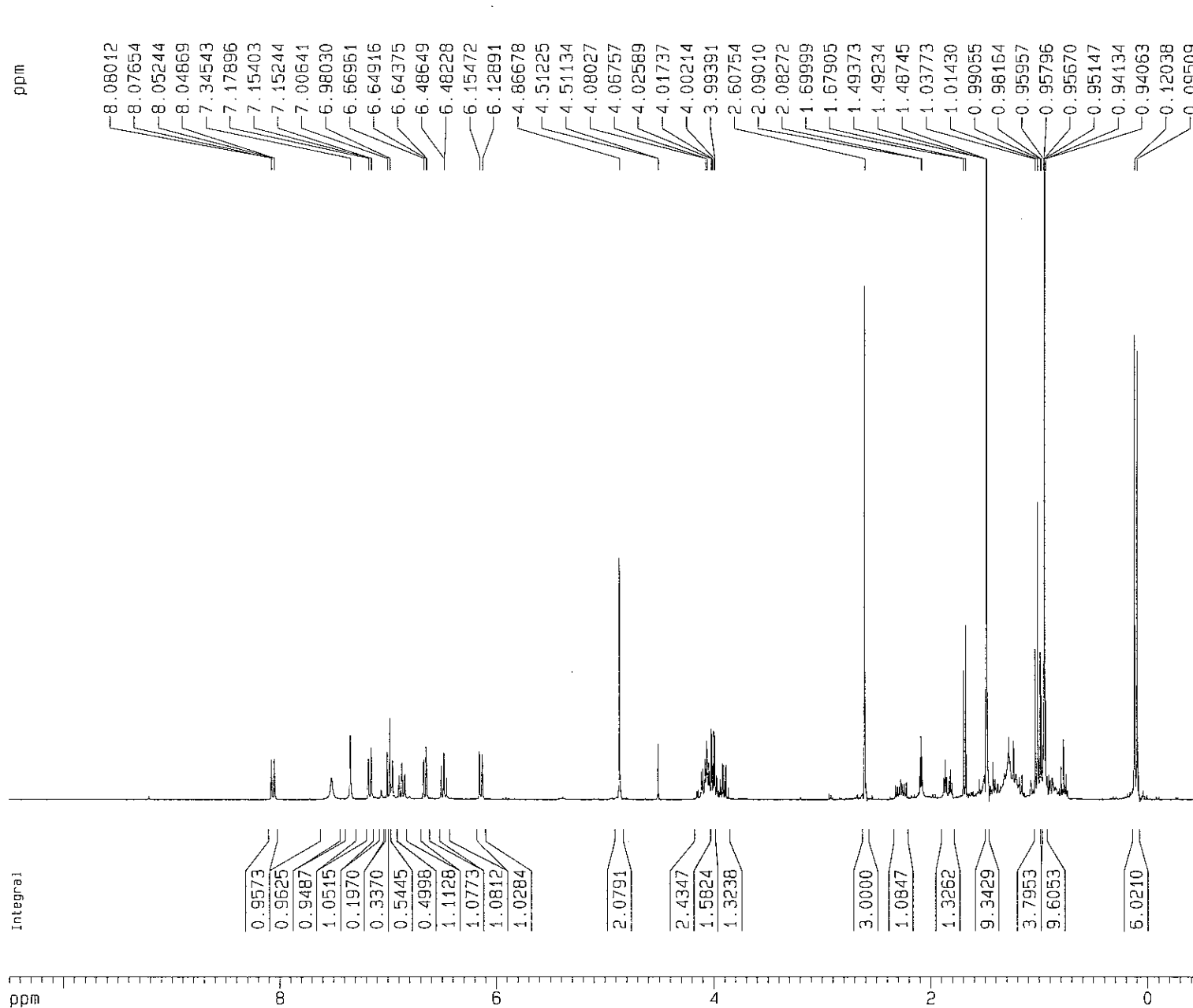
F2 - Processing parameters  
SI 32768  
SF 75.4673292 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

1D NMR plot parameters  
CX 20.00 cm  
F1P 205.000 ppm  
F1 15470.80 Hz  
F2P -10.000 ppm  
F2 -754.67 Hz  
PPMCM 10.75000 ppm/cm  
HZCM 811.27380 Hz/cm



in tol at 90 oC

29b



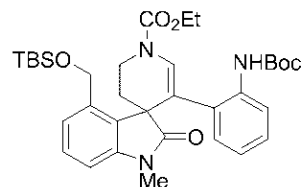
Current Data Parameters  
 NAME 1117-SJH  
 EXPNO 2  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20071117  
 Time 12.30  
 INSTRUM spect  
 PROBHD 5 mm QNP 1H/1  
 PULPROG zg30  
 TD 37036  
 SOLVENT Tol  
 NS 8  
 DS 2  
 SWH 6172.839 Hz  
 FIDRES 0.166671 Hz  
 AQ 2.9999659 sec  
 RG 228.1  
 DW 81.000 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 1.00000000 sec

----- CHANNEL f1 -----  
 NUC1 1H  
 P1 11.70 usec  
 PL1 0.00 dB  
 SF01 299.8697001 MHz

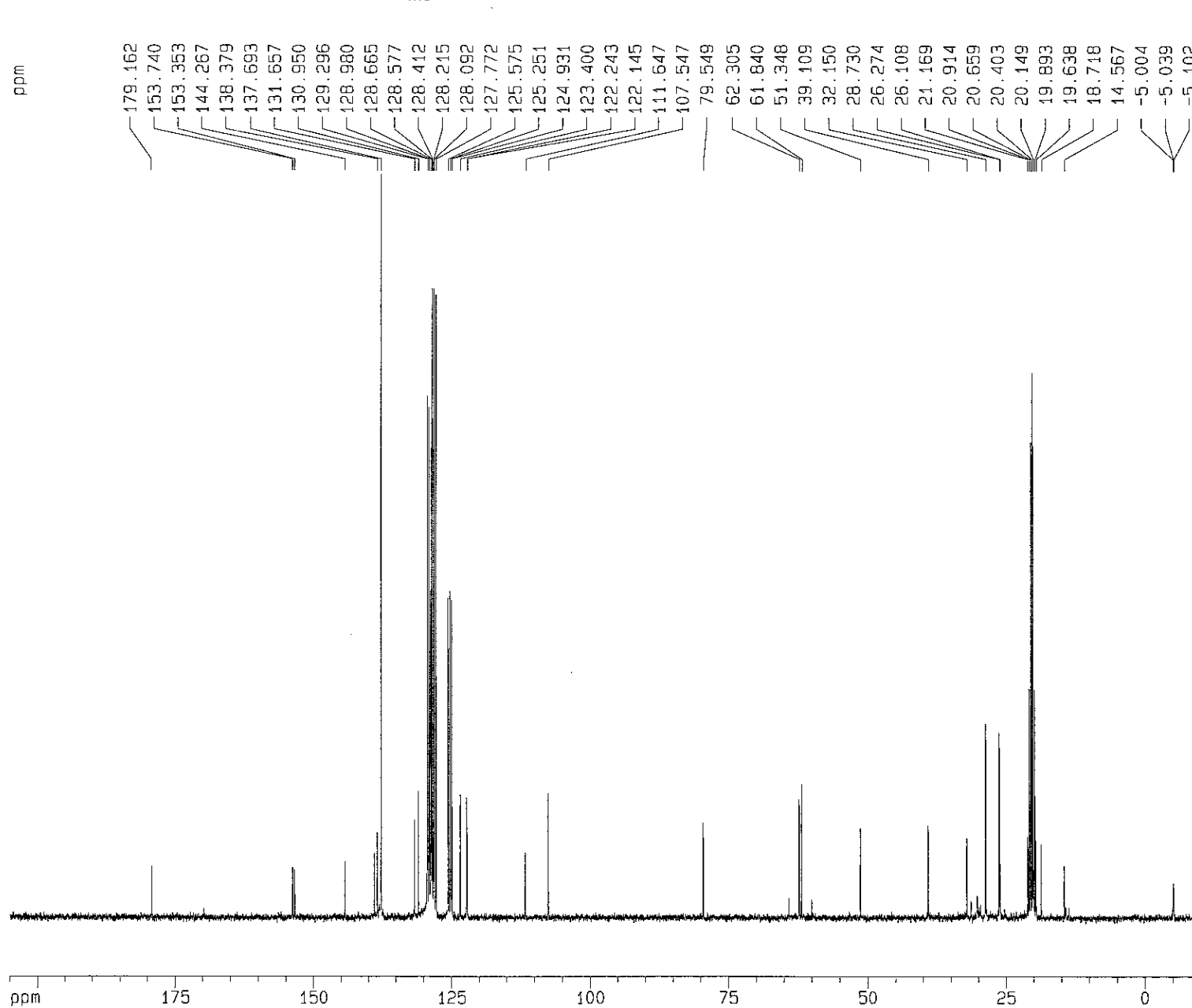
F2 - Processing parameters  
 SI 32768  
 SF 299.8685375 MHz  
 WDW no  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 10.500 ppm  
 F1 3148.62 Hz  
 F2P -0.500 ppm  
 F2 -149.93 Hz  
 PPMCM 0.55000 ppm/cm  
 HZCM 164.92769 Hz/cm



in tol at 90 oC 13C

29b



Current Data Parameters  
NAME 1117-SJH  
EXPNO 3  
PROCNO 1

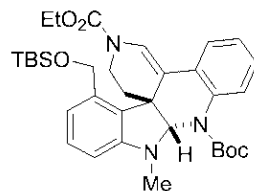
F2 - Acquisition Parameters  
Date\_ 20071117  
Time 12.37  
INSTRUM spect  
PROBHD 5 mm GNP 1H/1  
PULPROG zgpg30  
TD 65536  
SOLVENT Tol  
NS 3072  
DS 2  
SWH 18796.992 Hz  
FIDRES 0.286819 Hz  
AQ 1.7433076 sec  
RG 1448.2  
DW 26.600 usec  
DE 6.00 usec  
TE 300.0 K  
D1 2.0000000 sec  
D11 0.0300000 sec  
D12 0.0000200 sec

===== CHANNEL f1 =====  
NUC1 13C  
P1 5.40 usec  
PL1 -6.00 dB  
SF01 75.4106357 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 115.00 usec  
PL2 0.00 dB  
PL12 20.00 dB  
PL13 20.00 dB  
SF02 299.8711995 MHz

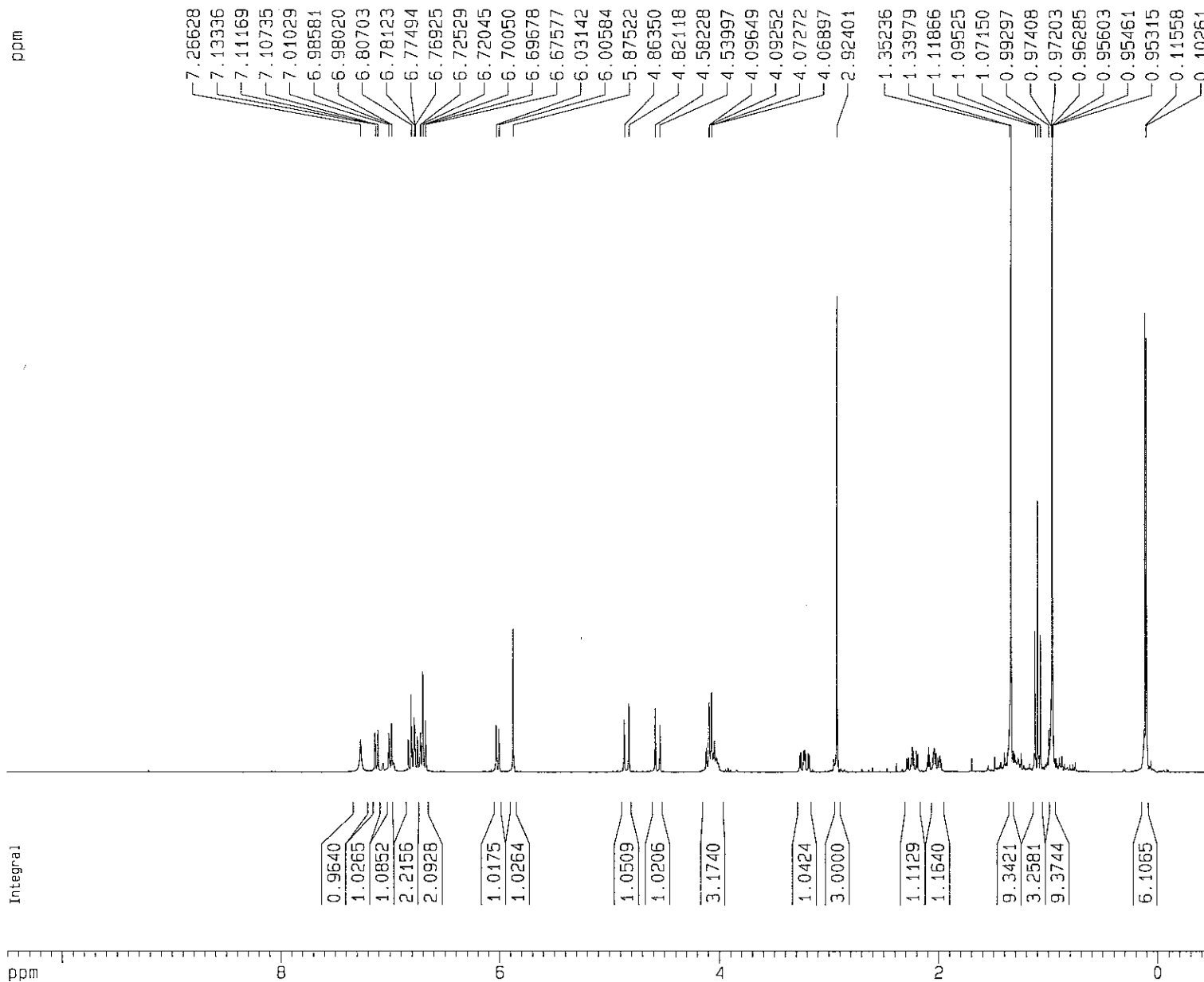
F2 - Processing parameters  
SI 32768  
SF 75.4019523 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

1D NMR plot parameters  
CX 20.00 cm  
F1P 205.000 ppm  
F1 15457.40 Hz  
F2P -10.000 ppm  
F2 -754.02 Hz  
PPMCM 10.75000 ppm/cm  
HZCM 810.57098 Hz/cm



in tol at 90 oC

30b



## Current Data Parameters

NAME 1123-SJH  
EXPNO 2  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20071124  
Time 20.49  
INSTRUM spect  
PROBHD 5 mm QNP 1H/1  
PULPROG zg30  
TD 37036  
SOLVENT Tol  
NS 8  
DS 2  
SWH 6172.839 Hz  
FIDRES 0.166671 Hz  
AQ 2.9999659 sec  
RG 128  
DW 81.000 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec

## ===== CHANNEL f1 =====

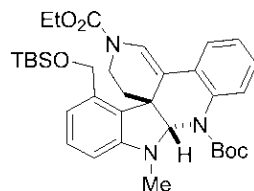
NUC1 1H  
P1 11.70 usec  
PL1 0.00 dB  
SFO1 299.8700000 MHz

## F2 - Processing parameters

SI 32768  
SF 299.8685371 MHz  
WDW no  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

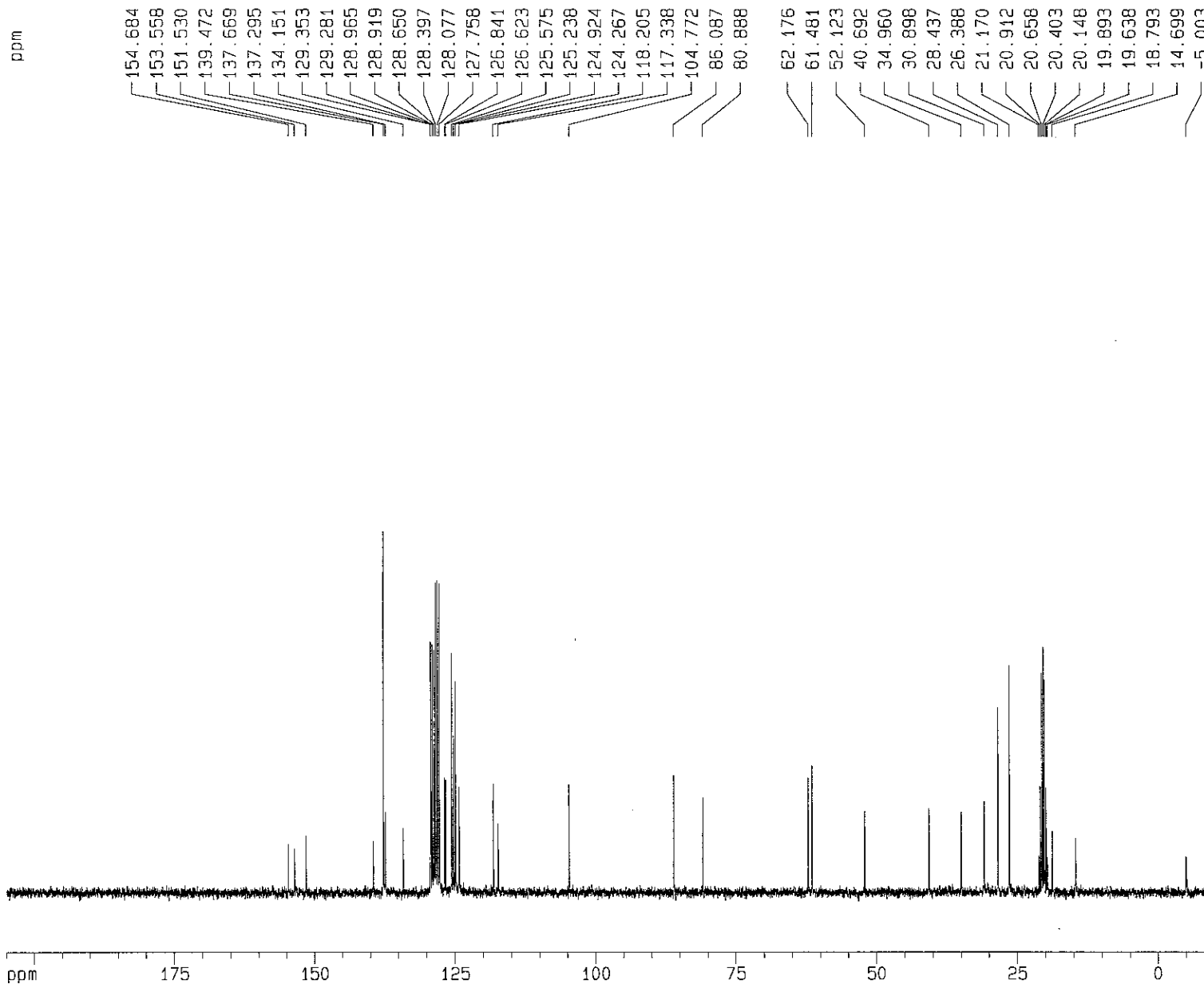
## 1D NMR plot parameters

CX 20.00 cm  
F1P 10.500 ppm  
F1 3148.62 Hz  
F2P -0.500 ppm  
F2 -149.93 Hz  
PPMCM 0.55000 ppm/cm  
HZCM 164.92769 Hz/cm



in tol at 90 oC 13C

30b



Current Data Parameters  
NAME 1123-SJH  
EXPNO 3  
PROCNO 1

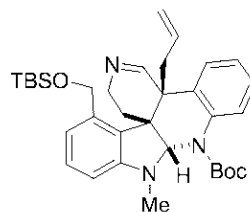
F2 - Acquisition Parameters  
Date\_ 20071124  
Time 21.01  
INSTRUM spect  
PROBHD 5 mm GNP 1H/1  
PULPROG zgpg30  
TD 65536  
SOLVENT Tol  
NS 344  
DS 2  
SWH 18796.992 Hz  
FIDRES 0.286819 Hz  
AQ 1.7433076 sec  
RG 2048  
DW 26.600 usec  
DE 6.00 usec  
TE 300.0 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
D12 0.00002000 sec

===== CHANNEL f1 =====  
NUC1 13C  
P1 5.40 usec  
PL1 -6.00 dB  
SF01 75.4106357 MHz

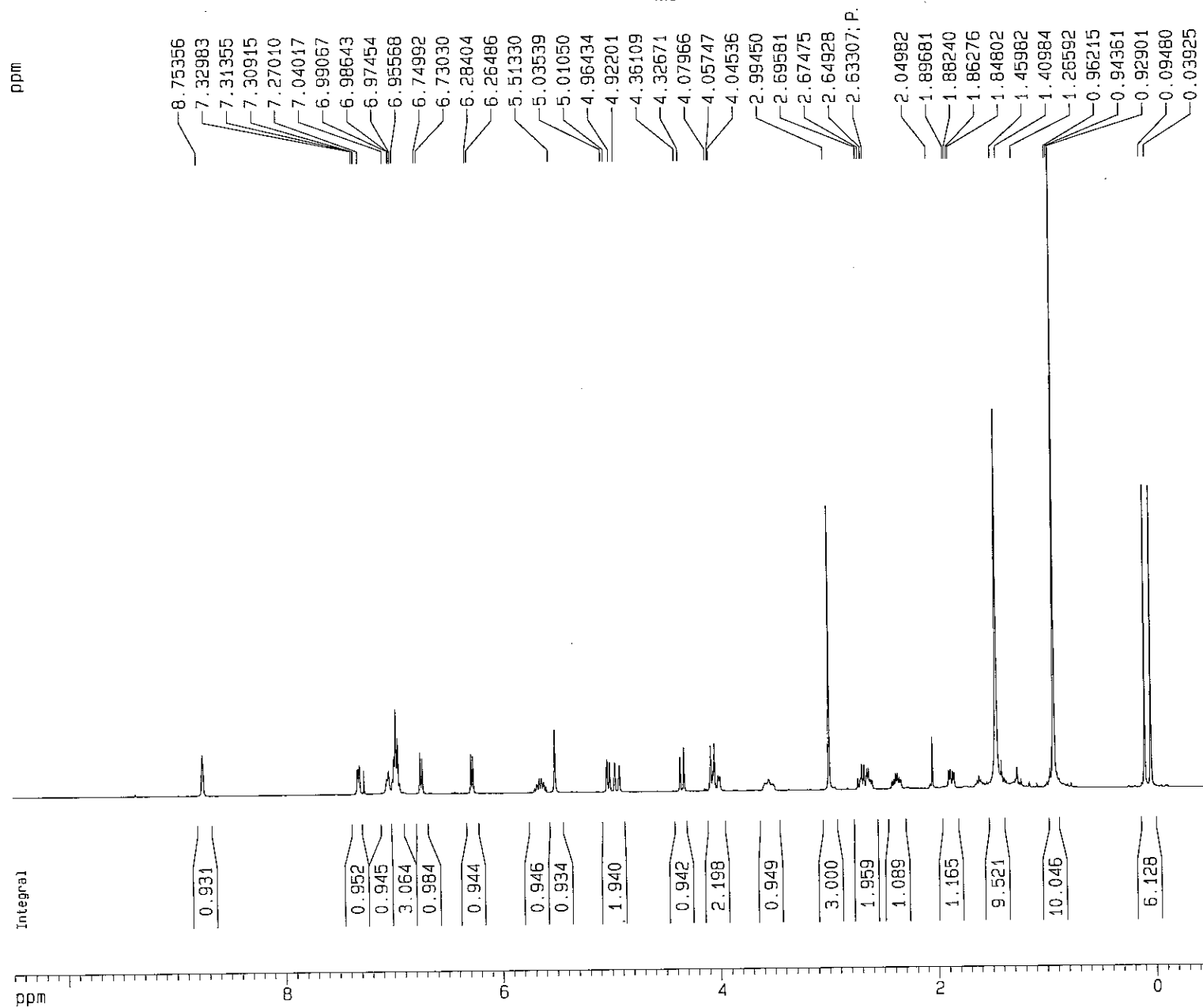
===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 115.00 usec  
PL2 0.00 dB  
PL12 20.00 dB  
PL13 20.00 dB  
SF02 299.8711995 MHz

F2 - Processing parameters  
SI 32768  
SF 75.4019540 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

1D NMR plot parameters  
CX 20.00 cm  
F1P 205.000 ppm  
F1 15457.40 Hz  
F2P -10.000 ppm  
F2 -754.02 Hz  
PPMCM 10.75000 ppm/cm  
HZCM 810.57098 Hz/cm



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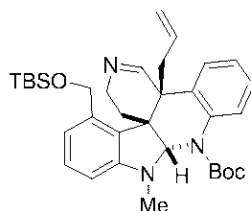
Current Data Parameters  
 NAME Dec20-SJH  
 EXPNO 2  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20071220  
 Time 19.22  
 INSTRUM spect  
 PROBHD 5 mm BBI 1H-B  
 PULPROG zg  
 TD 49668  
 SOLVENT CDC13  
 NS 1  
 DS 0  
 SWH 8278.146 Hz  
 FIDRES 0.166670 Hz  
 AQ 2.9999971 sec  
 RG 20.2  
 DW 60.400 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 1.00000000 sec

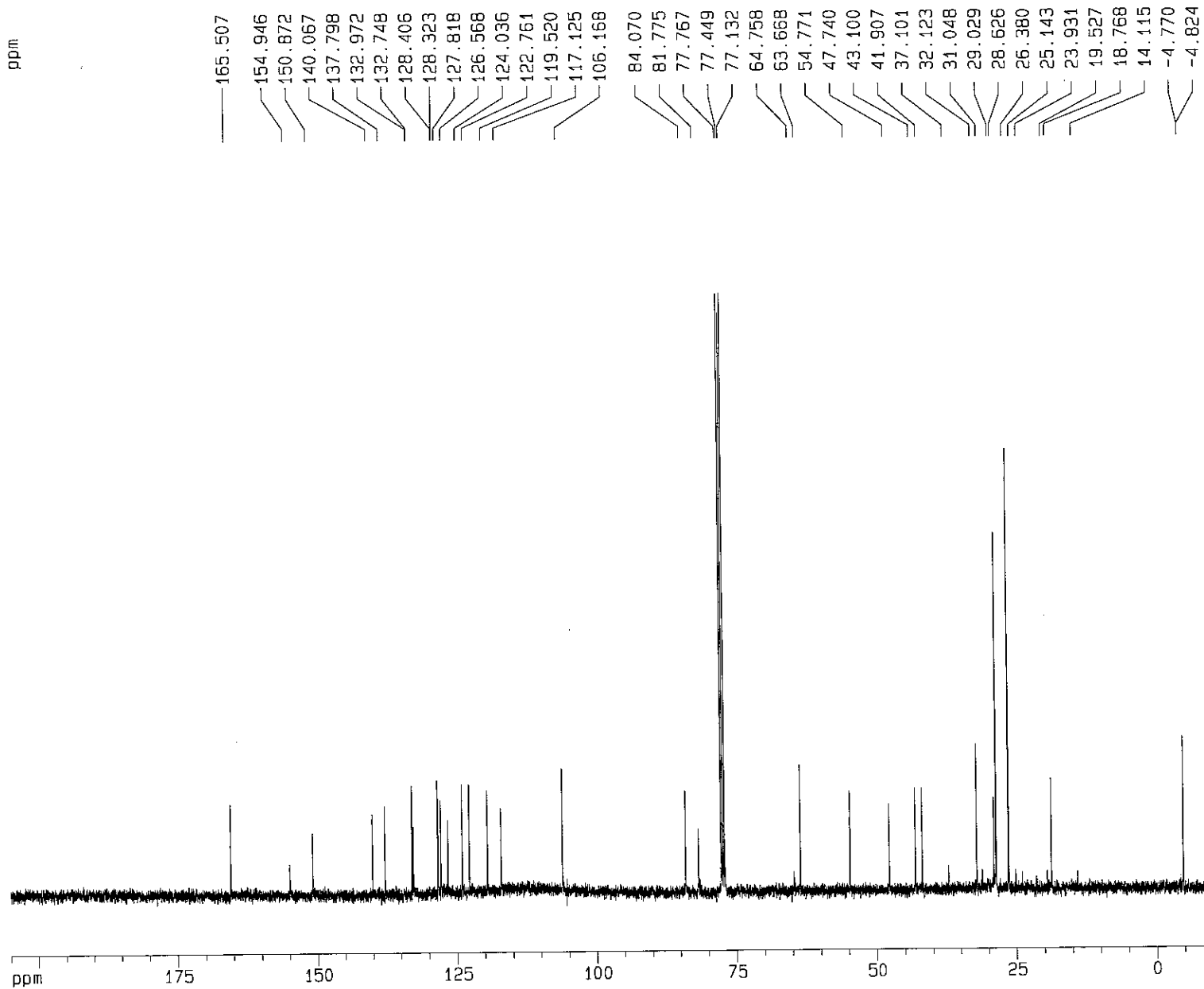
===== CHANNEL f1 =====  
 NUC1 1H  
 P1 7.53 usec  
 PL1 0.00 dB  
 SFO1 400.1324710 MHz

F2 - Processing parameters  
 SI 32768  
 SF 400.1300054 MHz  
 WDW no  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 10.500 ppm  
 F1 4201.37 Hz  
 F2P -0.500 ppm  
 F2 -200.07 Hz  
 PPMCM 0.55000 ppm/cm  
 HZCM 220.07150 Hz/cm



32



## Current Data Parameters

NAME Dec20-SJH  
EXPNO 3  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20071220  
Time 21.17  
INSTRUM spect  
PROBHD 5 mm BBI 1H-B  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 1536  
DS 2  
SWH 25125.629 Hz  
FIDRES 0.383387 Hz  
AQ 1.3042164 sec  
RG 8192  
DW 19.900 usec  
DE 6.00 usec  
TE 300.0 K  
D1 2.0000000 sec  
d11 0.0300000 sec  
d12 0.00002000 sec

## ===== CHANNEL f1 =====

NUC1 13C  
P1 16.35 usec  
PL1 -6.00 dB  
SF01 100.6237959 MHz

## ===== CHANNEL f2 =====

CPDPRG2 waltz16  
NUC2 1H  
PCPD2 114.00 usec  
PL2 0.00 dB  
PL12 24.00 dB  
PL13 24.00 dB  
SF02 400.1316005 MHz

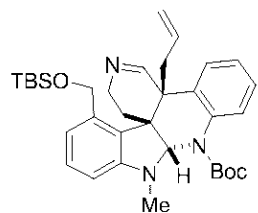
## F2 - Processing parameters

SI 32768  
SF 100.6127290 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

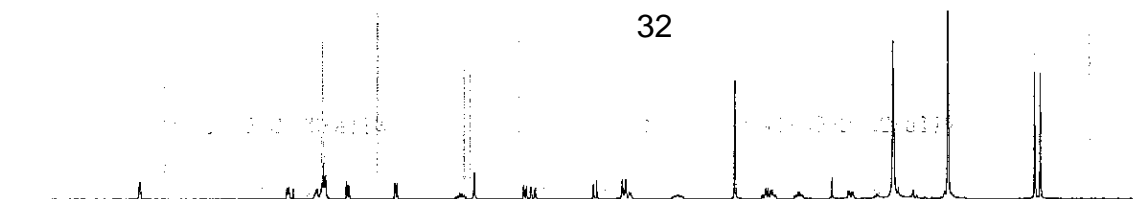
## 1D NMR plot parameters

CX 20.00 cm  
F1P 205.000 ppm  
F1 20625.61 Hz  
F2P -10.000 ppm  
F2 -1006.13 Hz  
PPMCM 10.75000 ppm/cm  
HZCM 1081.58691 Hz/cm

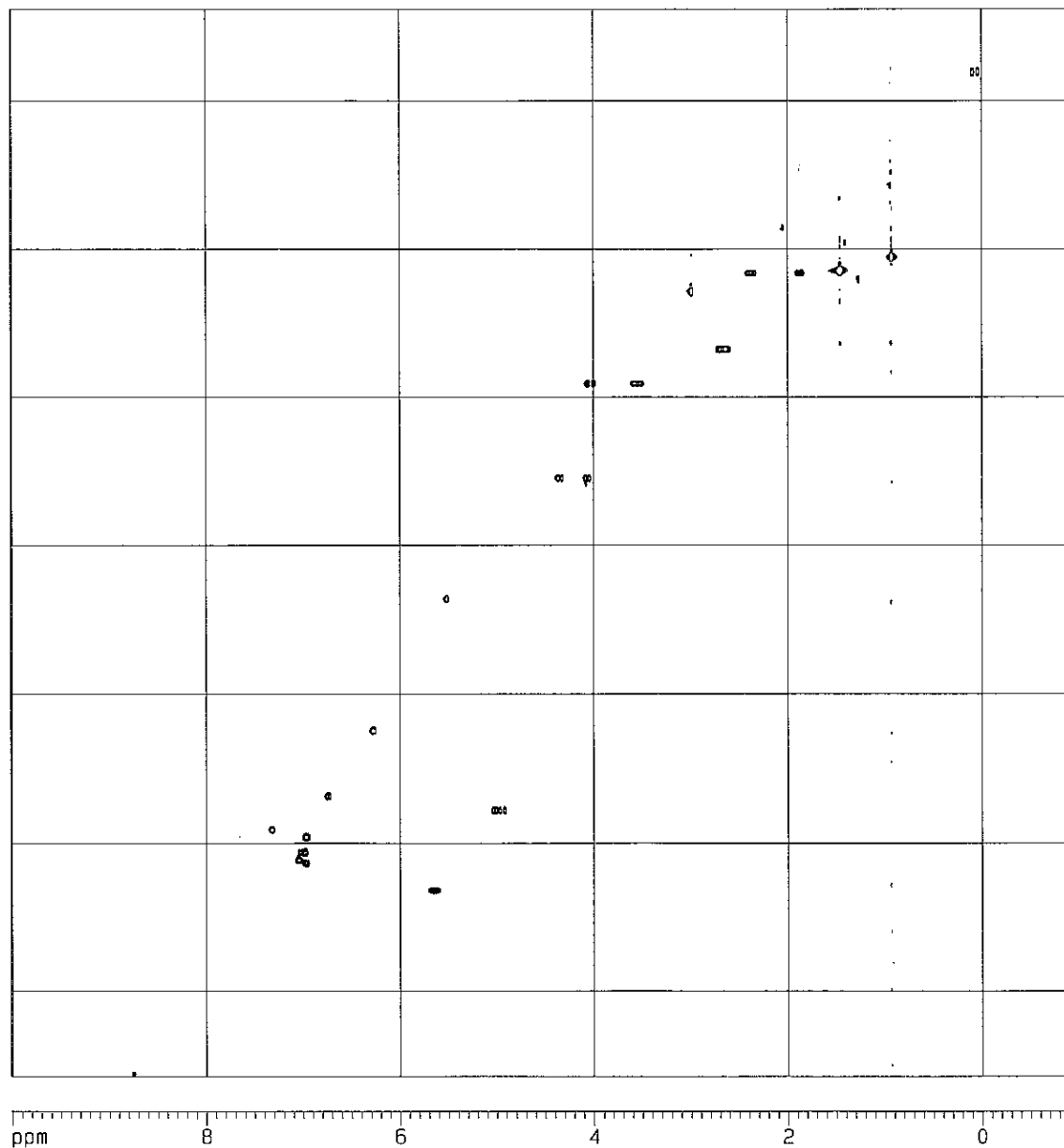




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HMQC



Current Data Parameters  
 NAME Dec20-5JH  
 EXPNO 5  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20071220  
 Time 21.49  
 INSTRUM spect  
 PROBHD 5 mm BBI 1H-0  
 PULPROG inv4gptp  
 TD 2048  
 SOLVENT CDCl3  
 NS 8  
 DS 80  
 SWH 4401.408 Hz  
 FIDRES 2.149125 Hz  
 AQ 0.2322028 sec  
 RG 16384  
 DK 113.800 usec  
 DE 6.00 usec  
 TE 300.0 K  
 CNSTE 145.0000000  
 d0 0.0000000 sec  
 d1 1.5951298 sec  
 d2 0.00344808 sec  
 d4 0.00172404 sec  
 d11 0.03000000 sec  
 d13 0.00000000 sec  
 d16 0.00020000 sec  
 d20 0.00052414 sec  
 d21 0.00224428 sec  
 INO 0.00001300 sec

\*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
 NUC1 1H  
 P1 7.33 usec  
 p2 15.05 usec  
 PL1 0.00 dB  
 SFO1 400.1518130 MHz

\*\*\*\*\* CHANNEL f2 \*\*\*\*\*  
 CPDPRG2 srrp  
 NUC2 13C  
 P2 17.00 usec  
 p4 34.00 usec  
 PCDP2 64.00 usec  
 PL2 -6.00 dB  
 PL12 6.30 dB  
 SFO2 100.6202532 MHz

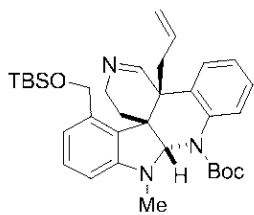
\*\*\*\*\* GRADIENT CHANNEL \*\*\*\*\*  
 GRAM1 SINE 100  
 GRAM2 SINE 100  
 GRAM3 SINE 100  
 GPX1 17.00 %  
 GPX2 20.00 %  
 GPX3 25.00 %  
 GPY1 17.00 %  
 GPY2 20.00 %  
 GPY3 25.00 %  
 GPZ1 17.00 %  
 GPZ2 20.00 %  
 GPZ3 25.00 %  
 P16 1000.00 usec

F1 - Acquisition parameters  
 ND0 4  
 YD 256  
 SFO1 100.6202 MHz  
 FIDRES 70.765356 Hz  
 SK 160.043 ppb

F2 - Processing parameters  
 SI 2048  
 SF 400.1500054 MHz  
 KW 65INE  
 SSB 2  
 LB 0.00 Hz  
 GB 0  
 PC 1.40

F1 - Processing parameters  
 SI 1024  
 MC2 1PPH  
 SF 100.6127560 MHz  
 KW 65INE  
 SSB 2  
 LB 0.00 Hz  
 GE 0

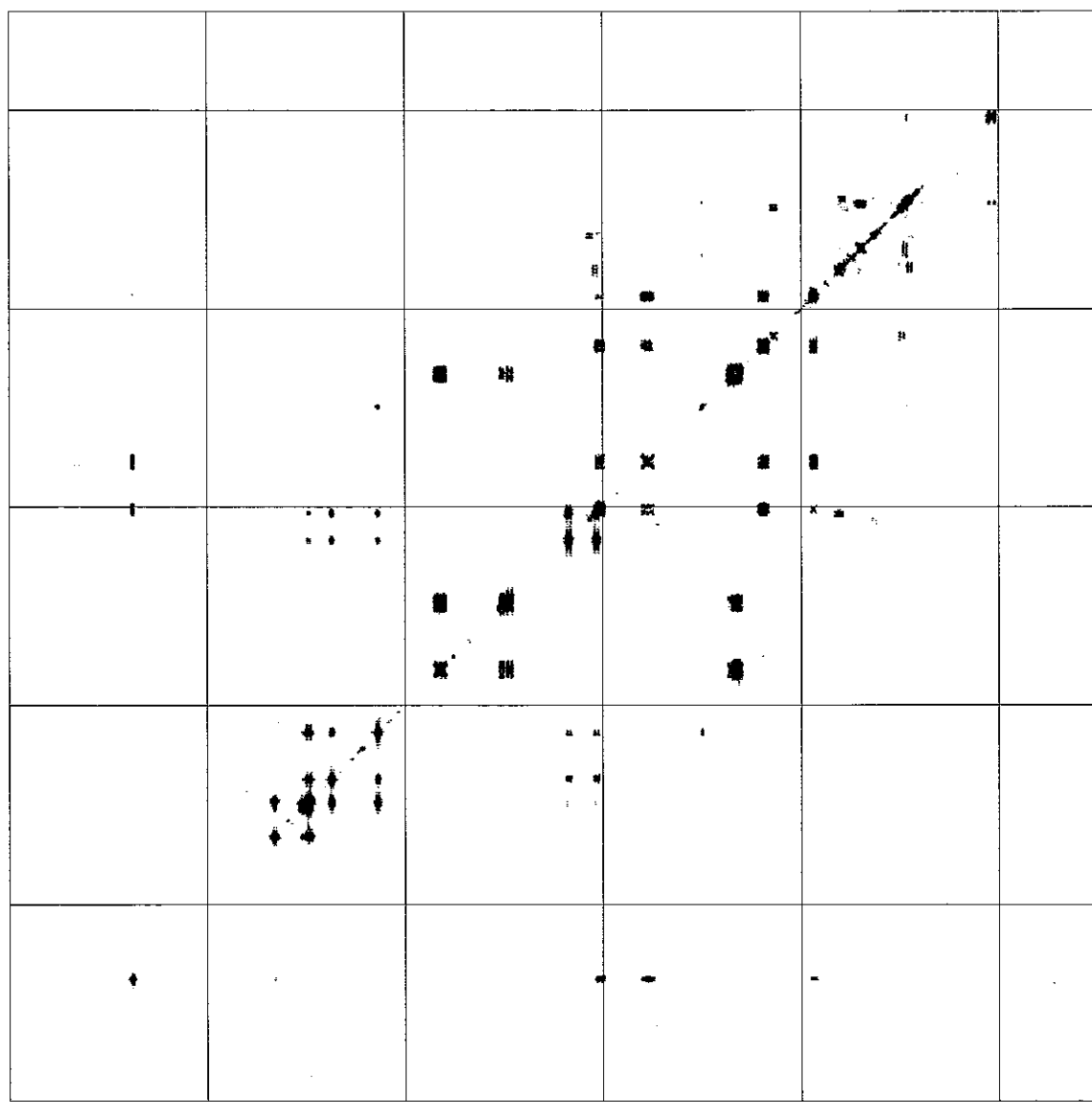
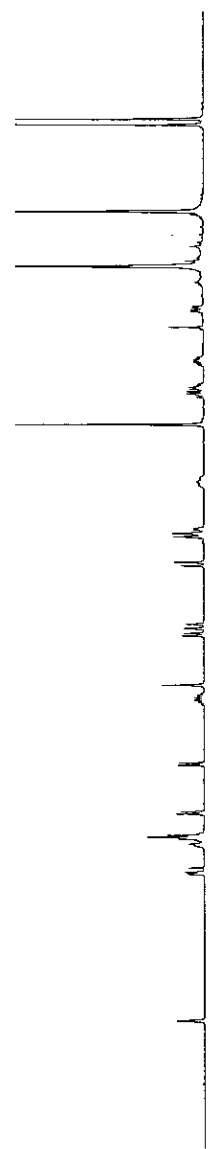
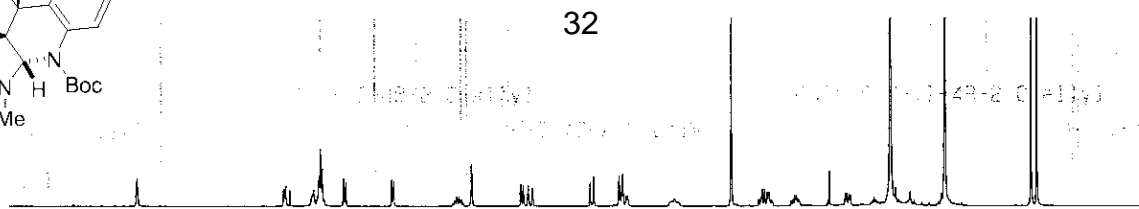
2D NMR plot parameters  
 CX2 15.00 cm  
 CN1 15.00 cm  
 FZPLO 10.017 ppm  
 FZLD 4008.87 Hz  
 FZPHL -0.983 ppm  
 FZHL -395.15 Hz  
 F1PLO 164.835 ppm  
 F1LD 16562.16 Hz  
 F1PHL -15.443 ppm  
 F1HI 1003.76 Hz  
 FZPHCK 0.72335 ppm/cm  
 FZKCM 293.42752 Hz/cm  
 F1PHCK 12.00374 ppm/cm  
 F1KCM 1207.72946 Hz/cm



32

S114

COSY



ppm

Current Data Parameters  
 NAME Dec20-SJH  
 EXPNO 8  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20071221  
 Time 9.03  
 INSTRUM spect  
 PROBD 5 mm BBI 1H-9  
 PULPROG ajb.cosygsnftp  
 TO 2048  
 SOLVENT CDCl3  
 NS 4  
 DS 6  
 SMI 4401.408 Hz  
 FIDRES 2.149125 Hz  
 AQ 0.2327028 sec  
 RG 456.1  
 DW 119.600 usec  
 DE 6.00 usec  
 TE 300.0 K  
 DO 0.02500000 sec  
 D1 4.00000000 sec  
 d13 0.00000300 sec  
 D16 0.00020000 sec  
 d20 0.00120300 sec  
 IN0 0.00011360 sec

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 7.53 usec  
 P2 15.06 usec  
 PL1 0.00 dB  
 SFO1 400.1318005 MHz

===== GRADIENT CHANNEL =====  
 P16 1000.00 usec

F1 - Acquisition parameters  
 NDO 2  
 TO 650  
 SFO1 400.1318 MHz  
 FIDRES 6.771398 Hz  
 SW 11.000 ppm

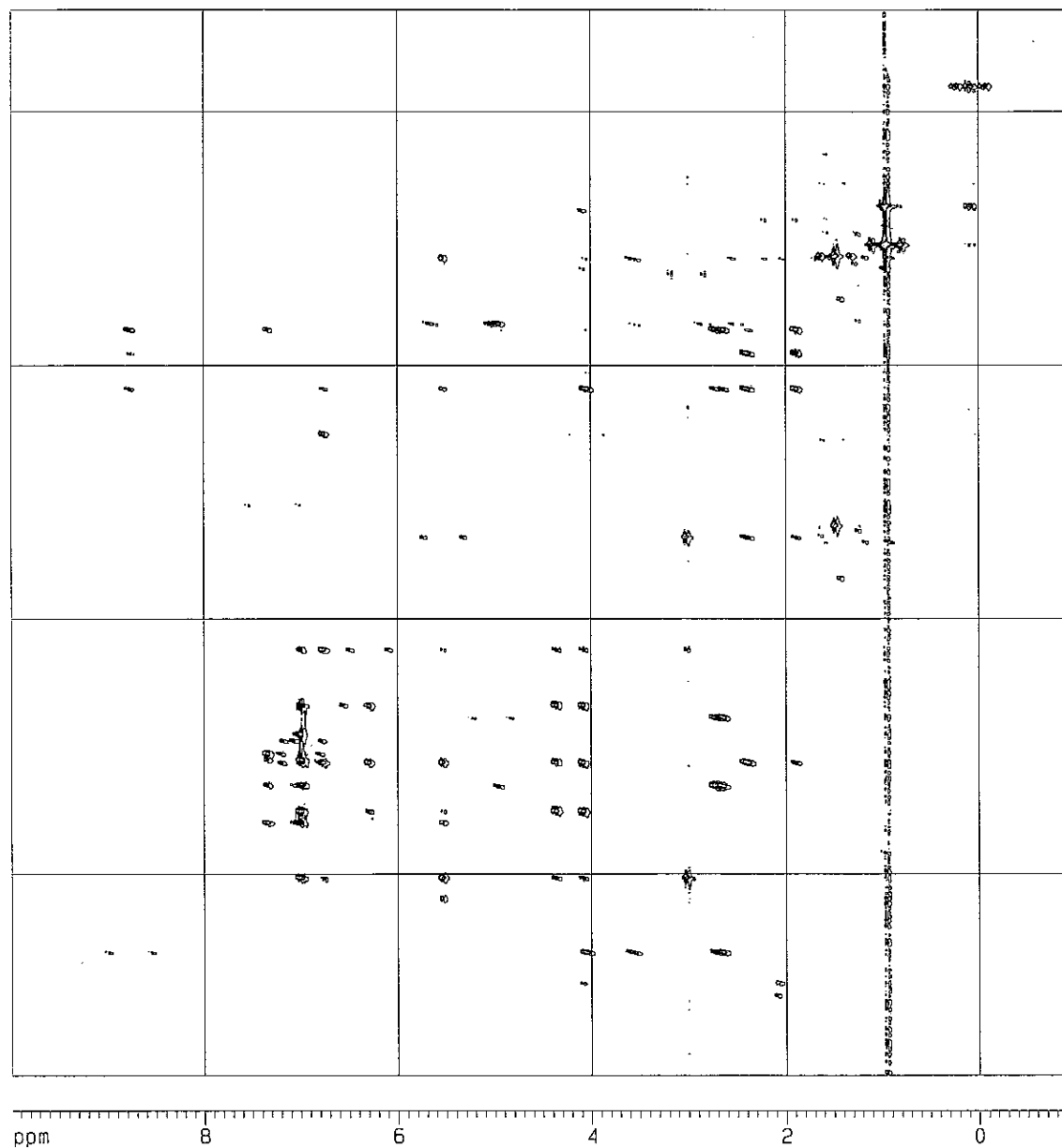
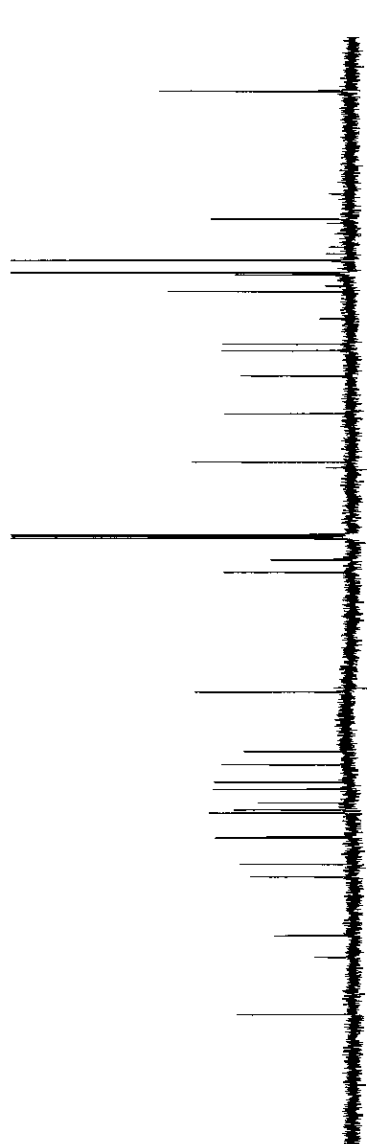
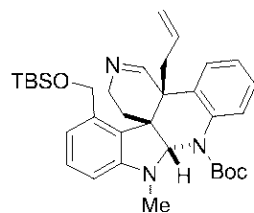
F2 - Processing parameters  
 SI 2048  
 SF 400.1300054 MHz  
 WDW SINE  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

F1 - Processing parameters  
 SI 1024  
 MC2 TPPI  
 SF 400.1300054 MHz  
 WDW SINE  
 SSB 0  
 LB 0.00 Hz  
 GB 0

2D NMR plot parameters  
 CX2 15.00 cm  
 CX1 15.00 cm  
 F2PLO 10.000 ppm  
 F2LO 4001.30 Hz  
 F2PHI -1.000 ppm  
 F2HI -400.13 Hz  
 F1PLO 10.000 ppm  
 F1LO 4001.30 Hz  
 F1PHI -1.000 ppm  
 F1HI -400.13 Hz  
 F2PRMCK 0.73333 ppm/cm  
 F2HZCM 293.42868 Hz/cm  
 F1PRMCK 0.73333 ppm/cm  
 F1HZCM 293.42868 Hz/cm

32

HMBC.



Current Data Parameters  
 NAME Dec20-SJH  
 EXPNO 6  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20071220  
 Time 23.07  
 INSTRUM spect  
 PROBHD 5 mm BBI 1H-0  
 PULPROG invg4sli1rhd  
 TD 2048  
 SOLVENT CDCl3  
 NS 16  
 DS 16  
 SWH 4401.409 Hz  
 FIDRES 2.149125 Hz  
 AQ 0.2327026 sec  
 RG 2192  
 DW 113.600 usec  
 DE 6.00 usec  
 TE 300.0 K  
 CNST2 145.000000  
 d0 0.00000300 sec  
 d1 1.50000000 sec  
 d2 0.00344828 sec  
 d3 0.02500000 sec  
 d13 0.00000300 sec  
 d15 0.00020000 sec  
 INO 0.0002368 sec

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 7.53 usec  
 p2 15.06 usec  
 PL1 0.00 dB  
 SFO1 400.1318006 MHz

===== CHANNEL f2 =====  
 NUC2 13C  
 P3 17.00 usec  
 PL2 -6.00 dB  
 SFO2 100.6212611 MHz

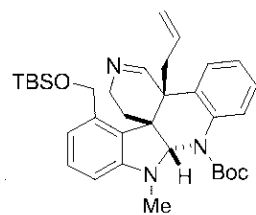
===== GRADIENT CHANNEL =====  
 P15 1000.00 usec

F1 - Acquisition parameters  
 ND0 2  
 TD 512  
 SFO1 100.6213 MHz  
 FIDRES 41.270470 Hz  
 SW 210.000 ppm

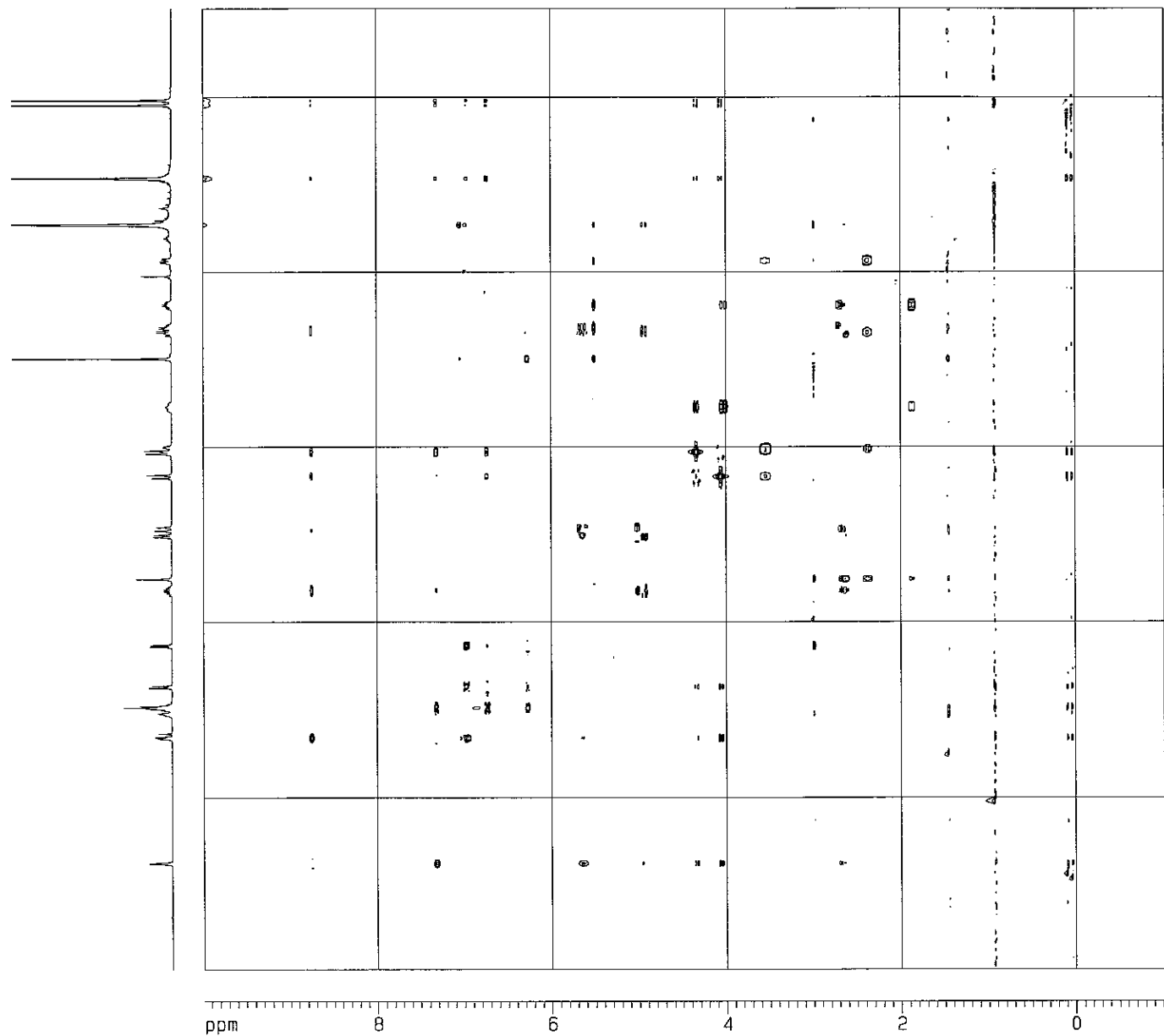
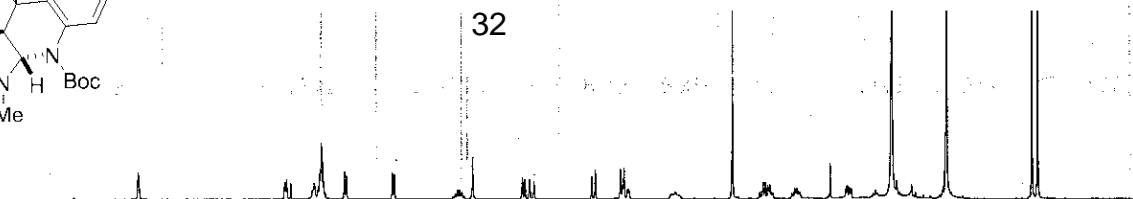
F2 - Processing parameters  
 SI 2048  
 SF 400.1300054 MHz  
 WHW SINE  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.40

F1 - Processing parameters  
 SI 1024  
 MC2 DF  
 SF 100.6127280 MHz  
 WHW SINE  
 SSB 0  
 LB 0.00 Hz  
 GB 0

2D NMR plot parameters  
 CX2 15.00 cm  
 CX1 15.00 cm  
 F2PLD 9.987 ppm  
 F2LO 3995.90 Hz  
 F2PHI -1.013 ppm  
 F2HI -405.51 Hz  
 F1PLD 190.009 ppm  
 F1LO 19117.32 Hz  
 F1PHI -20.009 ppm  
 F1HI -2013.16 Hz  
 F2PPMCM 0.73333 ppm/cm  
 F2HZCM 293.4272 Hz/cm  
 F1PPMCM 14.00120 ppm/cm  
 F1HZCM 1408.69865 Hz/cm



NOESY



Current Data Parameters  
 NAME Dec20-54H  
 EXPNO 7  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20071221  
 Time 3.16  
 INSTRUM spect  
 PROBHD 5 mm BBI 1H-8  
 PULPROG noesygptp  
 TD 2048  
 SOLVENT CDCl3  
 NS 8  
 DS 8  
 SWH 4401.409 Hz  
 FIDRES 2.149128 Hz  
 AQ 0.2327028 sec  
 RG 40.3  
 DK 113.800 usec  
 DE 6.00 usec  
 TE 300.0 K  
 d0 0.0000300 sec  
 D1 4.0000000 sec  
 DB 0.0000001 sec  
 D16 0.0002000 sec  
 d20 0.3588002 sec  
 INO 0.00011360 sec

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 7.53 usec  
 P2 15.06 usec  
 PL1 0.00 dB  
 SF01 400.1318005 MHz

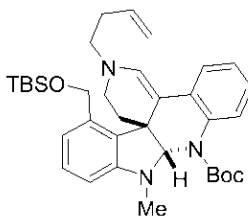
===== GRADIENT CHANNEL =====  
 GPNAM1 sine.100  
 GPNAM2 sine.100  
 GPX1 0.00 %  
 GPX2 0.00 %  
 GPY1 0.00 %  
 GPY2 0.00 %  
 GZ1 40.00 %  
 GZ2 -40.00 %  
 P16 1000.00 usec

F1 - Acquisition parameters  
 ND0 2  
 TD 512  
 SF01 400.1318 MHz  
 FIDRES 0.598501 Hz  
 SW 11.000 ppm

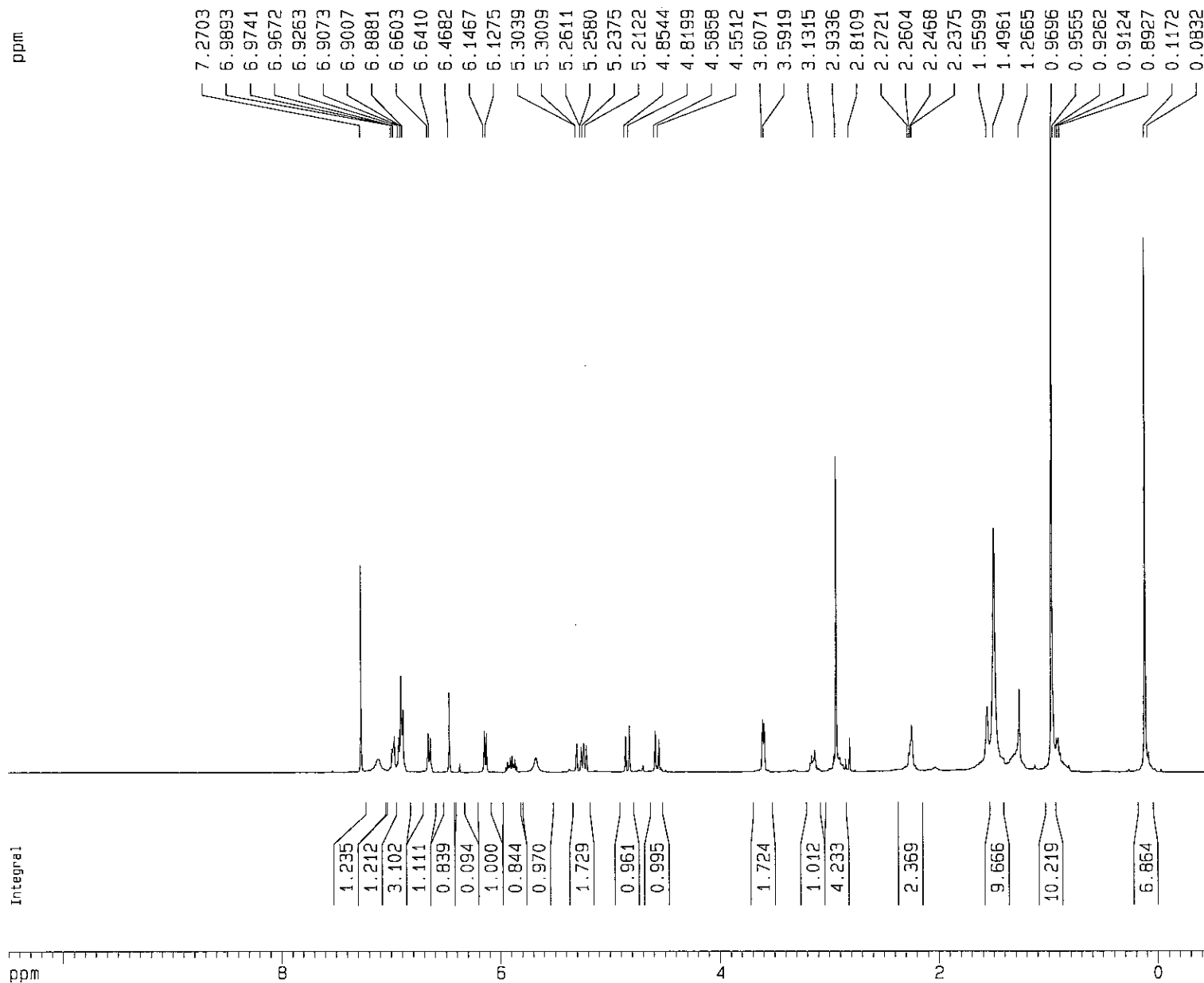
F2 - Processing parameters  
 SI 2048  
 SF 400.1300054 MHz  
 WDW GSINE  
 SSB 2  
 LB 0.00 Hz  
 GB 0  
 PC 1.40

F1 - Processing parameters  
 SI 1024  
 MC2 TPII  
 SF 400.1300054 MHz  
 WDW GSINE  
 SSB 2  
 LB 0.00 Hz  
 GB 0

20 NMR plot parameters  
 CX2 15.00 cm  
 CX1 15.00 cm  
 F2PLO 9.997 ppm  
 F2LO 3995.90 Hz  
 F2PHI -1.013 ppm  
 F2HI -405.51 Hz  
 F1PLO 9.997 ppm  
 F1LO 3995.90 Hz  
 F1PHI -1.013 ppm  
 F1HI -405.51 Hz  
 F2PPMCM 0.73333 ppm/cm  
 F2HZCM 293.42722 Hz/cm  
 F1PPMCM 0.73333 ppm/cm  
 F1HZCM 293.42722 Hz/cm



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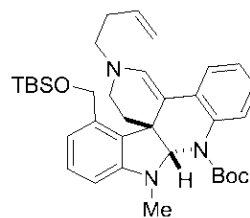
Current Data Parameters  
NAME Dec20-SJH  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20071220  
Time 19.02  
INSTRUM spect  
PROBHD 5 mm BBI 1H-B  
PULPROG zg30  
TD 49668  
SOLVENT CDCl3  
NS 17  
DS 2  
SWH 8278.146 Hz  
FIDRES 0.166670 Hz  
AQ 2.9999971 sec  
RG 406.4  
DW 60.400 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec

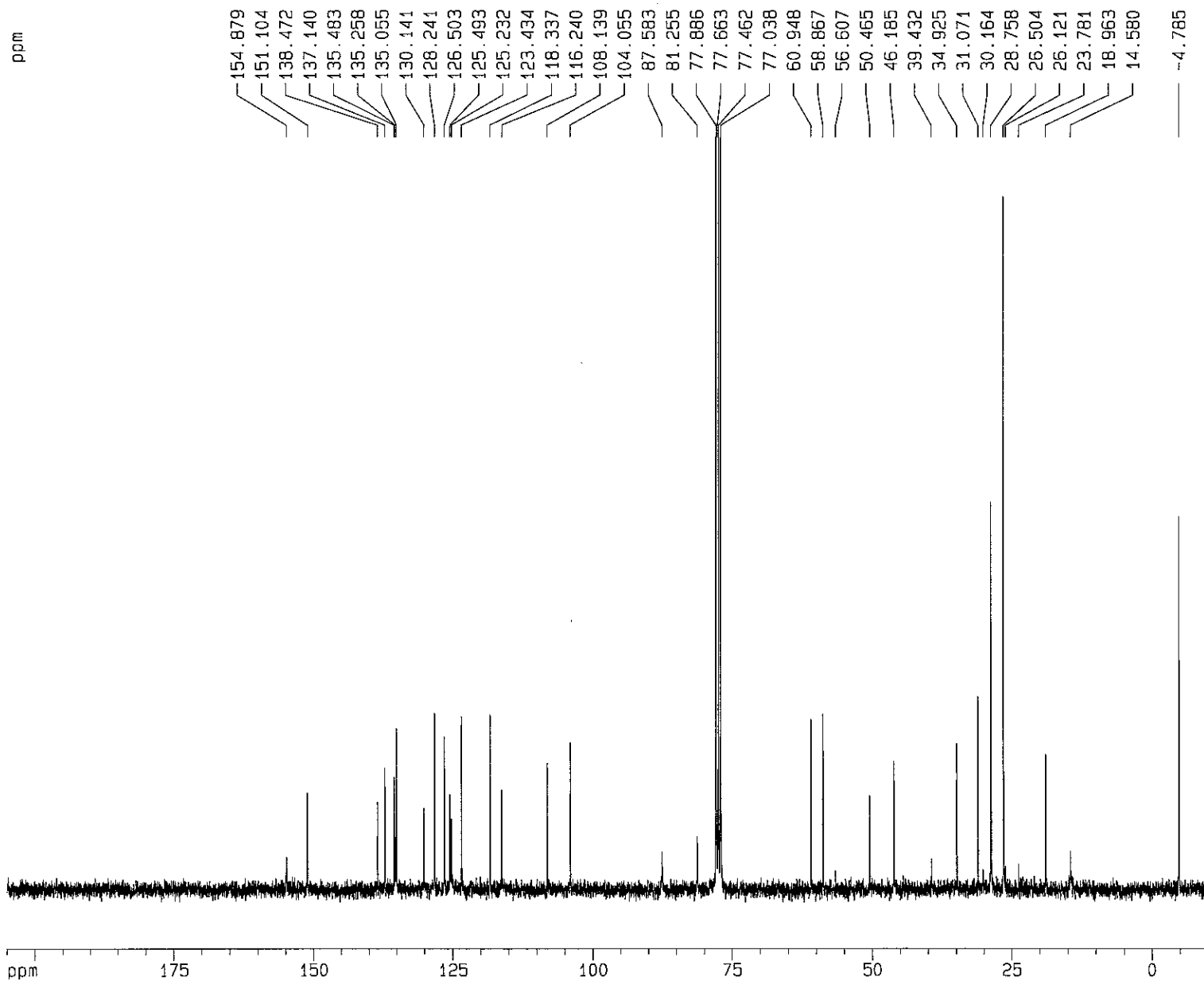
===== CHANNEL f1 =====  
NUC1 1H  
P1 6.45 usec  
PL1 0.00 dB  
SF01 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300054 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

1D NMR plot parameters  
CX 20.00 cm  
F1P 10.500 ppm  
F1 4201.37 Hz  
F2P -0.500 ppm  
F2 -200.07 Hz  
PPMCM 0.55000 ppm/cm  
HZCM 220.07150 Hz/cm



33



Current Data Parameters  
 NAME May31-SJH  
 EXPNO 2  
 PROCNO 1

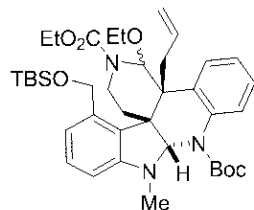
F2 - Acquisition Parameters  
 Date\_ 20080531  
 Time 10.22  
 INSTRUM spect  
 PROBHD 5 mm QNP 1H/1  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 1324  
 DS 2  
 SWH 18796.992 Hz  
 FIDRES 0.286819 Hz  
 AQ 1.7433076 sec  
 RG 512  
 DW 26.600 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 2.0000000 sec  
 D11 0.0300000 sec  
 D12 0.0000200 sec

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 5.40 usec  
 PL1 -6.00 dB  
 SF01 75.4106357 MHz

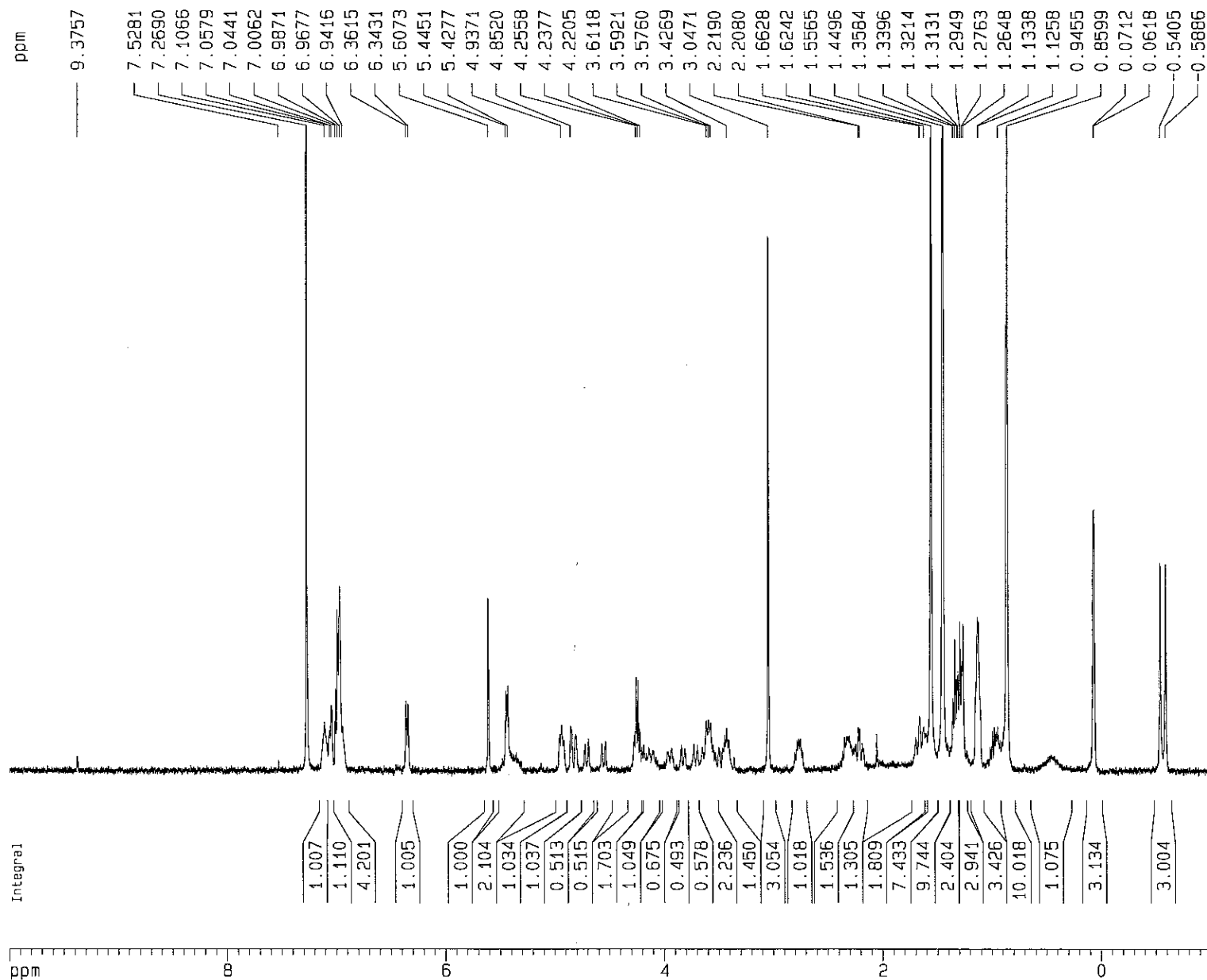
===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 PCPD2 115.00 usec  
 PL2 0.00 dB  
 PL12 20.00 dB  
 PL13 20.00 dB  
 SF02 299.8711995 MHz

F2 - Processing parameters  
 SI 32768  
 SF 75.4023410 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 205.000 ppm  
 F1 15457.48 Hz  
 F2P -10.000 ppm  
 F2 -754.02 Hz  
 PPMCM 10.75000 ppm/cm  
 HZCM 810.57520 Hz/cm



34

more polar  
major diastereomer

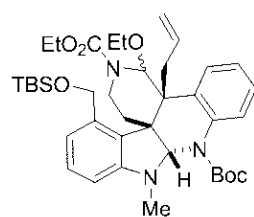
Current Data Parameters  
NAME 1124-SJH  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20071125  
Time 13.29  
INSTRUM spect  
PROBHD 5 mm BBI 1H-B  
PULPROG zg30  
TD 49668  
SOLVENT CDCl3  
NS 71  
DS 2  
SWH 8278.146 Hz  
FIDRES 0.166670 Hz  
AQ 2.9999971 sec  
RG 4096  
DW 60.400 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec

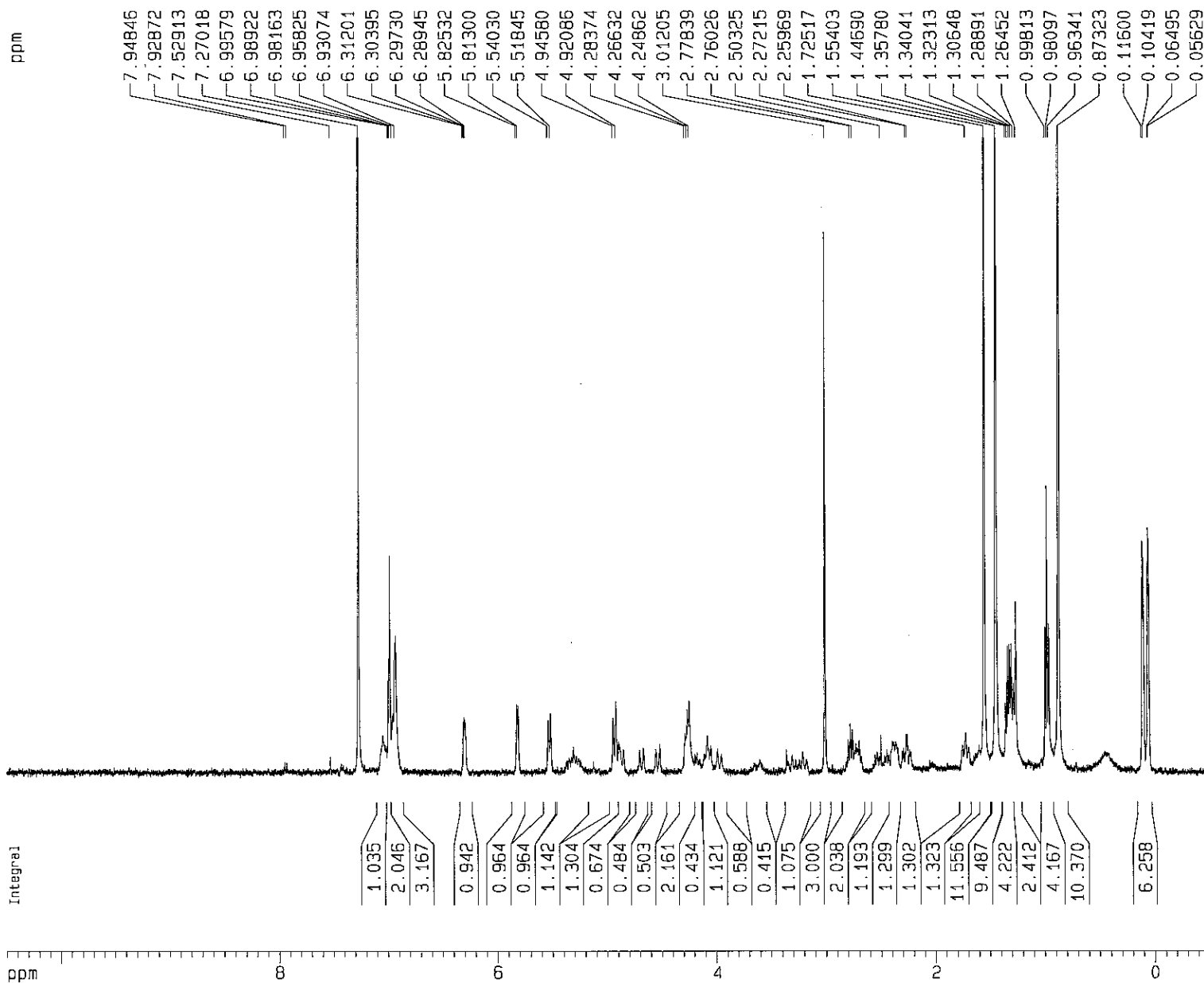
===== CHANNEL f1 =====  
NUC1 1H  
P1 6.45 usec  
PL1 0.00 dB  
SF01 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300046 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

1D NMR plot parameters  
CX 20.00 cm  
F1P 10.000 ppm  
F1 4001.30 Hz  
F2P -1.000 ppm  
F2 -400.13 Hz  
PPMCM 0.55000 ppm/cm  
HZCM 220.07150 Hz/cm

less polar  
minor diastereomer

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Current Data Parameters  
 NAME 1124-SJH  
 EXPNO 1  
 PROCNO 1

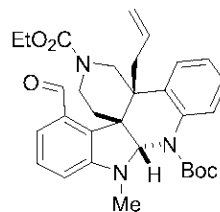
F2 - Acquisition Parameters  
 Date\_ 20071125  
 Time 13.15  
 INSTRUM spect  
 PROBHD 5 mm BBI 1H-B  
 PULPROG zg30  
 TD 49668  
 SOLVENT CDCl3  
 NS 128  
 DS 2  
 SWH 8278.146 Hz  
 FIDRES 0.166670 Hz  
 AQ 2.9999971 sec  
 RG 2580.3  
 DW 60.400 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 1.00000000 sec

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 6.45 usec  
 PL1 0.00 dB  
 SFO1 400.1324710 MHz

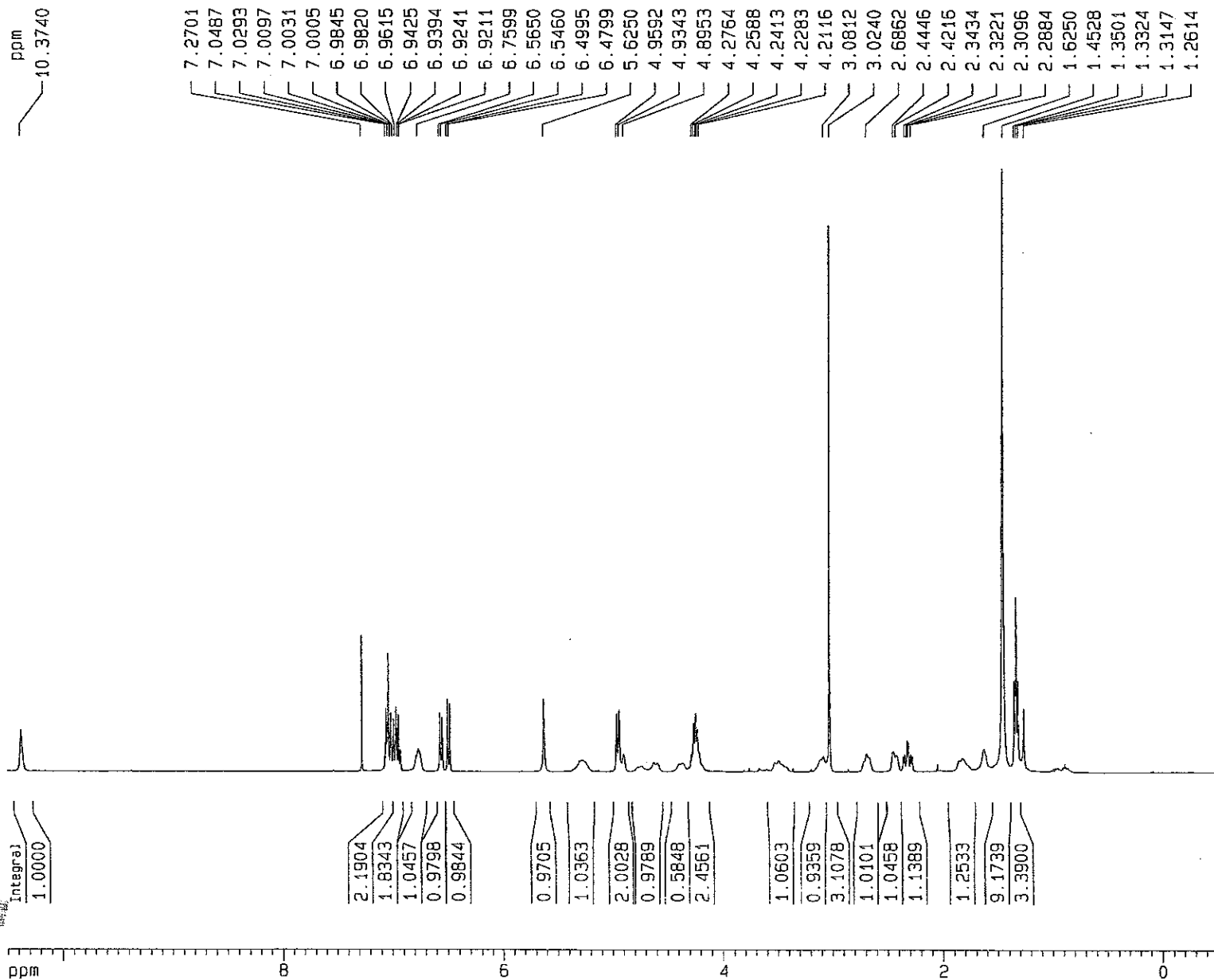
F2 - Processing parameters  
 SI 32768  
 SF 400.1300046 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 10.500 ppm  
 F1 4201.37 Hz  
 F2P -0.500 ppm  
 F2 -200.07 Hz  
 PPMCM 0.55000 ppm/cm  
 HZCM 220.07150 Hz/cm





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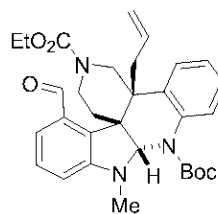
Current Data Parameters  
 NAME 0910-SJH  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20070910  
 Time 16.24  
 INSTRUM spect  
 PROBHD 5 mm BBI 1H-B  
 PULPROG zg30  
 TD 49668  
 SOLVENT CDCl3  
 NS 8  
 DS 0  
 SWH 8278.146 Hz  
 FIDRES 0.165670 Hz  
 AQ 2.9999971 sec  
 RG 35.9  
 DW 60.400 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 1.00000000 sec

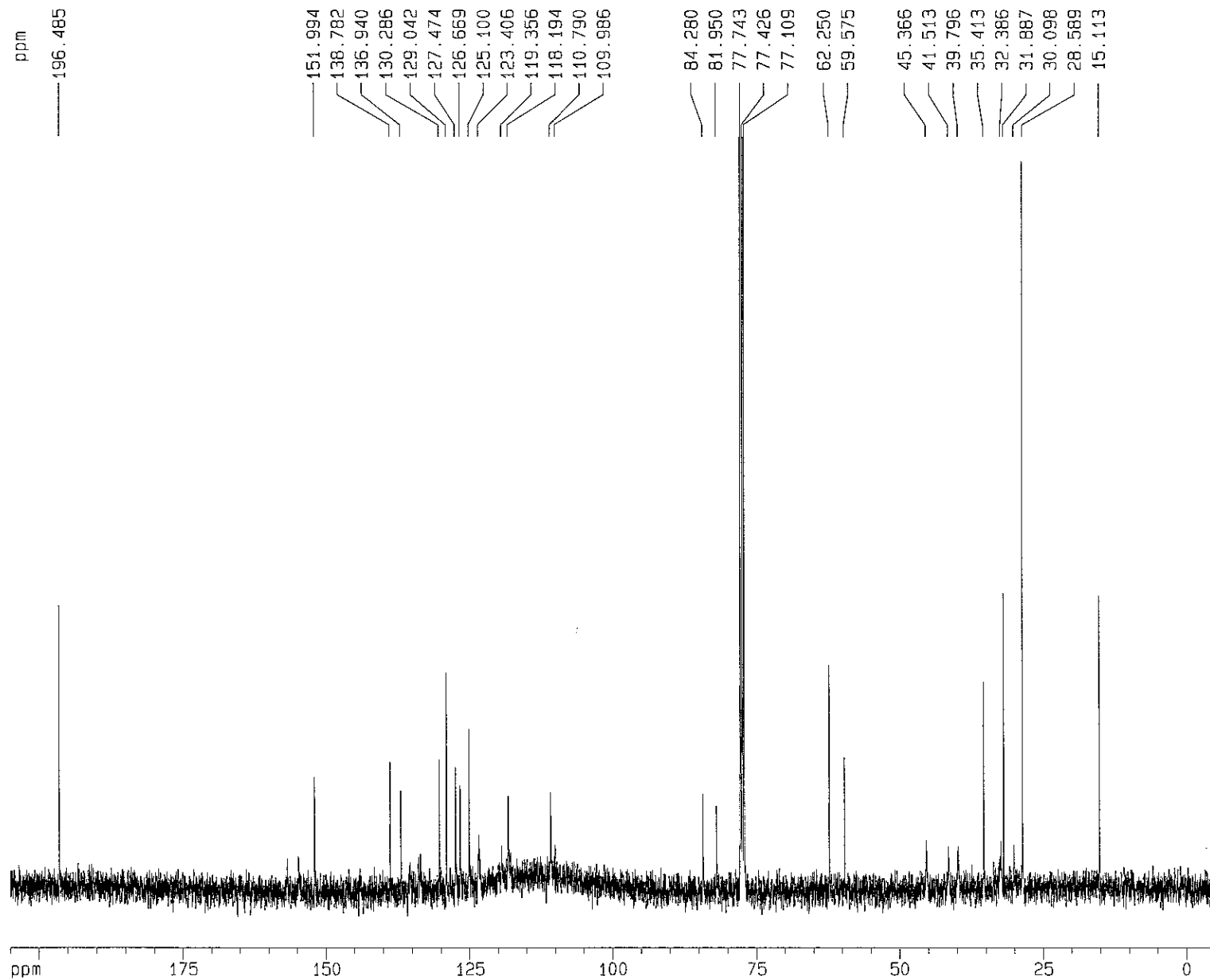
===== CHANNEL f1 =====  
 NUC1 1H  
 P1 7.15 usec  
 PL1 0.00 dB  
 SF01 400.1324710 MHz

F2 - Processing parameters  
 SI 32768  
 SF 400.1300051 MHz  
 WDW no  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 10.500 ppm  
 F1 4201.37 Hz  
 F2P -0.500 ppm  
 F2 -200.07 Hz  
 PPMCM 0.55000 ppm/cm  
 HZCM 220.07150 Hz/cm



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Current Data Parameters  
NAME 0910-SJH  
EXPNO 2  
PROCNO 1

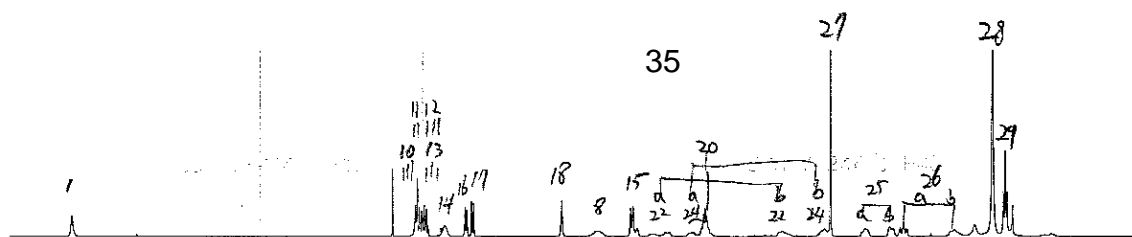
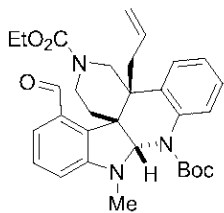
F2 - Acquisition Parameters  
Date\_ 20070910  
Time 16.54  
INSTRUM spect  
PROBHD 5 mm BBI 1H-8  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 3072  
DS 2  
SWH 25125.629 Hz  
FIDRES 0.383387 Hz  
AQ 1.3042164 sec  
RG 16384  
DW 19.900 usec  
DE 6.00 usec  
TE 300.0 K  
D1 2.0000000 sec  
d11 0.0300000 sec  
d12 0.0000200 sec

===== CHANNEL f1 =====  
NUC1 13C  
P1 15.35 usec  
PL1 -6.00 dB  
SF01 100.6237959 MHz

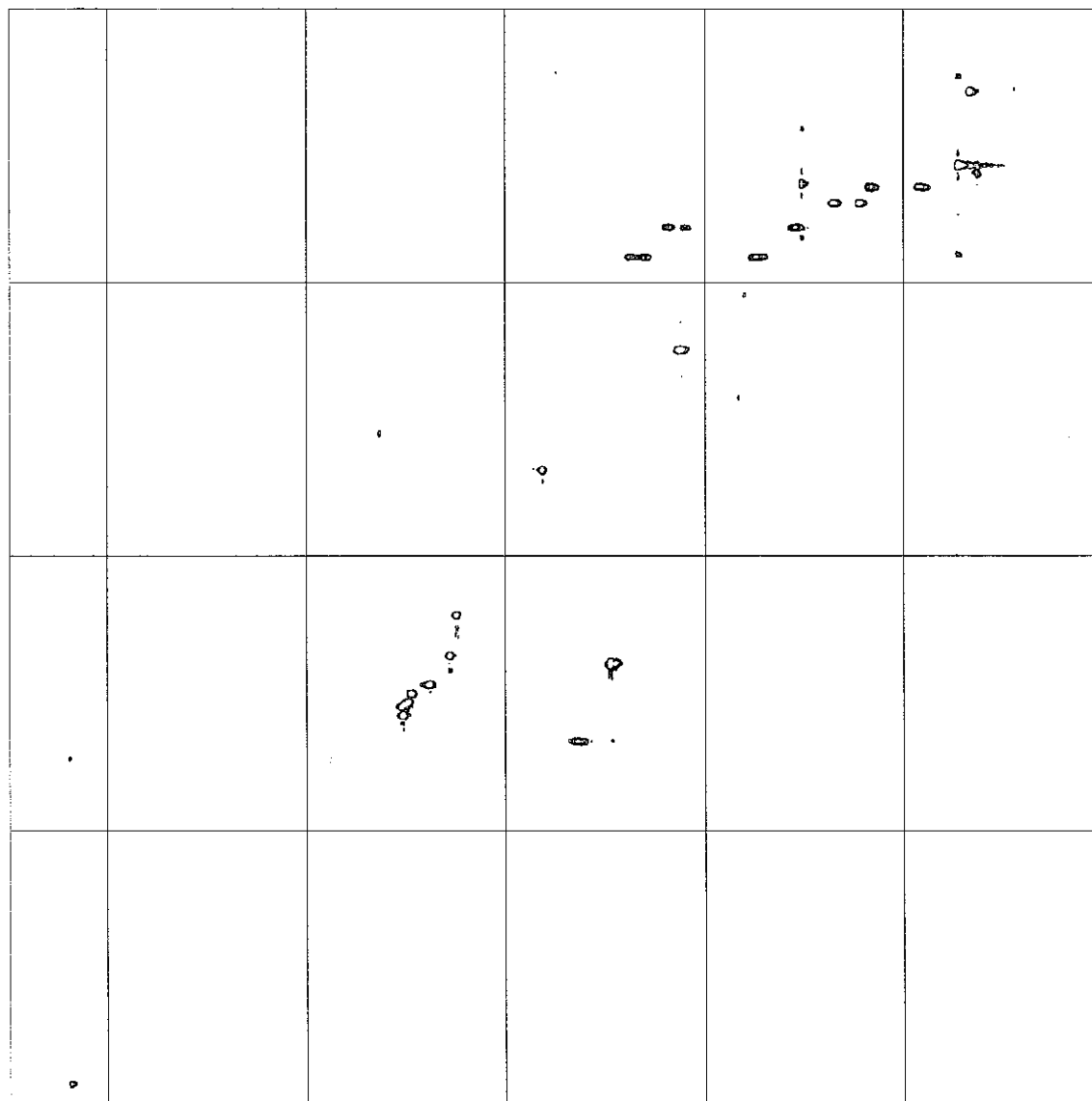
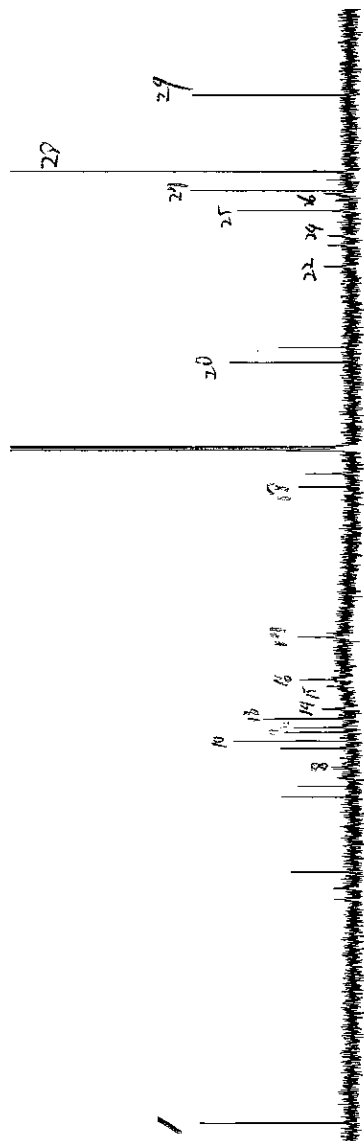
===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 114.00 usec  
PL2 0.00 dB  
PL12 24.00 dB  
PL13 24.00 dB  
SF02 400.1316005 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127290 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

1D NMR plot parameters  
CX 20.00 cm  
F1P 205.000 ppm  
F1 20625.61 Hz  
F2P -5.000 ppm  
F2 -503.06 Hz  
PPMCM 10.50000 ppm/cm  
HZCM 1056.43372 Hz/cm



HMQC



Current Data Parameters

NAME 0910-SJH  
 EXPNO 4  
 PROCNO 1

F2 - Acquisition Parameters

Date\_ 20070210  
 Time 20:42  
 INSTRUM spect  
 PROBHD 5 mm BBT 1H-B  
 PULPROG invgpg1p  
 TD 2048  
 SOLVENT CDCl3  
 NS 8  
 DS 8  
 SWH 4401.409 Hz  
 FIDRES 2.149125 Hz  
 AQ 0.2327029 sec  
 RG 23470.5  
 RM 113.500 usec  
 DE 9.00 usec  
 TE 300.0 K  
 CNST2 145.000000  
 D0 0.0000000 sec  
 D1 1.9983100 sec  
 d2 0.00344828 sec  
 d4 0.00172414 sec  
 d11 0.0300000 sec  
 d13 0.0000000 sec  
 d15 0.0002000 sec  
 d30 0.00053414 sec  
 d21 0.00224428 sec  
 IN0 0.00001243 sec

\*\*\*\*\* CHANNEL f1 \*\*\*\*\*

NUC1 1H  
 P1 6.45 usec  
 P2 12.90 usec  
 PL1 9.00 dB  
 SFO1 400.132097 MHz

\*\*\*\*\* CHANNEL f2 \*\*\*\*\*

CPDPRG2 gprf  
 NUC2 13C  
 P3 17.00 usec  
 P4 36.00 usec  
 PPRG2 gprf  
 PL2 -6.00 dB  
 PL12 6.30 dB  
 SFO2 100.627903 MHz

\*\*\*\*\* GRADIENT CHANNEL \*\*\*\*\*

OPNAK1 SINE:100  
 OPNAK2 SINE:100  
 OPNAK3 SINE:100  
 GPC1 37.00 %  
 GPC2 20.00 %  
 GPC3 25.00 %  
 GPC4 17.00 %  
 GPC5 20.00 %  
 GPC6 25.00 %  
 GPC7 17.00 %  
 GPC8 20.00 %  
 GPC9 25.00 %  
 P15 1000.00 usec

F1 - Acquisition parameters

ND0 4  
 TD 2048  
 SFO1 100.6279 MHz  
 FIDRES 78.996581 Hz  
 SN 139.562 ppm

F2 - Processing parameters

SF 400.130051 MHz  
 MDW 051NE  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.40

F1 - Processing parameters

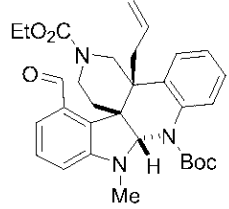
SF 1024  
 MC2 TPP1  
 SF 100.6127290 MHz  
 MDW 051NE  
 SSB 0  
 LB 0.00 Hz  
 GB 0

2D NMR plot parameters

CK2 15.00 cm  
 CK1 15.00 cm  
 F2PL0 10.987 ppm  
 F2L0 455.25 Hz  
 F2PH1 -0.013 ppm  
 F2H1 -5.12 Hz  
 F1PL0 189.981 ppm  
 F1L0 202.21 Hz  
 F1PH1 0.009 ppm  
 F1H1 0.92 Hz  
 F2PPCM 0.73323 ppm/cm  
 F2PDCM 233.42728 Hz/cm  
 F1PPCM 13.33213 ppm/cm  
 F1PDCM 1341.38159 Hz/cm

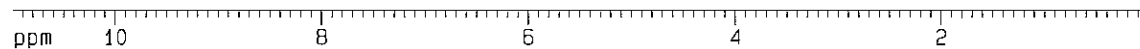
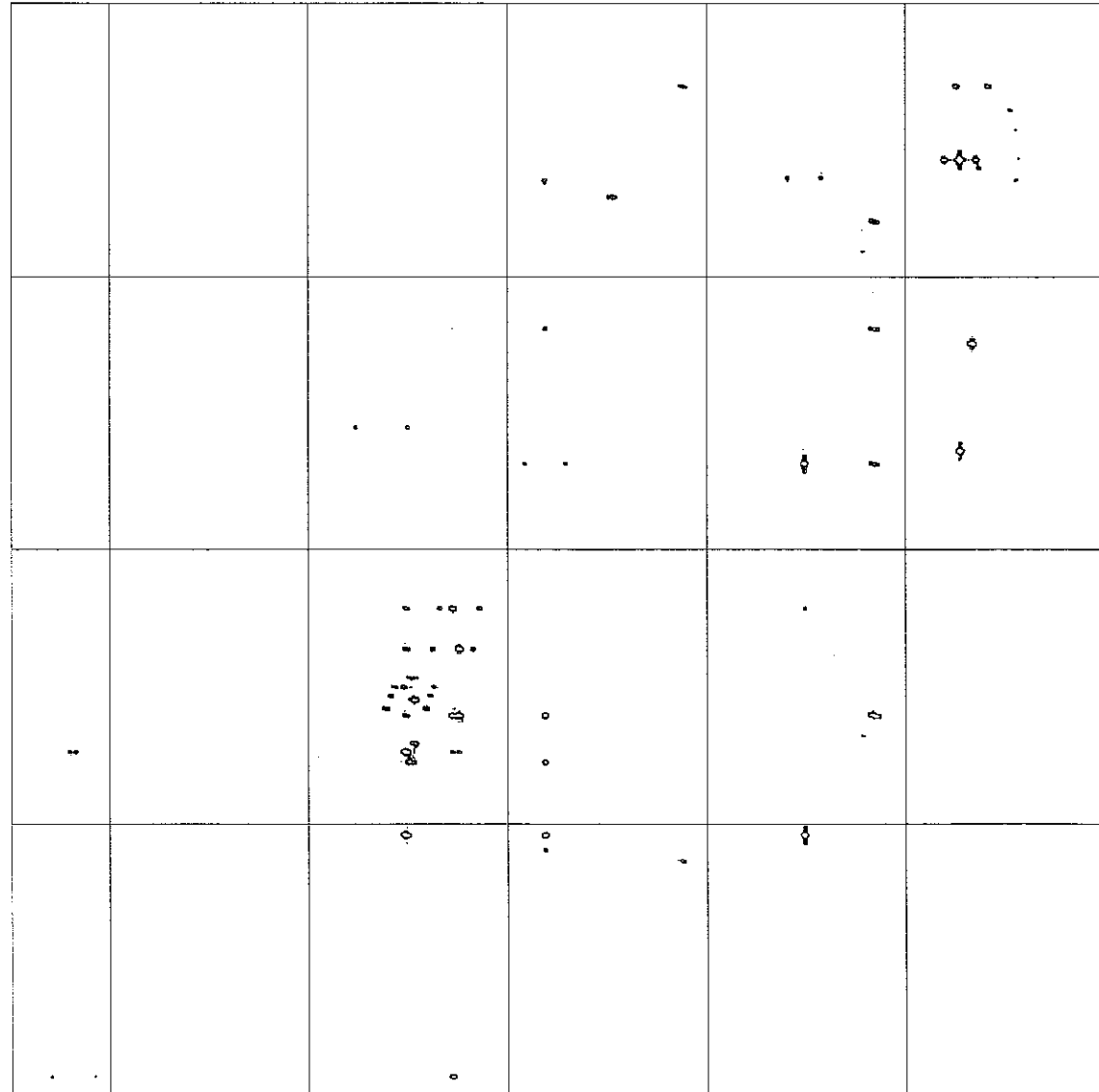
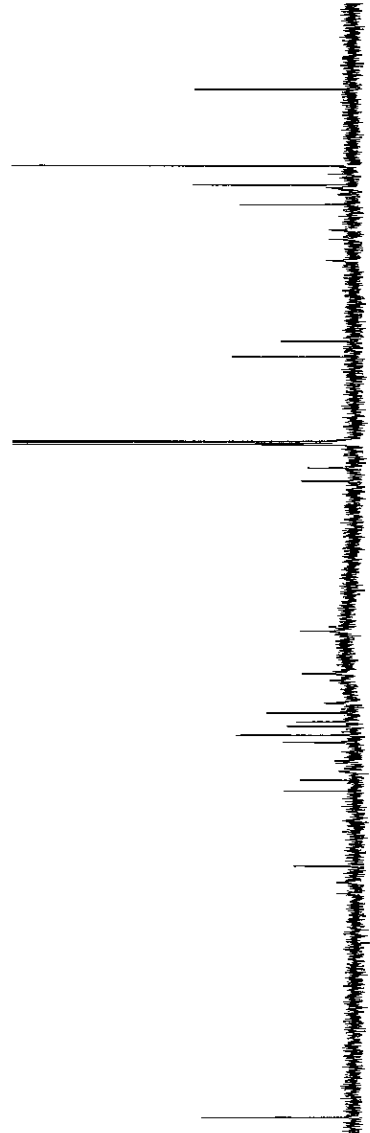
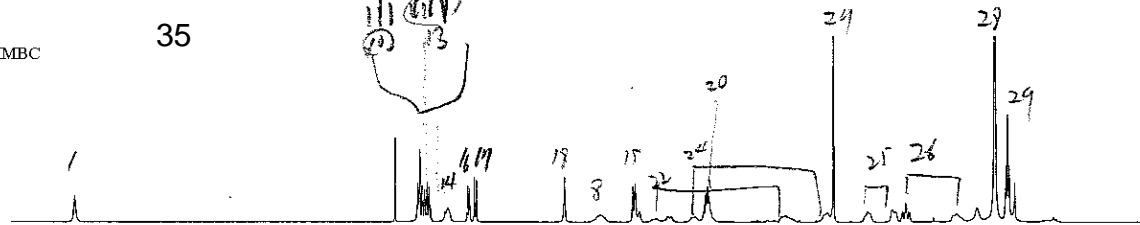


ppm



HMBC

35



S124

Current Data Parameters  
 NAME 0910-SJM  
 EXPNO 5  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20070910  
 Time 22.00  
 INSTRUM spect  
 PROBHD 5 mm BBI 1H-8  
 PULPROG inv4gs1d1rmd  
 TD 2048  
 SOLVENT CDCl3  
 NS 18  
 DS 15  
 SWH 4401.409 Hz  
 FIDRES 2.149125 Hz  
 AQ 0.2327028 sec  
 RG 15384  
 OW 113.600 usec  
 DE 6.00 usec  
 TE 300.0 K  
 CNST2 145.0000000  
 d0 0.0000300 sec  
 d1 1.500000000 sec  
 d2 0.00344828 sec  
 d5 0.06500000 sec  
 d13 0.0000300 sec  
 d15 0.0002000 sec  
 TMO 0.0002485 sec

CHANNEL f1  
 NUC1 1H  
 P1 6.45 usec  
 p2 12.90 usec  
 PL1 0.00 dB  
 SFO1 400.132007 MHz

CHANNEL f2  
 NUC2 13C  
 P3 17.00 usec  
 PL2 -6.00 dB  
 SFO2 100.627903 MHz

GRADIENT CHANNEL  
 P15 1000.00 usec

F1 - Acquisition parameters  
 NDO 2  
 TD 512  
 SFO1 100.627903 MHz  
 FIDRES 39.298290 Hz  
 SW 199.962 ppm

F2 - Processing parameters  
 SI 2048  
 SF 400.1300051 MHz  
 WDW SINE  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.40

F1 - Processing parameters  
 SI 1024  
 MC2 OF  
 SF 100.6127290 MHz  
 WDW SINE  
 SSB 0  
 LB 0.00 Hz  
 GB 0

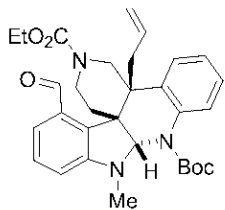
20 NMR plot parameters  
 CX2 15.00 cm  
 CX1 15.00 cm  
 F2PL0 10.987 ppm  
 F2L0 4396.29 Hz  
 F2PHI -0.013 ppm  
 F2H1 -5.12 Hz  
 F1PL0 199.991 ppm  
 F1L0 20321.64 Hz  
 F1PHI 0.009 ppm  
 F1H1 0.92 Hz  
 F2PPMCM 0.73333 ppm/cm  
 F2HZCM 293.42722 Hz/cm  
 F1PPMCM 13.33213 ppm/cm  
 F1HZCM 1341.39153 Hz/cm

50

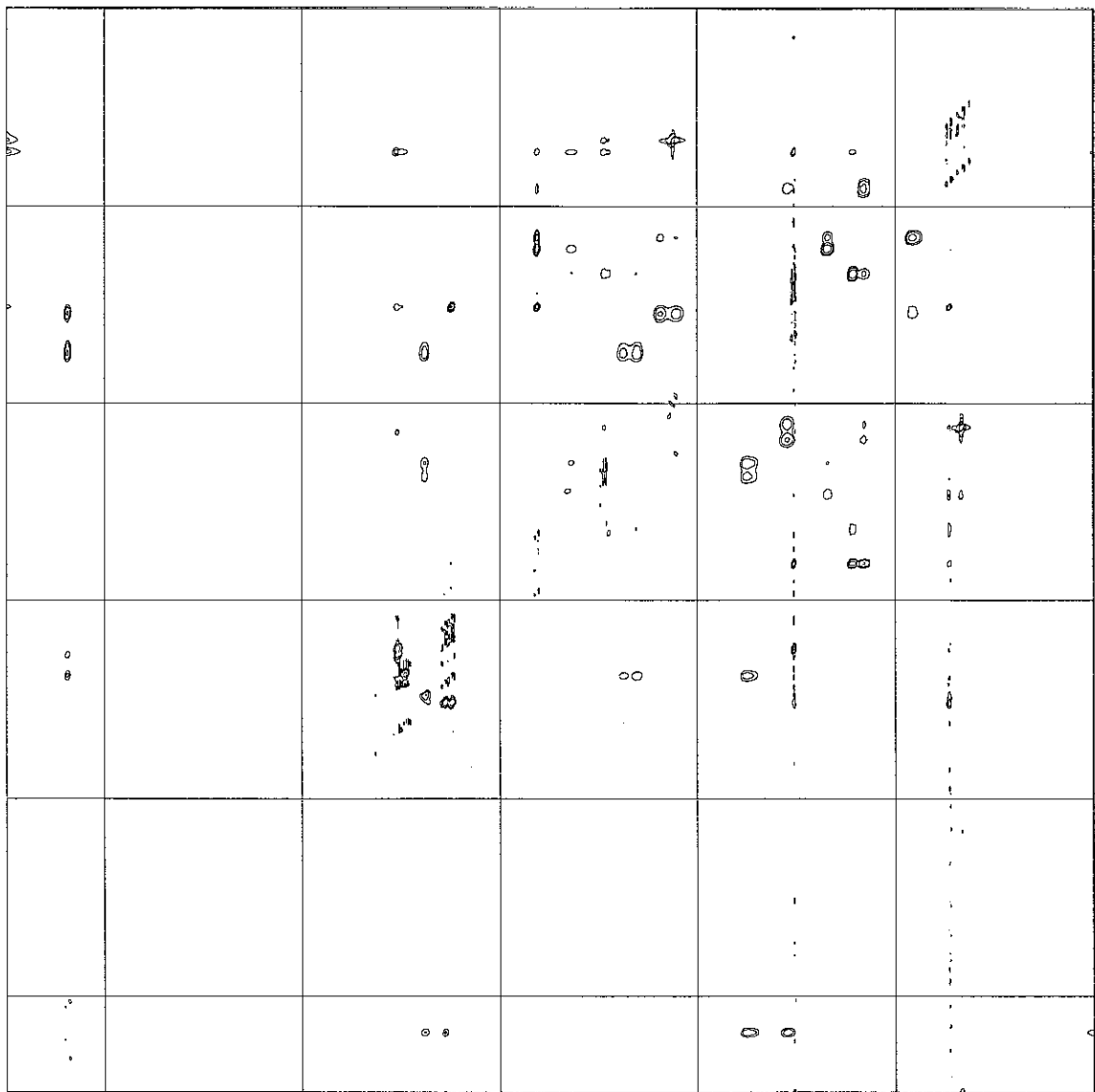
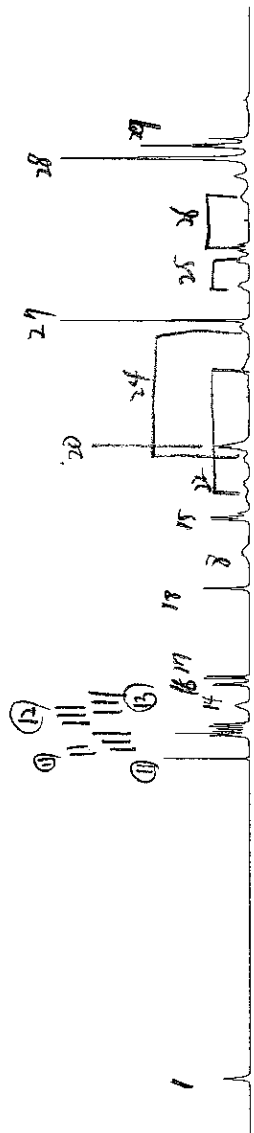
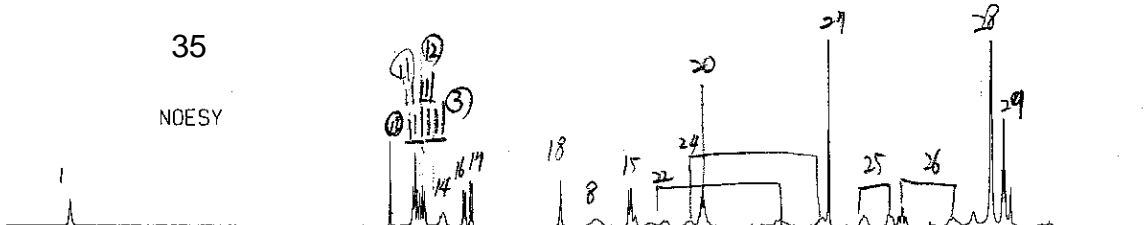
100

150

ppm



35  
NOESY



ppm 10 8 6 4 2

S125

Current Data Parameters  
NAME 0810-5JH  
EXPNO 6  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20070911  
Time 2.10  
INSTRUM spect  
PROBHD 5 mm BB1 1H-5  
PULPROG noesygprb  
TD 2048  
SOLVENT CDCl3  
NS 10  
DS 8  
SWH 4401.408 Hz  
FIDRES 2.149125 Hz  
AQ 0.2327028 sec  
RG 90.5  
DN 113.600 usec  
DE 6.00 usec  
TE 300.0 K  
d0 0.0000000 sec  
D1 4.0000000 sec  
D8 0.8000001 sec  
D16 0.0002000 sec  
d20 0.3980002 sec  
IN0 0.00011360 sec

----- CHANNEL f1 -----  
NUC1 1H  
P1 6.45 usec  
P2 12.90 usec  
PL1 0.00 dB  
SF01 400.1322007 MHz

----- GRADIENT CHANNEL -----  
GPNAM1 sine.100  
GPNAM2 sine.100  
GPX1 0.00 %  
GPX2 0.00 %  
GPY1 0.00 %  
GPY2 0.00 %  
GPZ1 40.00 %  
GPZ2 -40.00 %  
P16 1000.00 usec

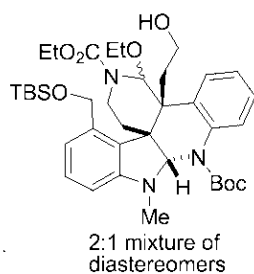
F1 - Acquisition parameters  
NUC 2  
TU 512  
SF01 400.1322 MHz  
FIDRES 8.596501 Hz  
SW 11.000 ppm

F2 - Processing parameters  
SI 2048  
SF 400.1300051 MHz  
WDW GSINE  
SSB 2  
LB 0.00 Hz  
GB 0  
PC 1.40

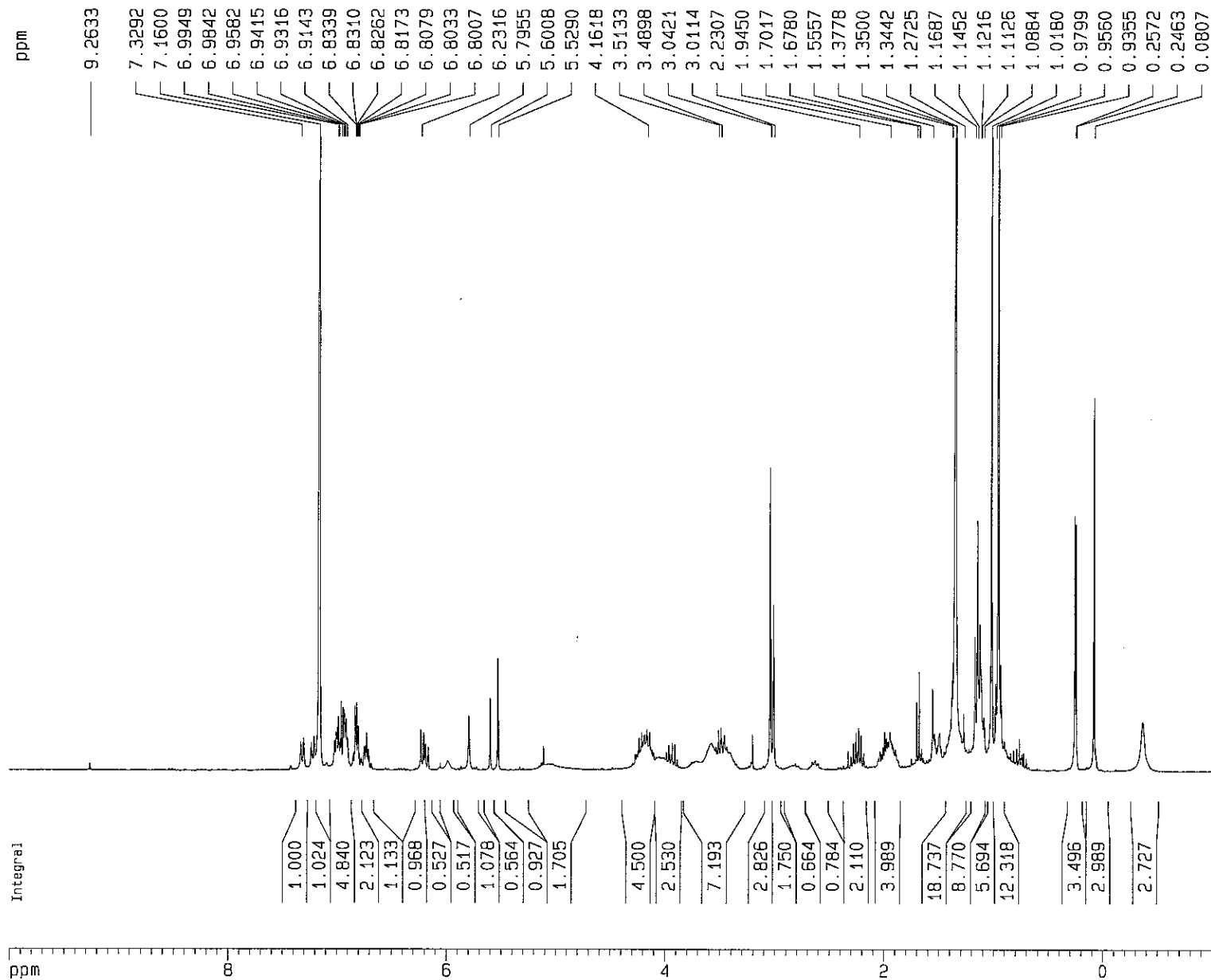
F1 - Processing parameters  
SI 1024  
WDW TRF1  
SF 400.1300051 MHz  
WDW GSINE  
SSB 2  
LB 0.00 Hz  
GB 0

2D NMR plot parameters  
CX2 15.00 cm  
CX1 15.00 cm  
F2PLO 10.987 ppm  
F2LO 4395.29 Hz  
F2PHI -0.013 ppm  
F2HI -5.12 Hz  
F1PLO 10.987 ppm  
F1LO 4395.29 Hz  
F1PHI -0.013 ppm  
F1HI -5.12 Hz  
F2PPMCM 0.73333 ppm/cm  
F2HZCM 293.42722 Hz/cm  
F1PPMCM 0.73333 ppm/cm  
F1HZCM 293.42722 Hz/cm

2  
4  
6  
8  
10  
ppm



36



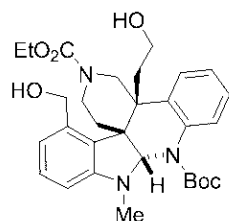
Current Data Parameters  
NAME Jun16-SJH  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20080616  
Time 12.38  
INSTRUM spect  
PROBHD 5 mm QNP 1H/1  
PULPROG zg30  
TD 37036  
SOLVENT C6D6  
NS 17  
DS 2  
SWH 6172.839 Hz  
FIDRES 0.166671 Hz  
AQ 2.9999659 sec  
RG 362  
DW 81.000 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec

===== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 0.00 dB  
SF01 299.8718518 MHz

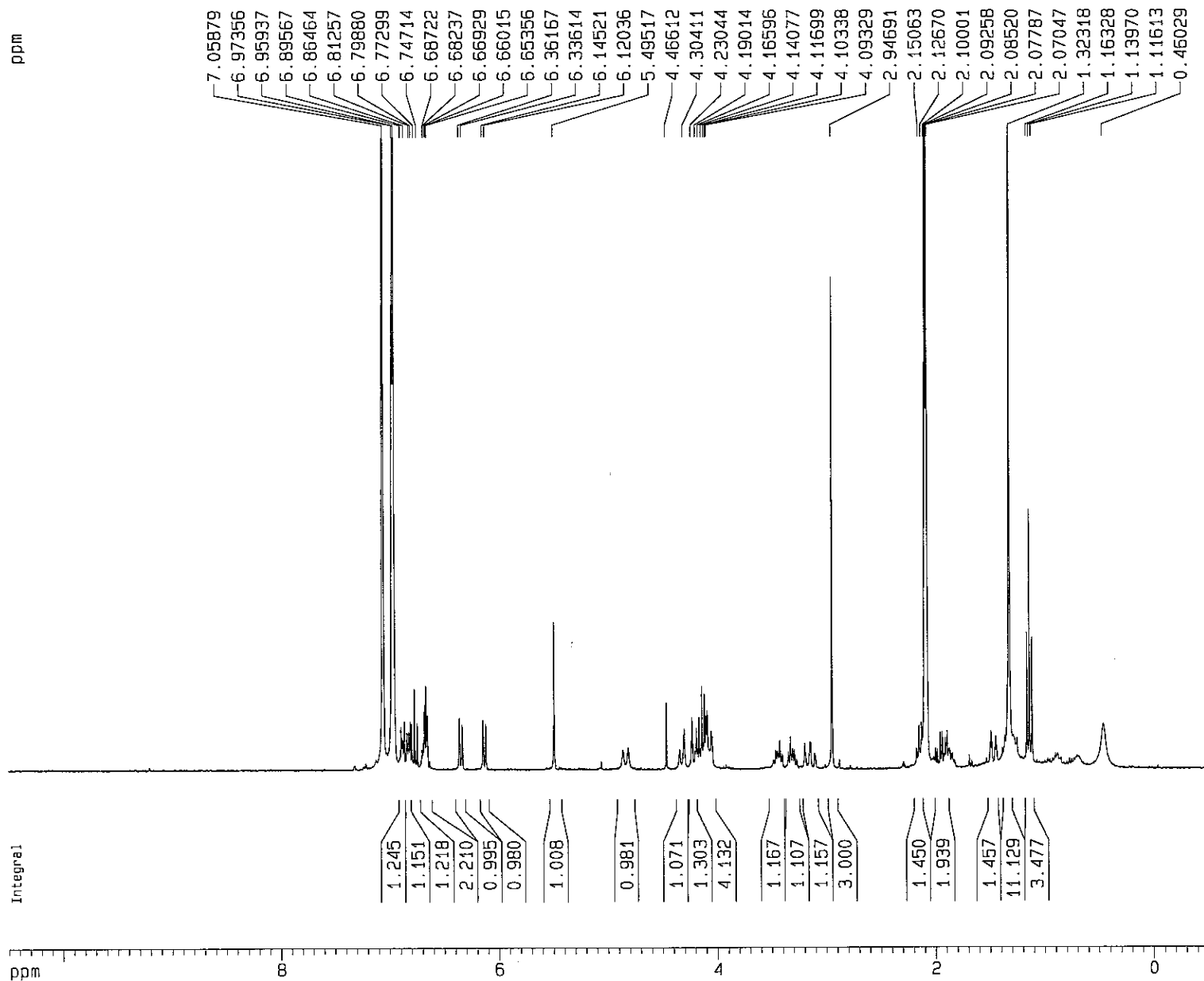
F2 - Processing parameters  
SI 32768  
SF 299.8700355 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

1D NMR plot parameters  
CX 20.00 cm  
F1P 10.000 ppm  
F1 2998.70 Hz  
F2P -1.000 ppm  
F2 -299.87 Hz  
PPMCM 0.55000 ppm/cm  
HZCM 164.92851 Hz/cm



in tol at 90 oC

37



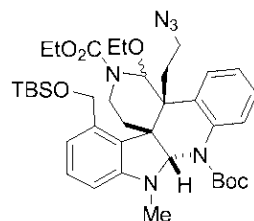
Current Data Parameters  
 NAME Jun06-SJH  
 EXPNO 10  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20080606  
 Time 22.22  
 INSTRUM spect  
 PROBHD 5 mm GNP 1H/1  
 PULPROG zg30  
 TD 37036  
 SOLVENT Tol  
 NS 65  
 DS 2  
 SWH 6172.839 Hz  
 FIDRES 0.166671 Hz  
 AQ 2.9999659 sec  
 RG 1625.5  
 DW 81.000 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 1.00000000 sec

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 11.70 usec  
 PL1 0.00 dB  
 SFO1 299.8718518 MHz

F2 - Processing parameters  
 SI 32768  
 SF 299.8700136 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

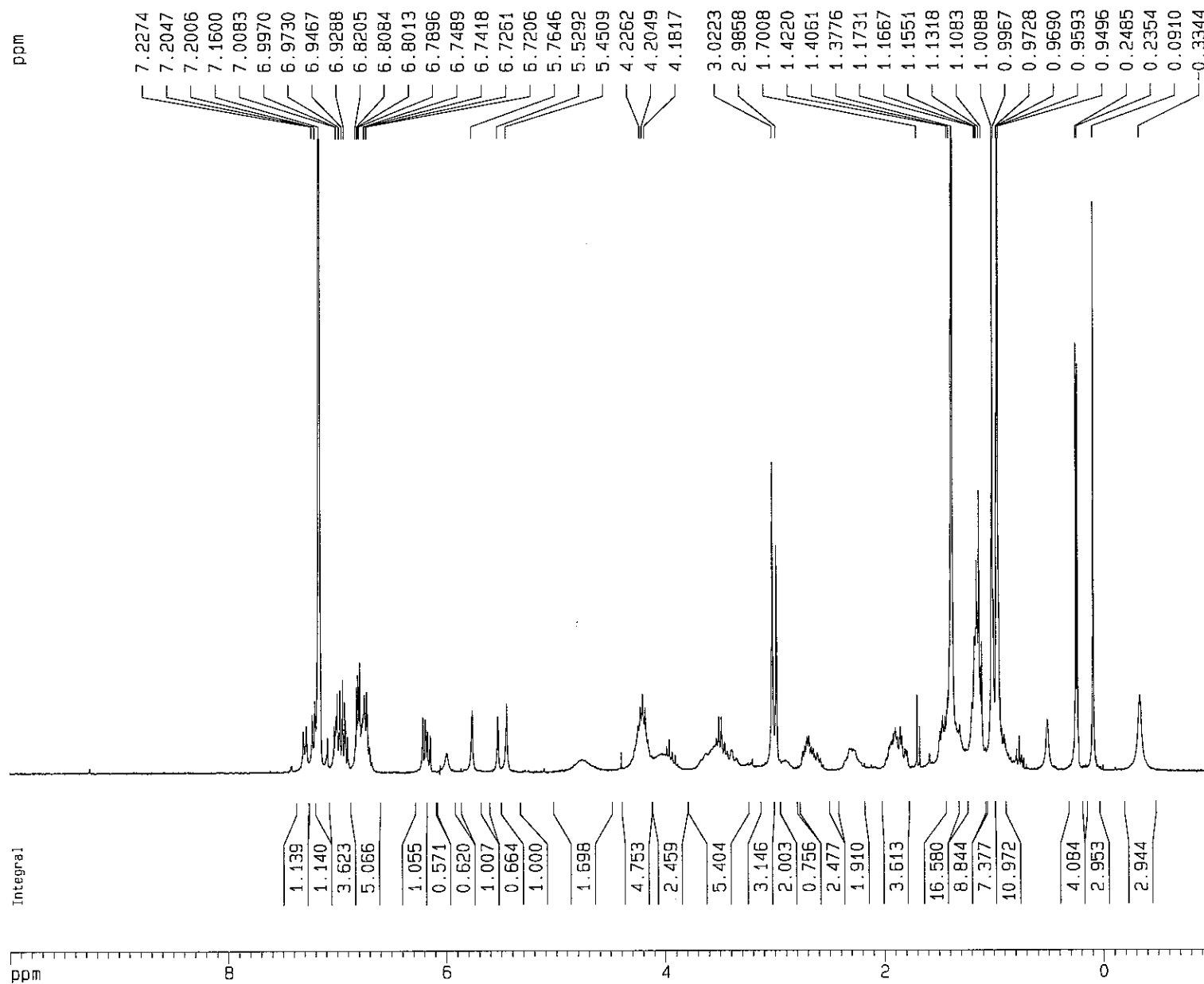
1D NMR plot parameters  
 CX 20.00 cm  
 F1P 10.500 ppm  
 F1 3148.64 Hz  
 F2P -0.500 ppm  
 F2 -149.94 Hz  
 PPMCM 0.55000 ppm/cm  
 HZCM 164.92851 Hz/cm



in benzene at 65 oC

65

1:0.6 mixture of diastereomers



## Current Data Parameters

NAME Jun17-SJH  
EXPNO 1  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20080617  
Time 14.10  
INSTRUM spect  
PROBHD 5 mm GNP 1H/1  
PULPROG zg30  
TD 37036  
SOLVENT C6D6  
NS 13  
DS 2  
SWH 6172.839 Hz  
FIDRES 0.166671 Hz  
AQ 2.9999659 sec  
RG 362  
DW 81.000 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec

## ===== CHANNEL f1 =====

NUC1 1H  
P1 11.70 usec  
PL1 0.00 dB  
SF01 299.8718518 MHz

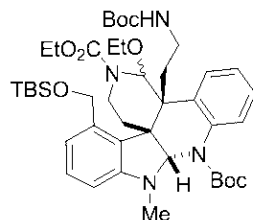
## F2 - Processing parameters

SI 32768  
SF 299.8700355 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

## 1D NMR plot parameters

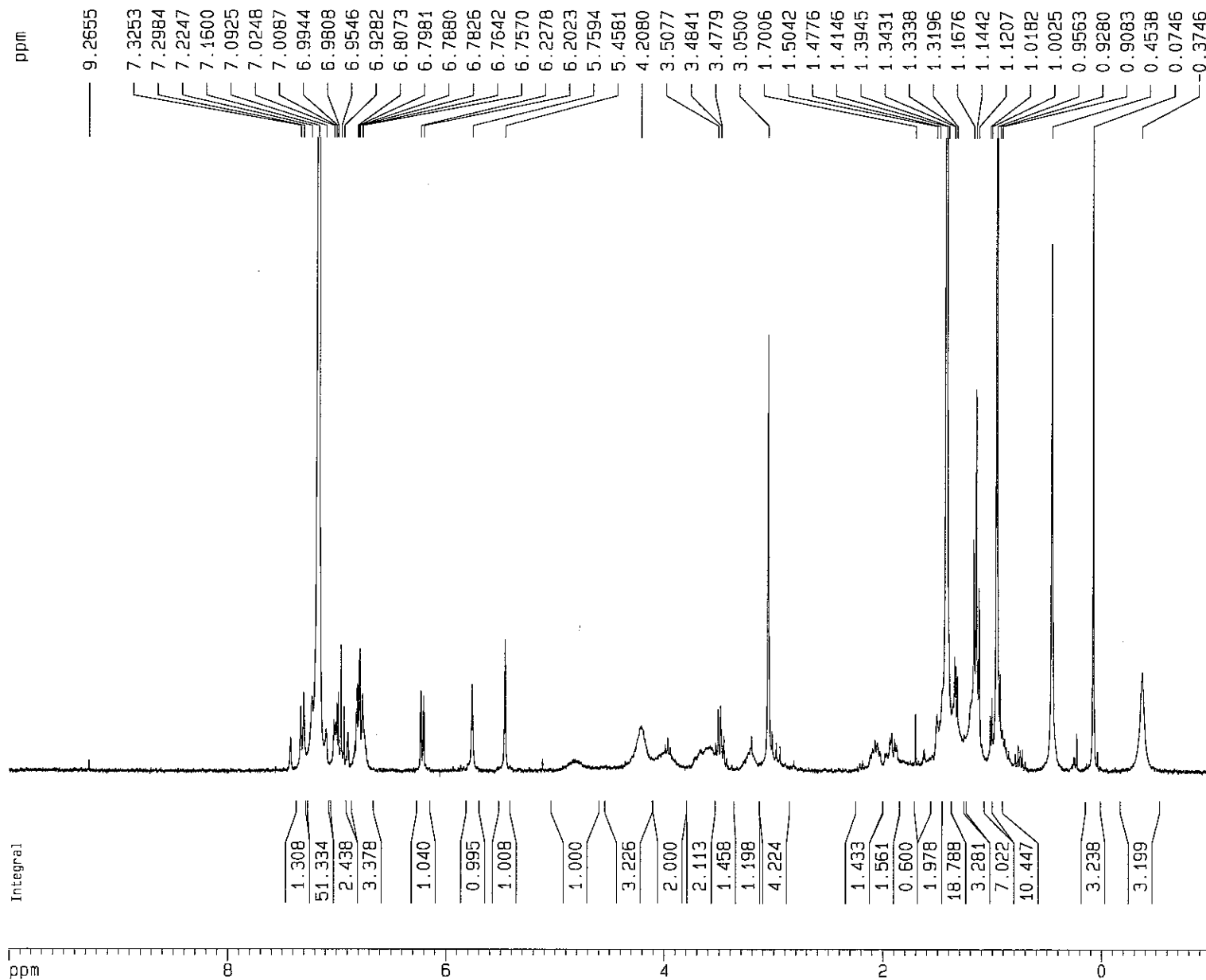
CX 20.00 cm  
F1P 10.000 ppm  
F1 2998.70 Hz  
F2P -1.000 ppm  
F2 -299.87 Hz  
PPMCM 0.55000 ppm/cm  
HZCM 164.92851 Hz/cm





in benzene-d6 at 65 oC

38

more polar  
major diastereomer

## Current Data Parameters

NAME Jun18-SJH  
EXPNO 3  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20080618  
Time 11.12  
INSTRUM spect  
PROBHD 5 mm QNP 1H/1  
PULPROG zg30  
TD 37036  
SOLVENT C6D6  
NS 51  
DS 2  
SWH 6172.839 Hz  
FIDRES 0.165671 Hz  
AQ 2.9999659 sec  
RG 1149.4  
DW 81.000 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec

## ===== CHANNEL f1 =====

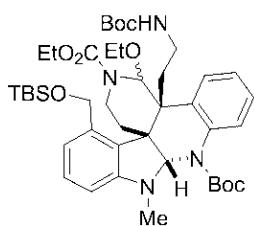
NUC1 1H  
P1 11.70 usec  
PL1 0.00 dB  
SF01 299.8718518 MHz

## F2 - Processing parameters

SI 32768  
SF 299.8700355 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

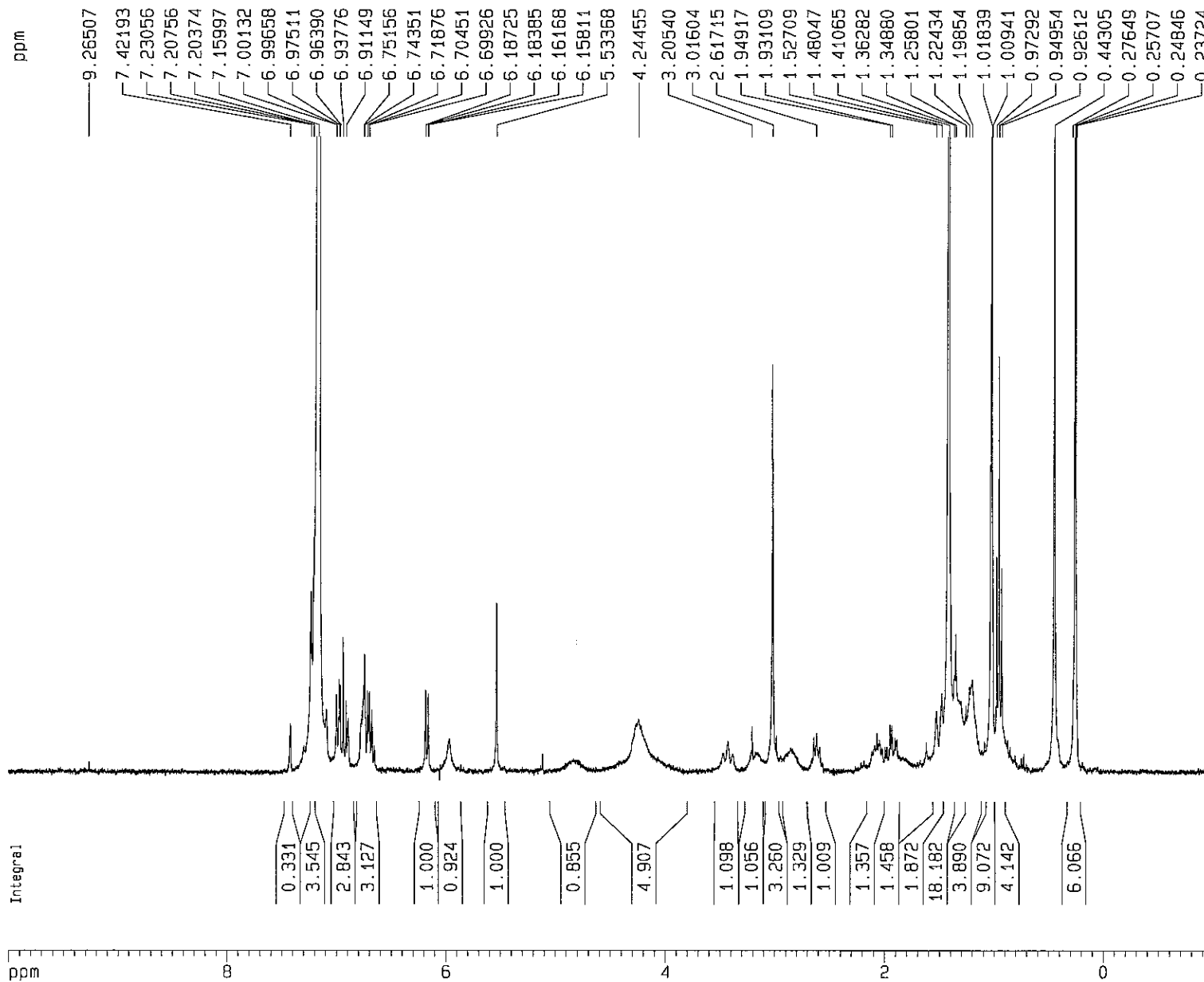
## 1D NMR plot parameters

CX 20.00 cm  
F1P 10.000 ppm  
F1 2998.70 Hz  
F2P -1.000 ppm  
F2 -299.87 Hz  
PPMCM 0.55000 ppm/cm  
HZCM 164.92851 Hz/cm



in benzene-d6 at 65 oC

38

less polar  
minor diastereomer

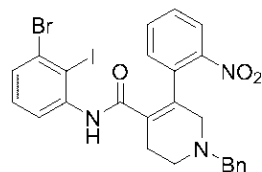
Current Data Parameters  
NAME Jun18-SJH  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20080618  
Time 10.51  
INSTRUM spect  
PROBHD 5 mm QNP 1H/1  
PULPROG zg30  
TD 37036  
SOLVENT C6D6  
NS 75  
DS 2  
SWH 6172.839 Hz  
FIDRES 0.166671 Hz  
AQ 2.9999659 sec  
RG 1149.4  
DW 81.000 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec

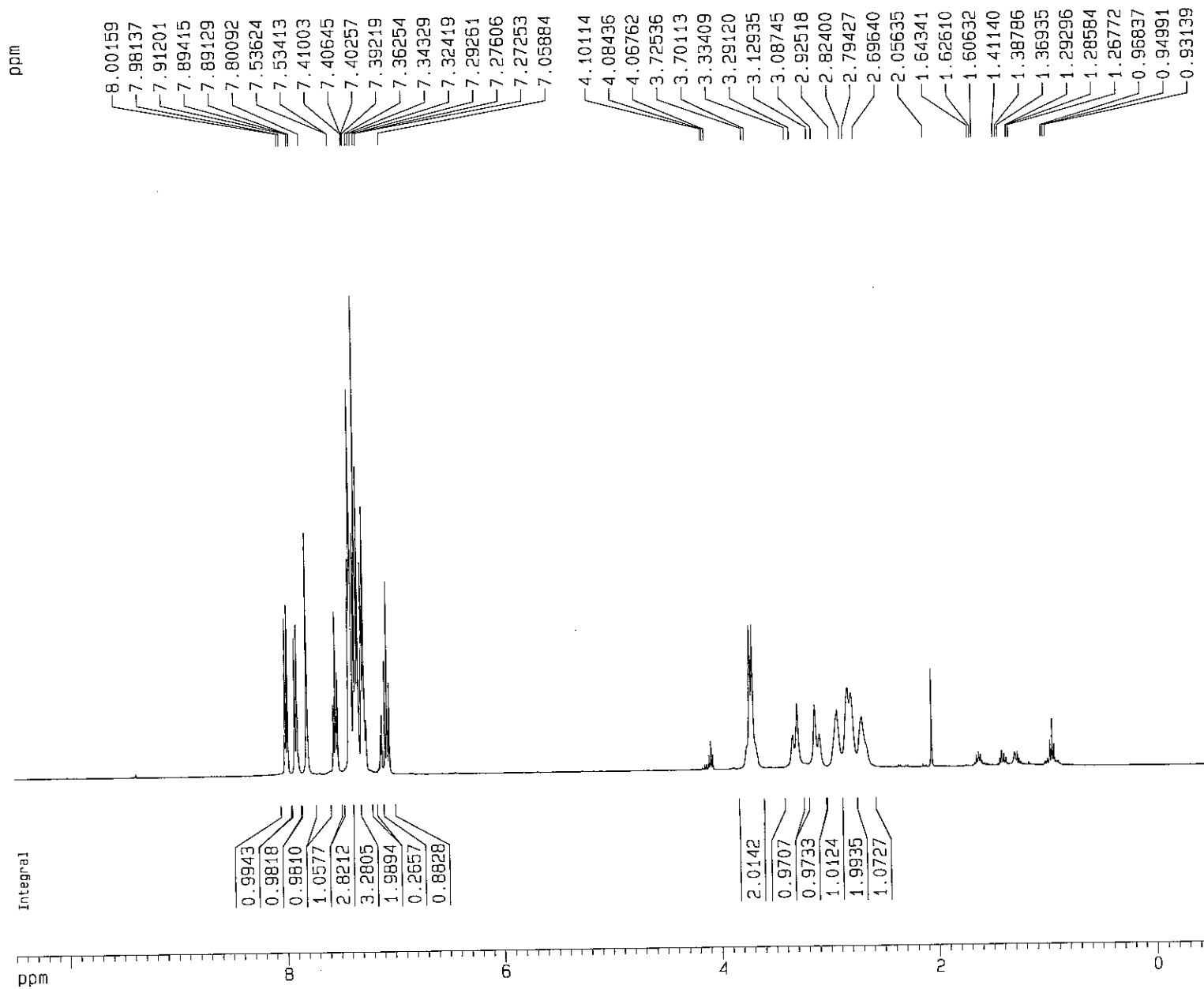
===== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 0.00 dB  
SF01 299.6718518 MHz

F2 - Processing parameters  
SI 32768  
SF 299.8700355 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

1D NMR plot parameters  
CX 20.00 cm  
F1P 10.000 ppm  
F1 2998.70 Hz  
F2P -1.000 ppm  
F2 -299.87 Hz  
PPMCM 0.55000 ppm/cm  
HZCM 164.92851 Hz/cm



23c



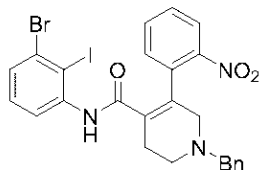
Current Data Parameters  
 NAME Jan16-SJH  
 EXPNO 9  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20080116  
 Time 22.35  
 INSTRUM spect  
 PROBHD 5 mm BBI 1H-B  
 PULPROG zg30  
 TD 49668  
 SOLVENT CDCl3  
 NS 4  
 DS 2  
 SWH 8278.146 Hz  
 FIDRES 0.166670 Hz  
 AQ 2.9999971 sec  
 RG 64  
 DW 60.400 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 1.00000000 sec

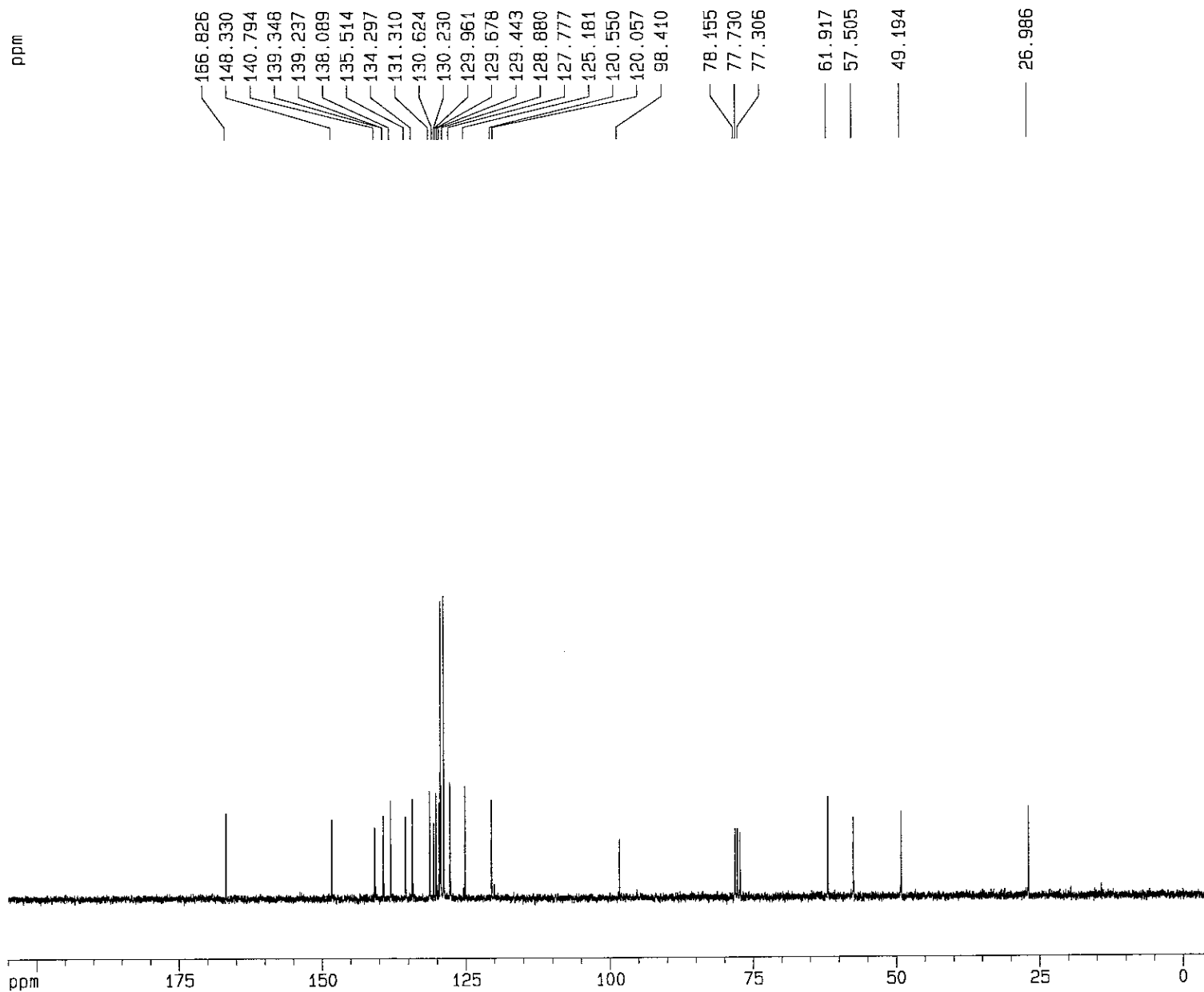
===== CHANNEL f1 =====  
 NUC1 1H  
 P1 6.45 usec  
 PL1 0.00 dB  
 SF01 400.1324710 MHz

F2 - Processing parameters  
 SI 32768  
 SF 400.1300054 MHz  
 WDW no  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 10.500 ppm  
 F1 4201.37 Hz  
 F2P -0.500 ppm  
 F2 -200.07 Hz  
 PPMCM 0.55000 ppm/cm  
 HZCM 220.07150 Hz/cm



23c



Current Data Parameters  
NAME Jan16-SJH  
EXPNO 1  
PROCNO 1

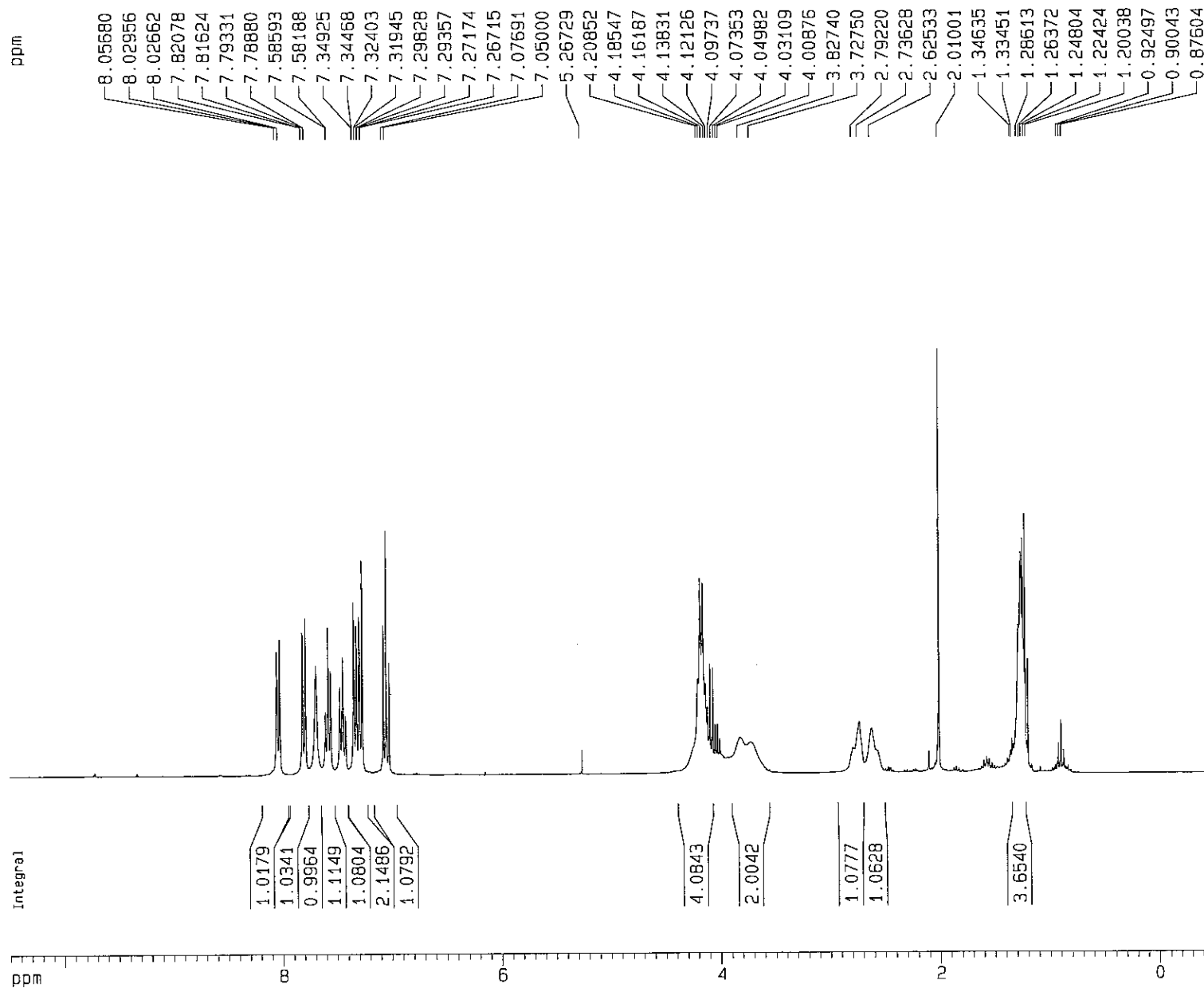
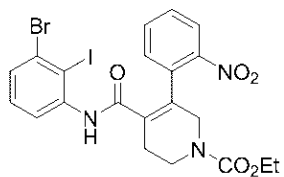
F2 - Acquisition Parameters  
Date\_ 20080116  
Time 23.34  
INSTRUM spect  
PROBHD 5 mm Multinu  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 28  
DS 2  
SWH 18832.393 Hz  
FIDRES 0.287360 Hz  
AQ 1.7400308 sec  
RG 16384  
DW 26.550 usec  
DE 6.00 usec  
TE 300.0 K  
D1 2.00000000 sec  
d11 0.03000000 sec  
d12 0.00002000 sec

===== CHANNEL f1 =====  
NUC1 13C  
P1 11.80 usec  
PL1 0.00 dB  
SF01 75.4760200 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 110.00 usec  
PL2 0.00 dB  
PL12 17.50 dB  
PL13 17.50 dB  
SF02 300.1312005 MHz

F2 - Processing parameters  
SI 32768  
SF 75.4677190 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

1D NMR plot parameters  
CX 20.00 cm  
F1P 205.000 ppm  
F1 15470.88 Hz  
F2P -5.000 ppm  
F2 -377.34 Hz  
PPMCM 10.50000 ppm/cm  
HZCM 792.41107 Hz/cm



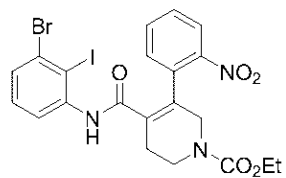
Current Data Parameters  
 NAME Mar02-SJH  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20080302  
 Time 10.34  
 INSTRUM spect  
 PROBHD 5 mm QNP 1H/1  
 PULPROG zg30  
 TD 37036  
 SOLVENT CDCl3  
 NS 6  
 DS 2  
 SWH 6172.839 Hz  
 FIDRES 0.166671 Hz  
 AQ 2.9999659 sec  
 RG 90.5  
 DW 81.000 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 1.00000000 sec

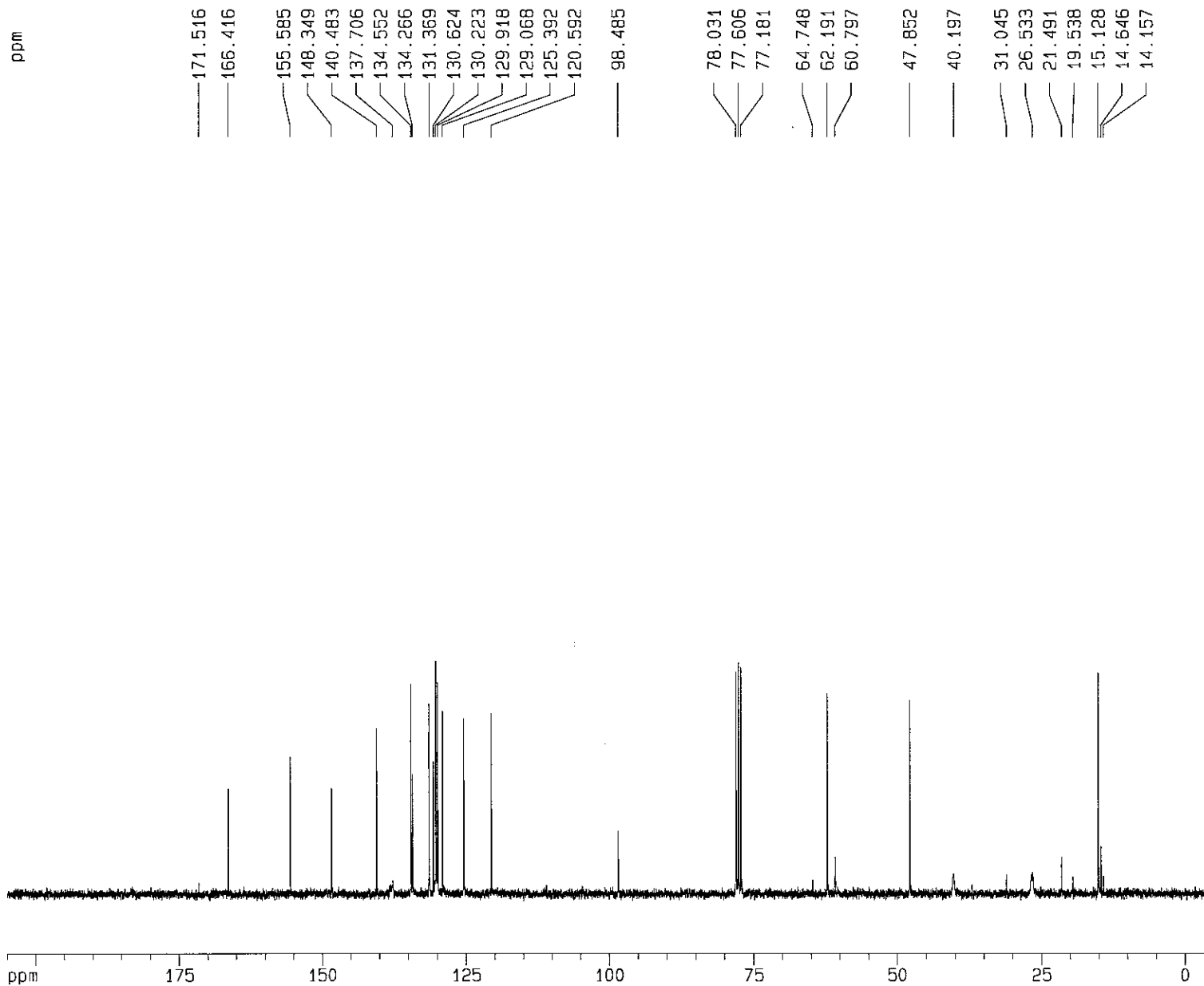
==== CHANNEL f1 =====  
 NUC1 1H  
 P1 11.70 usec  
 PL1 0.00 dB  
 SF01 299.8718518 MHz

F2 - Processing parameters  
 SI 32768  
 SF 299.8700067 MHz  
 WDW no  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 10.500 ppm  
 F1 3148.64 Hz  
 F2P -0.500 ppm  
 F2 -149.93 Hz  
 PPMCM 0.55000 ppm/cm  
 HZCM 164.92850 Hz/cm



24c



Current Data Parameters  
NAME Mar02-SUH  
EXPNO 2  
PROCNO 1

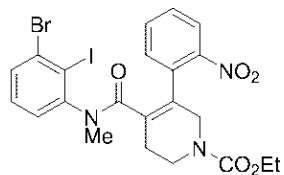
F2 - Acquisition Parameters  
Date\_ 20080302  
Time 10.36  
INSTRUM spect  
PROBHD 5 mm GNP 1H/1  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 130  
DS 2  
SWH 18796.992 Hz  
FIDRES 0.286819 Hz  
AQ 1.7433076 sec  
RG 512  
DW 26.600 usec  
DE 6.00 usec  
TE 300.0 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
D12 0.00002000 sec

===== CHANNEL f1 =====  
NUC1 13C  
P1 5.40 usec  
PL1 -6.00 dB  
SF01 75.4106357 MHz

===== CHANNEL f2 =====  
CPOPRG2 waltz16  
NUC2 1H  
PCPD2 115.00 usec  
PL2 0.00 dB  
PL12 20.00 dB  
PL13 20.00 dB  
SF02 299.8711995 MHz

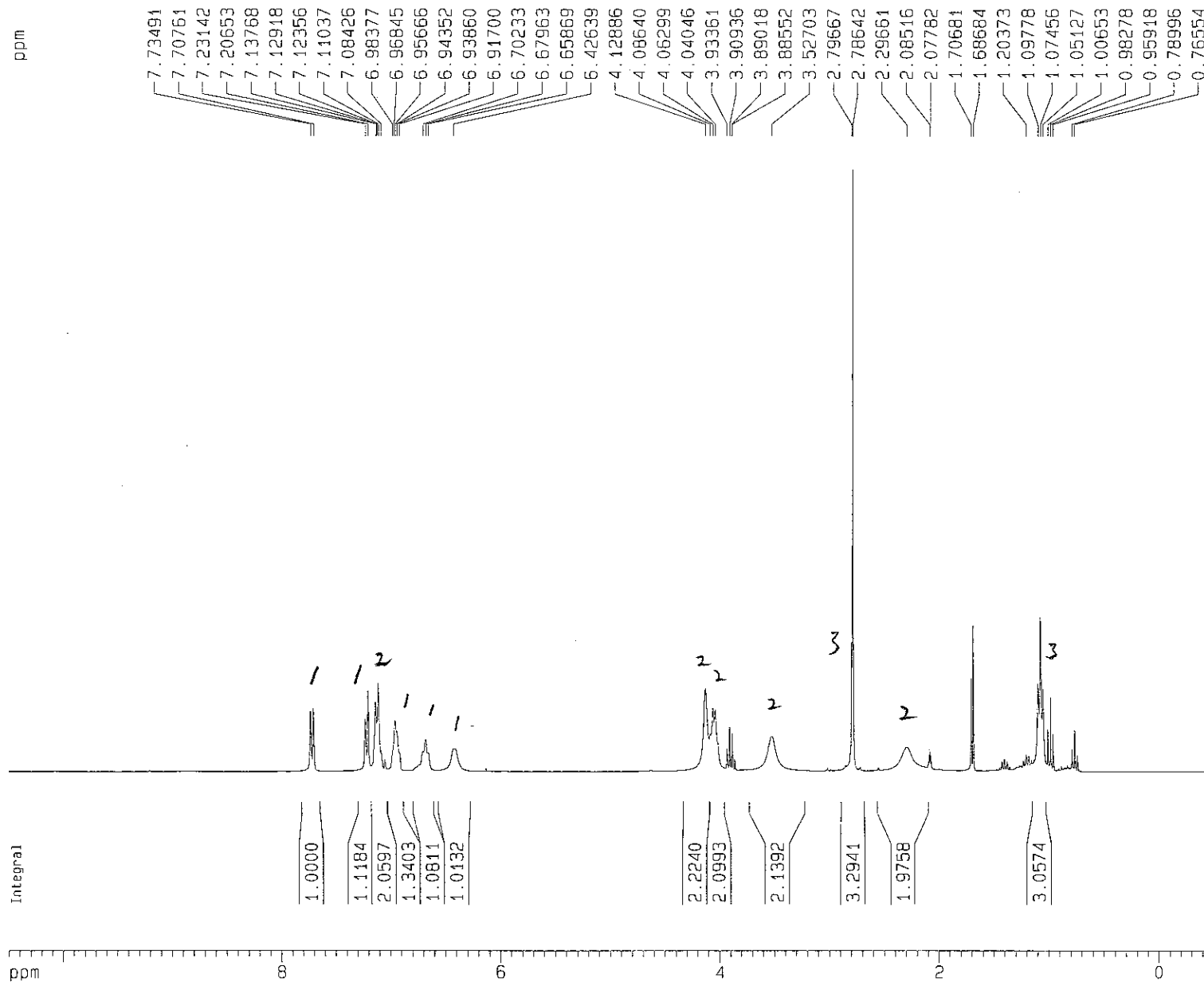
F2 - Processing parameters  
SI 32768  
SF 75.4023410 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

1D NMR plot parameters  
CX 20.00 cm  
F1P 205.000 ppm  
F1 15457.48 Hz  
F2P -5.000 ppm  
F2 -377.01 Hz  
PPMCM 10.50000 ppm/cm  
HZCM 791.72461 Hz/cm



in tol at 90 oC 1H

25c



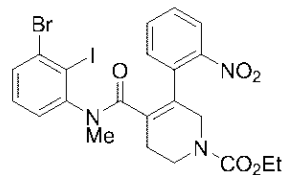
Current Data Parameters  
NAME Jan19-SJH  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20080119  
Time 10.26  
INSTRUM spect  
PROBHD 5 mm QNP 1H/1  
PULPROG zg30  
TD 37036  
SOLVENT Tol  
NS 16  
DS 2  
SWH 5172.839 Hz  
FIDRES 0.156671 Hz  
AQ 2.9999659 sec  
RG 114  
DW 81.000 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec

===== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 0.00 dB  
SFO1 299.8718518 MHz

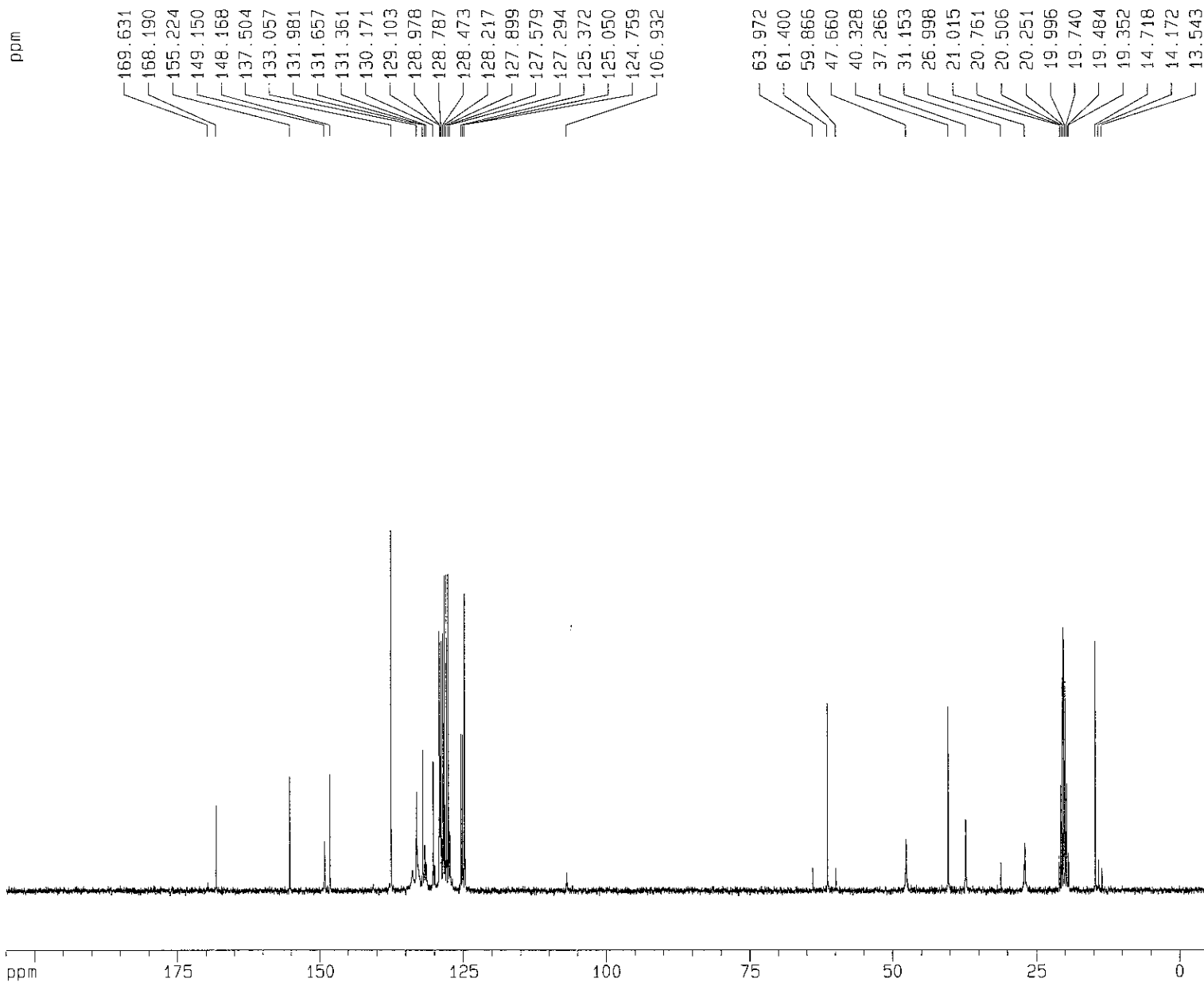
F2 - Processing parameters  
SI 32768  
SF 299.8700136 MHz  
WDW no  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

1D NMR plot parameters  
CX 20.00 cm  
F1P 10.500 ppm  
F1 3148.64 Hz  
F2P -0.500 ppm  
F2 -149.94 Hz  
PPMCM 0.55000 ppm/cm  
HZCM 164.92851 Hz/cm



in to1 at 90 oC 13C

25c



Current Data Parameters  
NAME Jan19-SJH  
EXPNO 3  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20080119  
Time 10.29  
INSTRUM spect  
PROBHD 5 mm GNP 1H/1  
PULPROG zgpg30  
TD 65536  
SOLVENT Tol  
NS 1121  
DS 2  
SWH 18796.992 Hz  
FIDRES 0.286819 Hz  
AQ 1.7433076 sec  
RG 8192  
DW 25.600 usec  
DE 6.00 usec  
TE 300.0 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
D12 0.00002000 sec

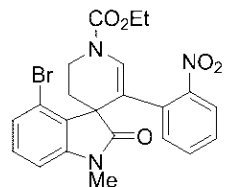
===== CHANNEL f1 =====  
NUC1 13C  
P1 5.40 usec  
PL1 -6.00 dB  
SFO1 75.4106357 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 115.00 usec  
PL2 0.00 dB  
PL12 20.00 dB  
PL13 20.00 dB  
SFO2 299.8711995 MHz

F2 - Processing parameters  
SI 32768  
SF 75.4023410 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

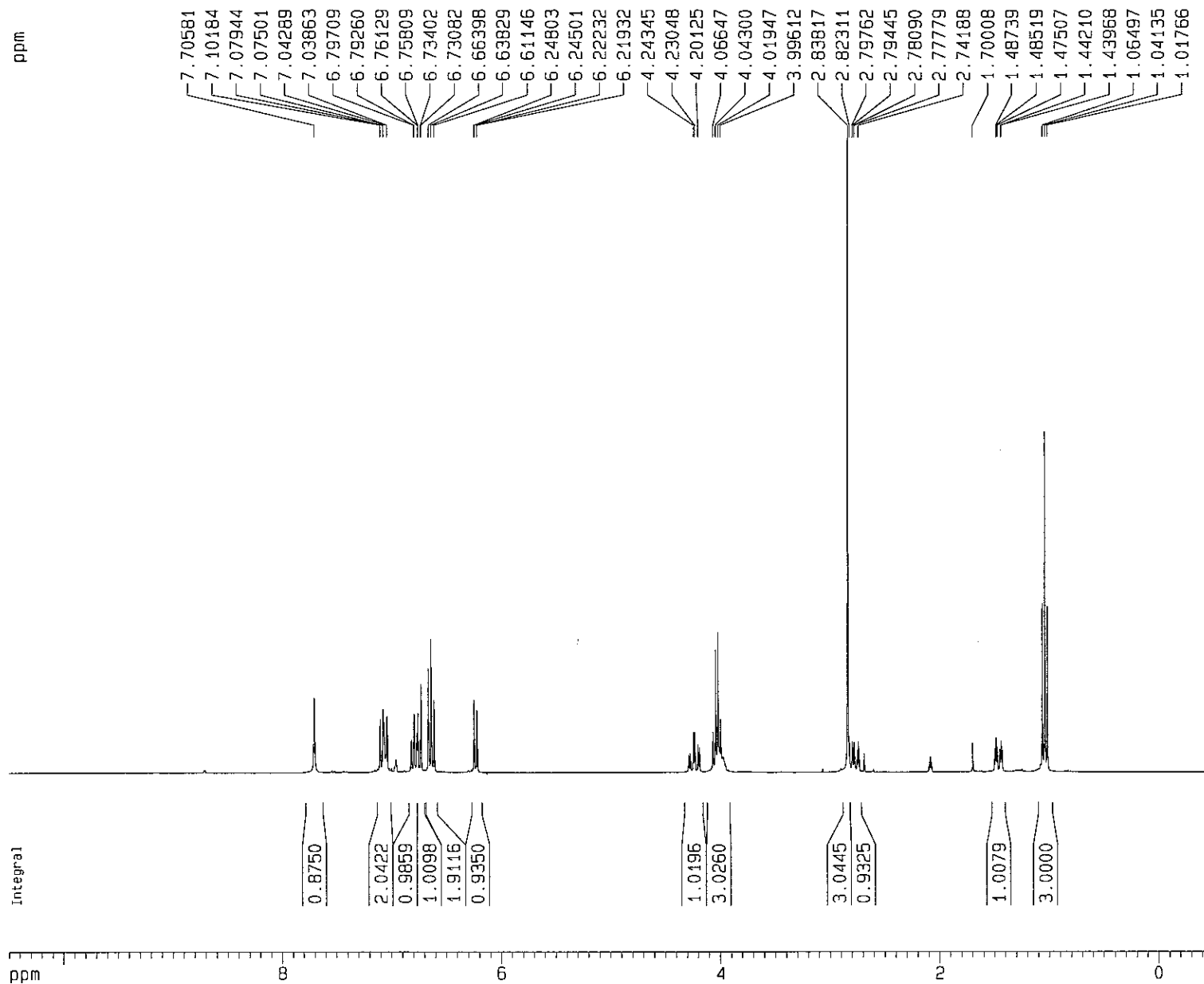
1D NMR plot parameters  
CX 20.00 cm  
F1P 205.000 ppm  
F1 15457.48 Hz  
F2P -5.000 ppm  
F2 -377.01 Hz  
PPMCM 10.50000 ppm/cm  
HZCM 791.72461 Hz/cm





in Tol at 90 °C 1H

26c



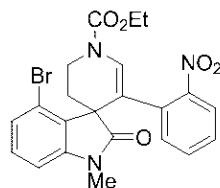
Current Data Parameters  
NAME Mar27-SJH  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20080327  
Time 8.24  
INSTRUM spect  
PROBHD 5 mm GNP 1H/1  
PULPROG zg30  
TD 37036  
SOLVENT Tol  
NS 8  
DS 2  
SWH 6172.839 Hz  
FIDRES 0.166671 Hz  
AQ 2.9999659 sec  
RG 57  
DW 81.000 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec

===== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 0.00 dB  
SF01 299.8718518 MHz

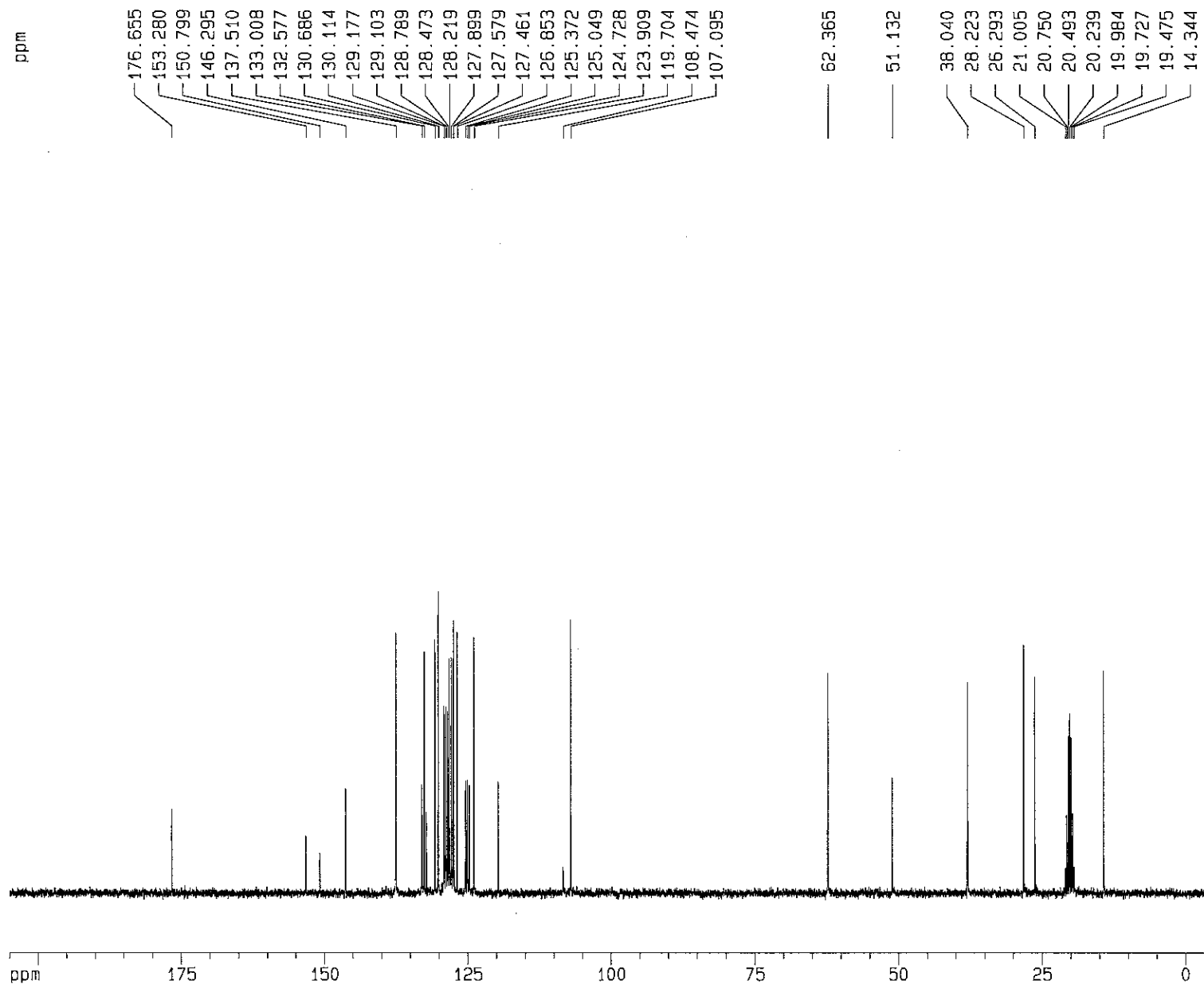
F2 - Processing parameters  
SI 32768  
SF 299.8700134 MHz  
WDW no  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

1D NMR plot parameters  
CX 20.00 cm  
F1P 10.500 ppm  
F1 3148.64 Hz  
F2P -0.500 ppm  
F2 -149.94 Hz  
PPMCM 0.55000 ppm/cm  
HZCM 164.92851 Hz/cm



in Tol at 90 oC 13C

26c



Current Data Parameters  
NAME Mar27-SJH  
EXPNO 3  
PROCNO 1

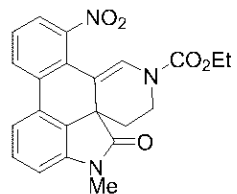
F2 - Acquisition Parameters  
Date\_ 20080327  
Time 8.34  
INSTRUM spect  
PROBHD 5 mm QNP 1H/1  
PULPROG zgpg30  
TD 65536  
SOLVENT Tol  
NS 234  
DS 2  
SWH 18796.992 Hz  
FIDRES 0.286819 Hz  
AQ 1.7433076 sec  
RG 4096  
DW 26.600 usec  
DE 6.00 usec  
TE 300.0 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
D12 0.00002000 sec

===== CHANNEL f1 =====  
NUC1 13C  
P1 5.40 usec  
PL1 -6.00 dB  
SF01 75.4106357 MHz

===== CHANNEL f2 =====  
CPOPRG2 waltz16  
NUC2 1H  
PCPD2 115.00 usec  
PL2 0.00 dB  
PL12 20.00 dB  
PL13 20.00 dB  
SF02 299.8711995 MHz

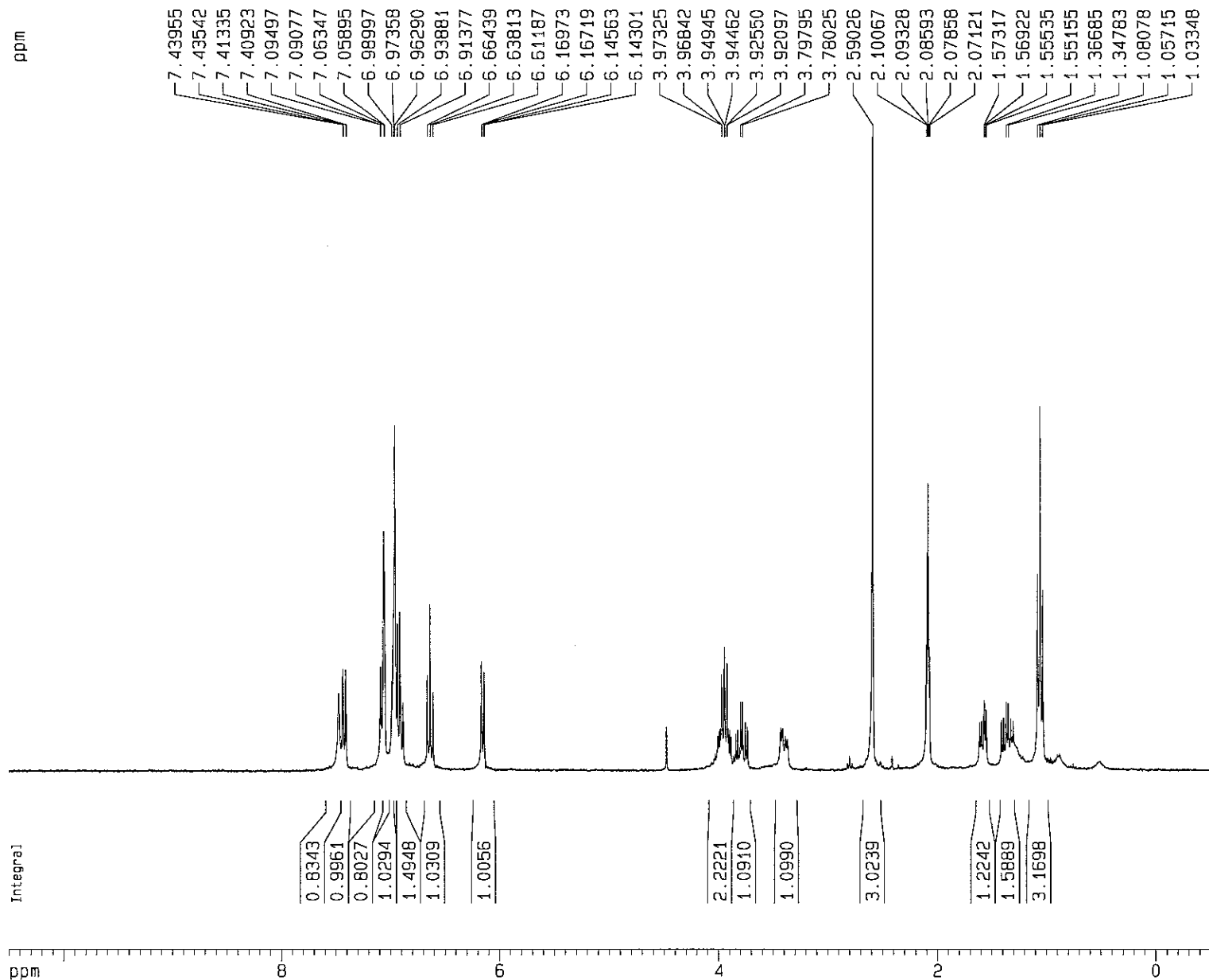
F2 - Processing parameters  
SI 32768  
SF 75.4023410 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

1D NMR plot parameters  
CX 20.00 cm  
F1P 205.000 ppm  
F1 15457.48 Hz  
F2P -5.000 ppm  
F2 -377.01 Hz  
PPMCM 10.50000 ppm/cm  
HZCM 791.72461 Hz/cm



in tol at 90 oC

27



Current Data Parameters  
NAME Mar27-SJH  
EXPNO 10  
PROCNO 1

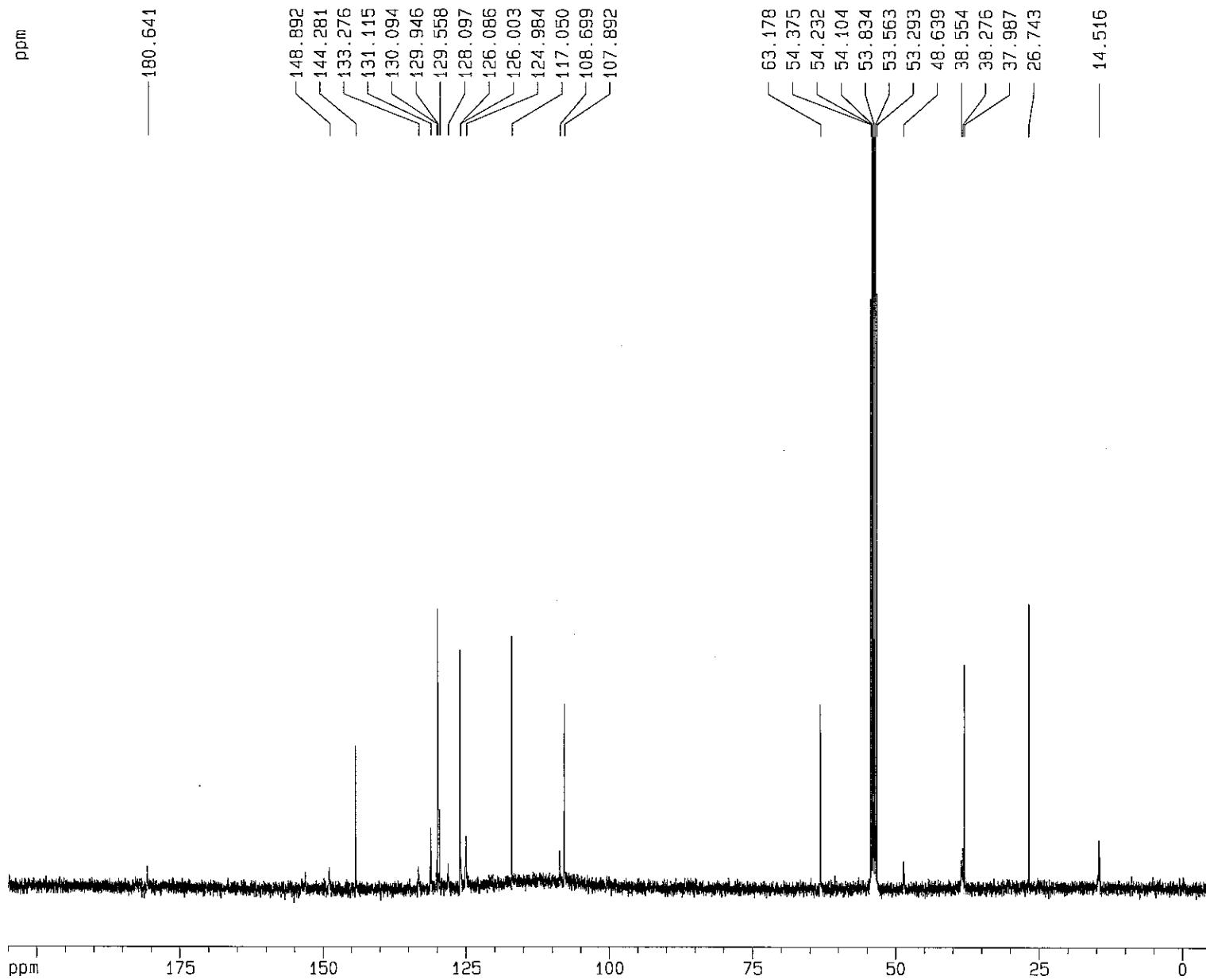
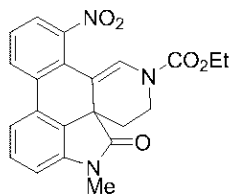
F2 - Acquisition Parameters  
Date\_ 20080327  
Time 15.32  
INSTRUM spect  
PROBHD 5 mm GNP 1H/1  
PULPROG zg30  
TD 37036  
SOLVENT Tol  
NS 4  
DS 2  
SWH 5172.839 Hz  
FIDRES 0.166671 Hz  
AQ 2.9999659 sec  
RG 645.1  
DW 81.000 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec

===== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 0.00 dB  
SFO1 299.8718518 MHz

F2 - Processing parameters  
SI 32768  
SF 299.8700134 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

1D NMR plot parameters  
CX 20.00 cm  
F1P 10.500 ppm  
F1 3148.64 Hz  
F2P -0.500 ppm  
F2 -149.94 Hz  
PPMCM 0.55000 ppm/cm  
HZCM 164.92851 Hz/cm

27



Current Data Parameters  
NAME May07-SJH  
EXPNO 2  
PROCNO 1

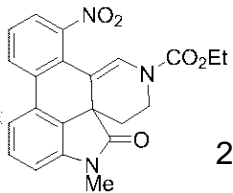
F2 - Acquisition Parameters  
Date\_ 20080507  
Time 21.43  
INSTRUM spect  
PROBHD 5 mm BBI 1H-8  
PULPROG zgpg30  
TD 65536  
SOLVENT CD2Cl2  
NS 3072  
DS 2  
SWH 25125.629 Hz  
FIDRES 0.383387 Hz  
AQ 1.3042164 sec  
RG 16384  
DW 19.900 usec  
DE 6.00 usec  
TE 300.0 K  
D1 2.0000000 sec  
d11 0.0300000 sec  
d12 0.0000200 sec

===== CHANNEL f1 =====  
NUC1 13C  
P1 15.35 usec  
PL1 -6.00 dB  
SF01 100.6237959 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 114.00 usec  
PL2 0.00 dB  
PL12 24.00 dB  
PL13 24.00 dB  
SF02 400.1316005 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127290 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

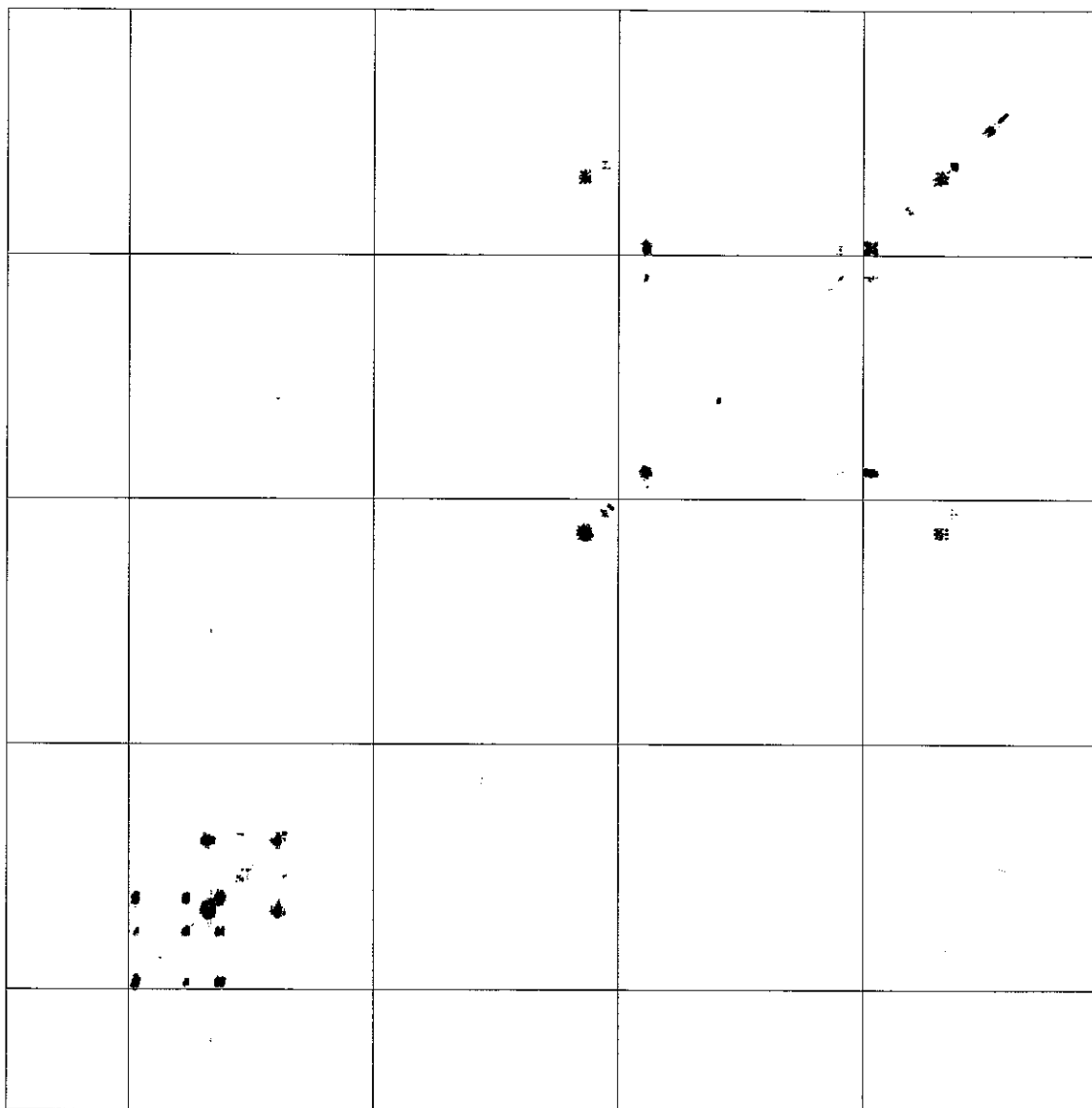
1D NMR plot parameters  
CX 20.00 cm  
F1P 205.000 ppm  
F1 20625.61 Hz  
F2P -5.000 ppm  
F2 -503.06 Hz  
PPMCM 10.50000 ppm/cm  
HZCM 1056.43372 Hz/cm



27



COSY



ppm 8 6 4 2

S141

Current Data Parameters

NAME May07-SJH  
EXPNO 6  
PROCNO 1

F2 - Acquisition Parameters

Date\_ 20080508  
Time 5.16  
INSTRUM spect  
PROBHD 5 mm BBI 1H-8  
PULPROG ajb.cosygsnftp  
TD 2048  
SOLVENT CDCl<sub>2</sub>  
NS 4  
DS 8  
SWH 3591.954 Hz  
FIDRES 1.753884 Hz  
AQ 0.2851316 sec  
RG 645  
DW 139.200 usec  
DE 6.00 usec  
TE 300.0 K  
D0 0.0000300 sec  
D1 4.0000000 sec  
d13 0.0000300 sec  
D16 0.0020000 sec  
d20 0.00120300 sec  
INO 0.00013884 sec

===== CHANNEL f1 =====

NUC1 <sup>1</sup>H  
P1 7.05 usec  
P2 14.10 usec  
PL1 0.00 dB  
SFO1 400.1318006 MHz

===== GRADIENT CHANNEL =====

P16 1000.00 usec

F1 - Acquisition parameters

ND0 2  
TD 650  
SFO1 400.1318 MHz  
FIDRES 5.540512 Hz  
SW 9.000 ppm

F2 - Processing parameters

SI 2048  
SF 400.1300000 MHz  
WDW SINE  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.40

F1 - Processing parameters

SI 1024  
MC2 TPPI  
SF 400.1300000 MHz  
WDW SINE  
SSB 0  
LB 0.00 Hz  
GB 0

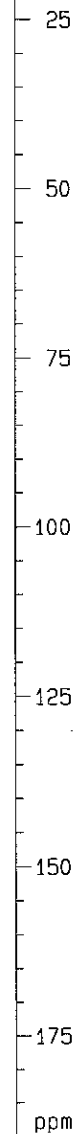
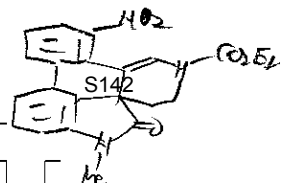
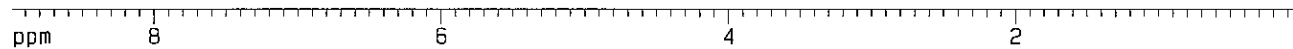
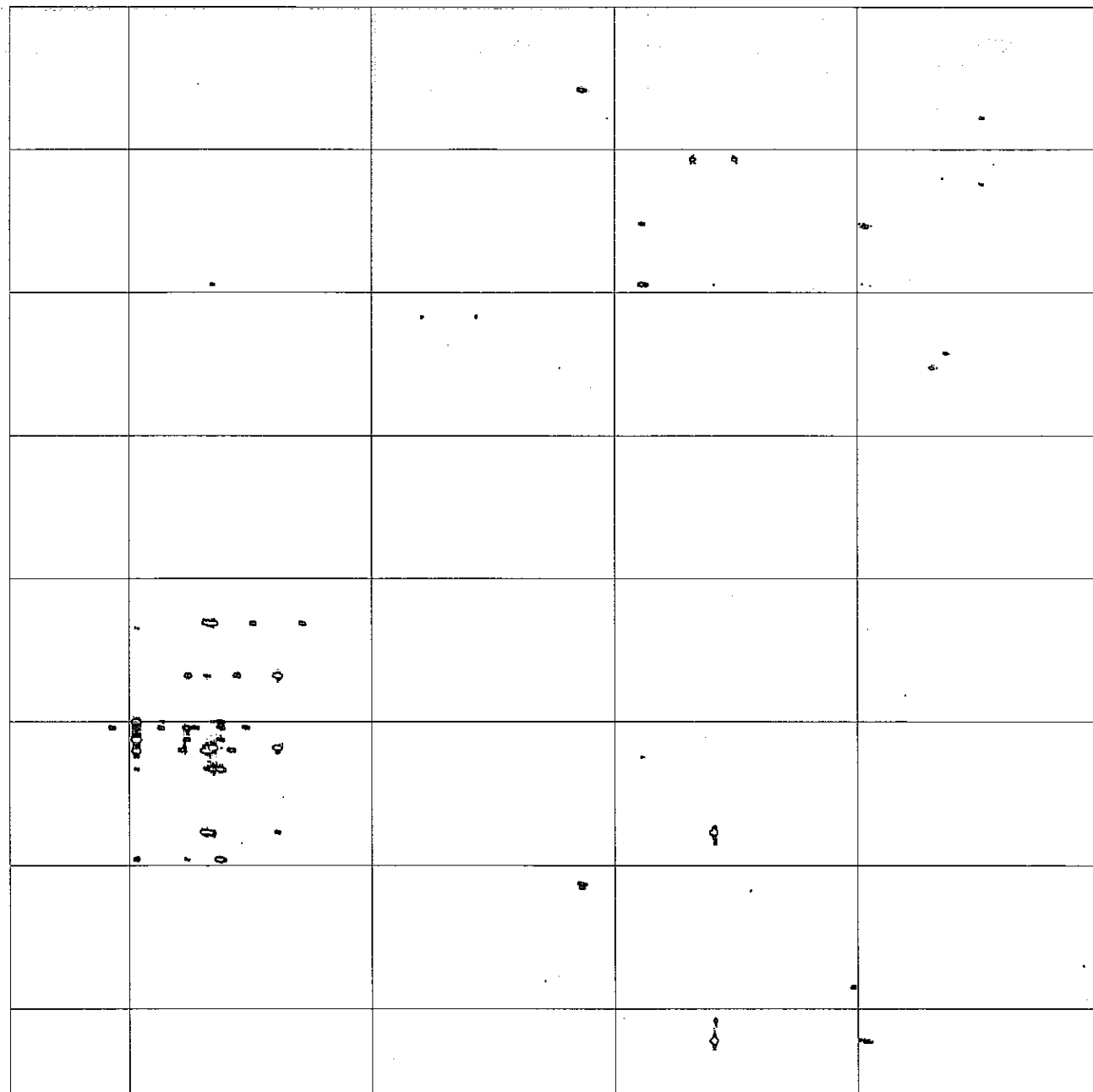
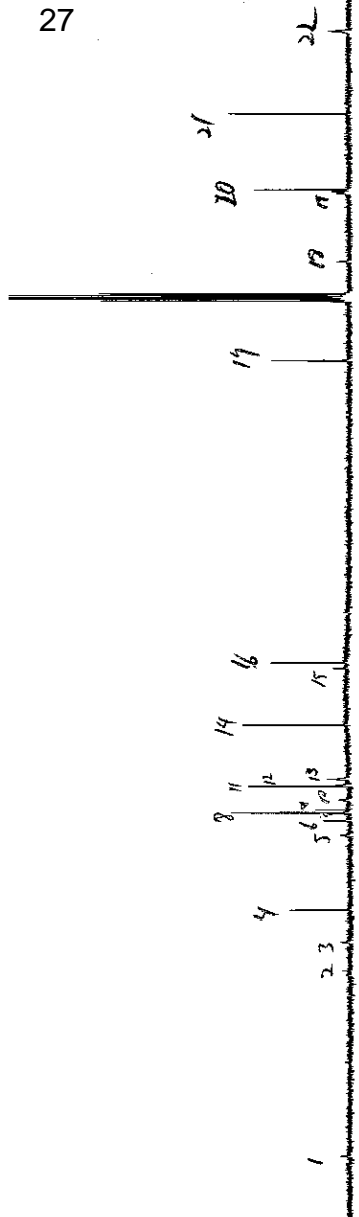
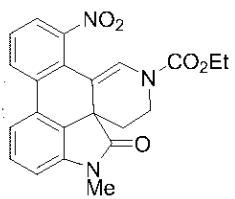
2D NMR plot parameters

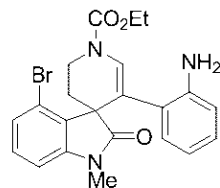
CX2 15.00 cm  
CX1 15.00 cm  
F2PLO 9.000 ppm  
F2LO 3601.17 Hz  
F2PHI 0.000 ppm  
F2HI 0.00 Hz  
F1PLO 9.000 ppm  
F1LO 3601.17 Hz  
F1PHI 0.000 ppm  
F1HI 0.00 Hz  
F2PPMCM 0.60000 ppm/cm  
F2HZCM 240.07800 Hz/cm  
F1PPMCM 0.60000 ppm/cm  
F1HZCM 240.07800 Hz/cm

2  
4  
6  
8  
ppm

HMBC

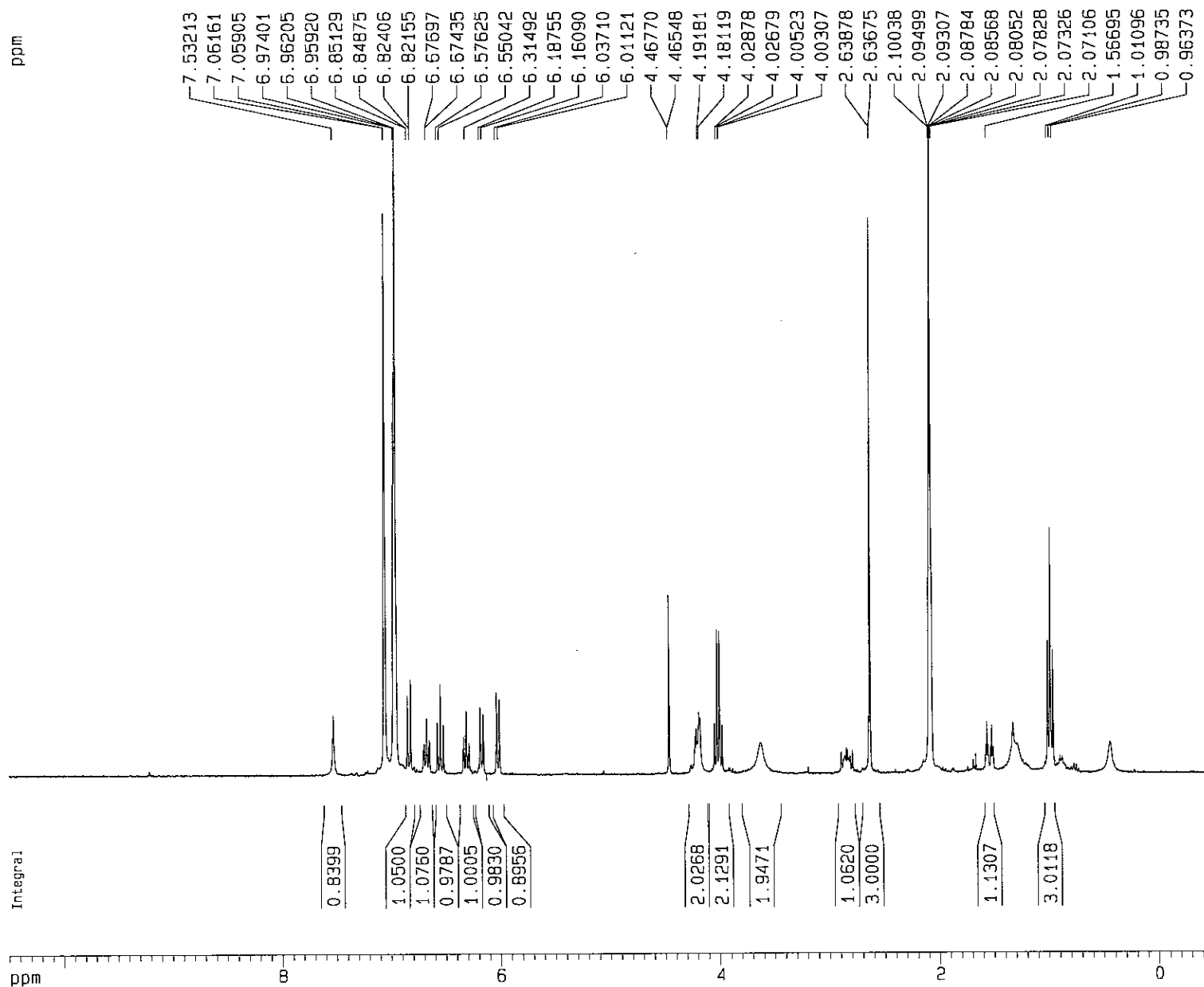
27





in tol at 90 oC

28



## Current Data Parameters

NAME Mar27-SJH  
EXPNO 6  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20080327  
Time 14.58  
INSTRUM spect  
PROBHD 5 mm QNP 1H/1  
PULPROG zg30  
TD 37036  
SOLVENT Tol  
NS 31  
DS 2  
SWH 6172.839 Hz  
FIDRES 0.166671 Hz  
AQ 2.9999659 sec  
RG 812.7  
DW 81.000 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec

## ===== CHANNEL f1 =====

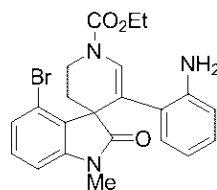
NUC1 1H  
P1 11.70 usec  
PL1 0.00 dB  
SF01 299.8718518 MHz

## F2 - Processing parameters

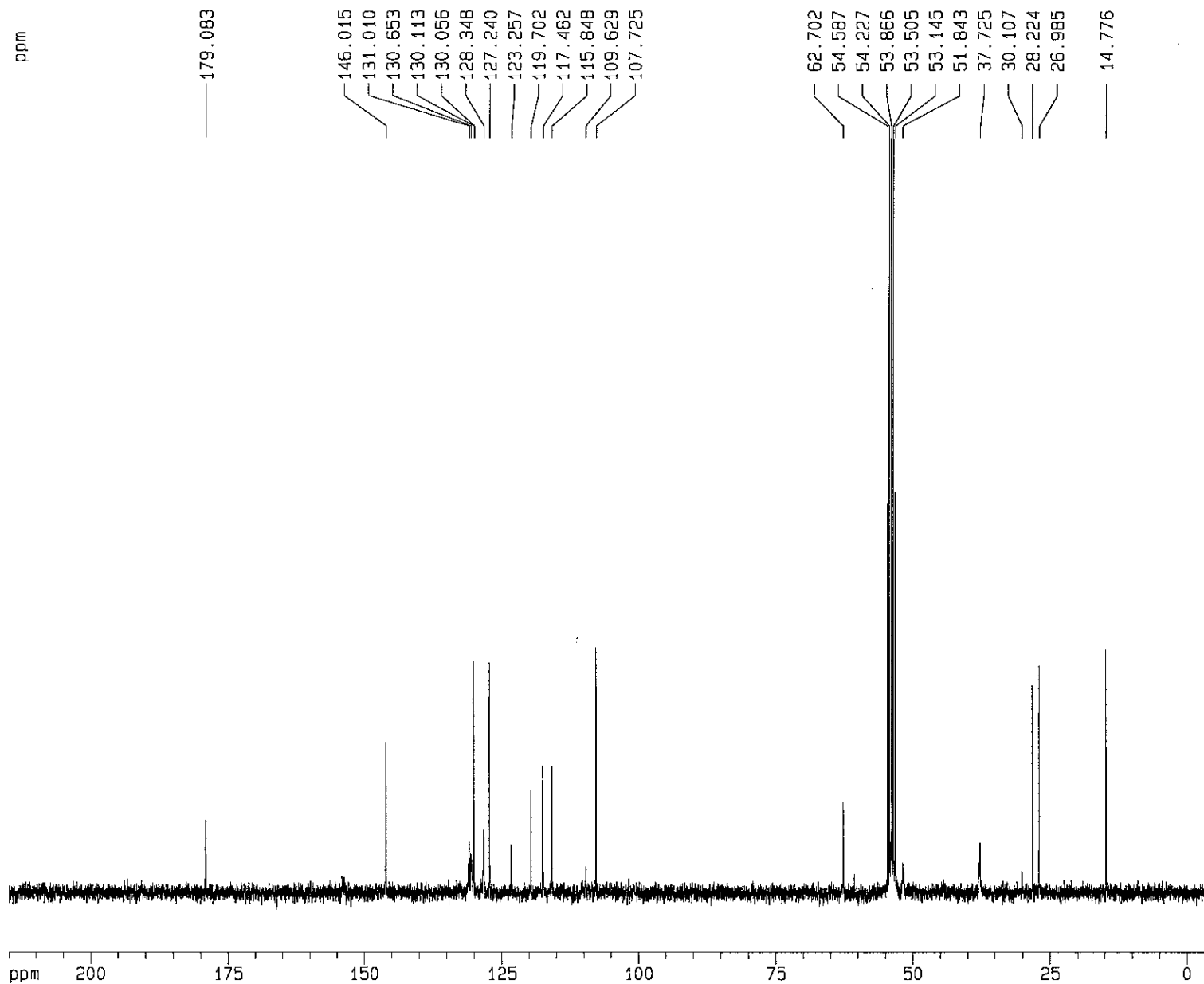
SI 32768  
SF 299.8700134 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

## 1D NMR plot parameters

CX 20.00 cm  
F1P 10.500 ppm  
F1 3148.64 Hz  
F2P -0.500 ppm  
F2 -149.94 Hz  
PPMCM 0.55000 ppm/cm  
HZCM 164.92851 Hz/cm



28



Current Data Parameters  
NAME Apr04-SJH  
EXPNO 10  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20080403  
Time 21.18  
INSTRUM spect  
PROBHD 5 mm GNP 1H/1  
PULPROG zgpg30  
TD 65536  
SOLVENT CD2Cl2  
NS 1008  
DS 2  
SWH 18795.992 Hz  
FIDRES 0.286819 Hz  
AQ 1.7433076 sec  
RG 1024  
DW 25.600 usec  
DE 6.00 usec  
TE 300.0 K  
D1 2.0000000 sec  
D11 0.0300000 sec  
D12 0.00002000 sec

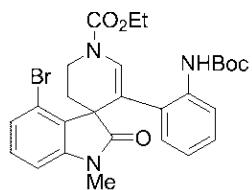
===== CHANNEL f1 =====  
NUC1 13C  
P1 5.40 usec  
PL1 -6.00 dB  
SF01 75.4106357 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 115.00 usec  
PL2 0.00 dB  
PL12 20.00 dB  
PL13 20.00 dB  
SF02 299.8711995 MHz

F2 - Processing parameters  
SI 32768  
SF 75.4023410 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

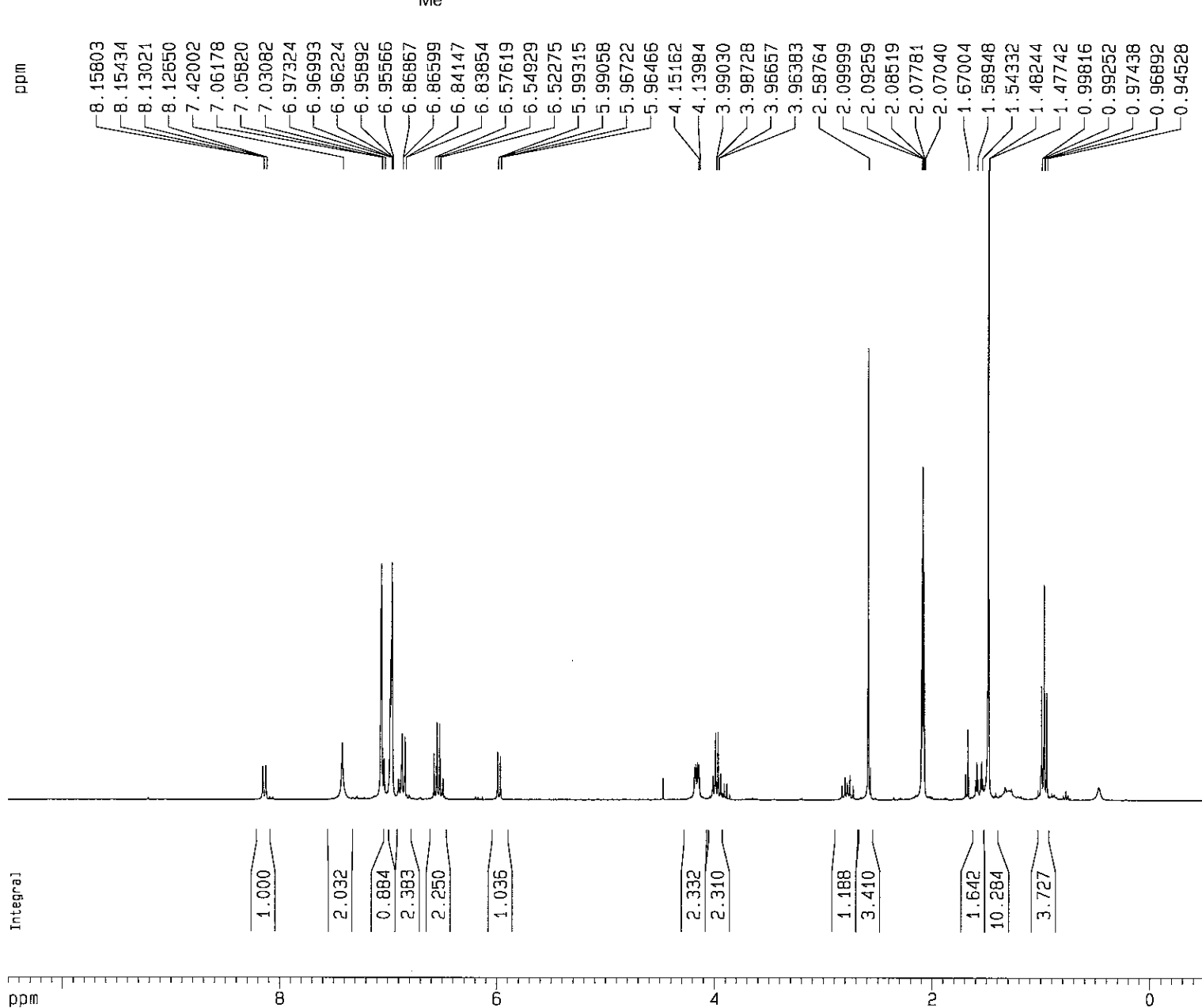
1D NMR plot parameters  
CX 20.00 cm  
F1P 215.000 ppm  
F1 16211.50 Hz  
F2P -5.000 ppm  
F2 -377.01 Hz  
PPMCM 11.00000 ppm/cm  
HZCM 829.42578 Hz/cm





in tol at 90 DC 1H

29c



## Current Data Parameters

NAME Apr01-SJH  
EXPNO 2  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20080401  
Time 8.23  
INSTRUM spect  
PROBHD 5 mm QNP 1H/1  
PULPROG zg30  
TD 37036  
SOLVENT Tol  
NS 11  
DS 2  
SWH 6172.839 Hz  
FIDRES 0.166671 Hz  
AQ 2.9999659 sec  
RG 512  
DW 81.000 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec

## ===== CHANNEL f1 =====

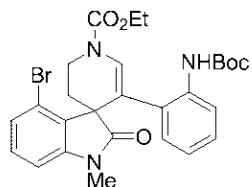
NUC1 1H  
P1 11.70 usec  
PL1 0.00 dB  
SF01 299.8718518 MHz

## F2 - Processing parameters

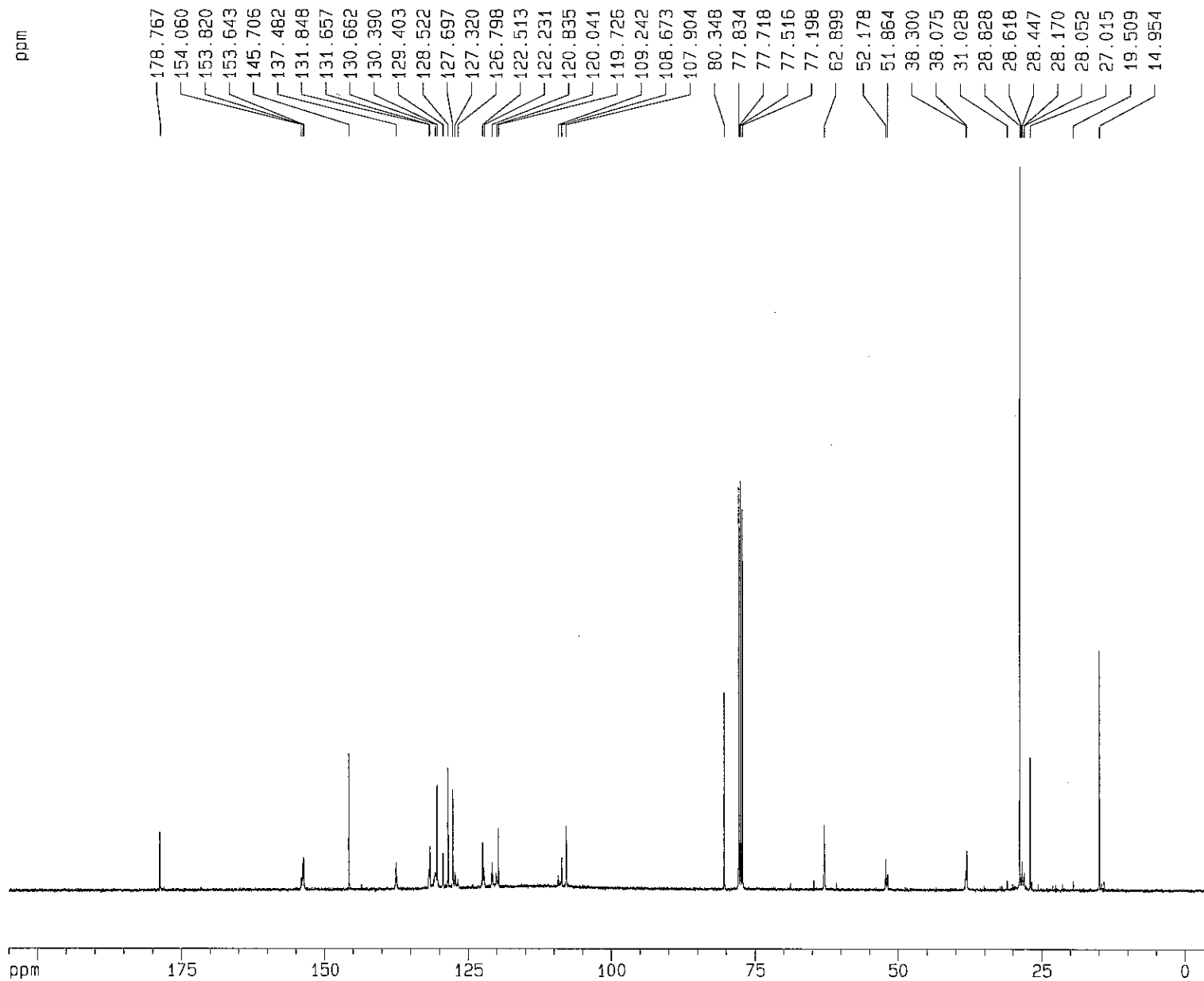
SI 32768  
SF 299.8700136 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

## 1D NMR plot parameters

CX 20.00 cm  
F1P 10.500 ppm  
F1 3148.64 Hz  
F2P -0.500 ppm  
F2 -149.94 Hz  
PPMCM 0.55000 ppm/cm  
HZCM 164.92851 Hz/cm



29c



Current Data Parameters  
NAME May08-SJH  
EXPNO 4  
PROCNO 1

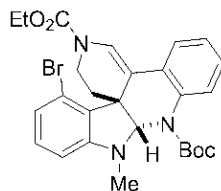
F2 - Acquisition Parameters  
Date\_ 20080509  
Time 22.16  
INSTRUM spect  
PROBHD 5 mm BBI 1H-B  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 8192  
DS 2  
SWH 25125.629 Hz  
FIDRES 0.383387 Hz  
AQ 1.3042164 sec  
RG 16384  
DW 19.900 usec  
DE 6.00 usec  
TE 300.0 K  
D1 2.00000000 sec  
d11 0.03000000 sec  
d12 0.00002000 sec

===== CHANNEL f1 =====  
NUC1 13C  
P1 16.35 usec  
PL1 -6.00 dB  
SF01 100.6237959 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 114.00 usec  
PL2 0.00 dB  
PL12 24.00 dB  
PL13 24.00 dB  
SF02 400.1316005 MHz

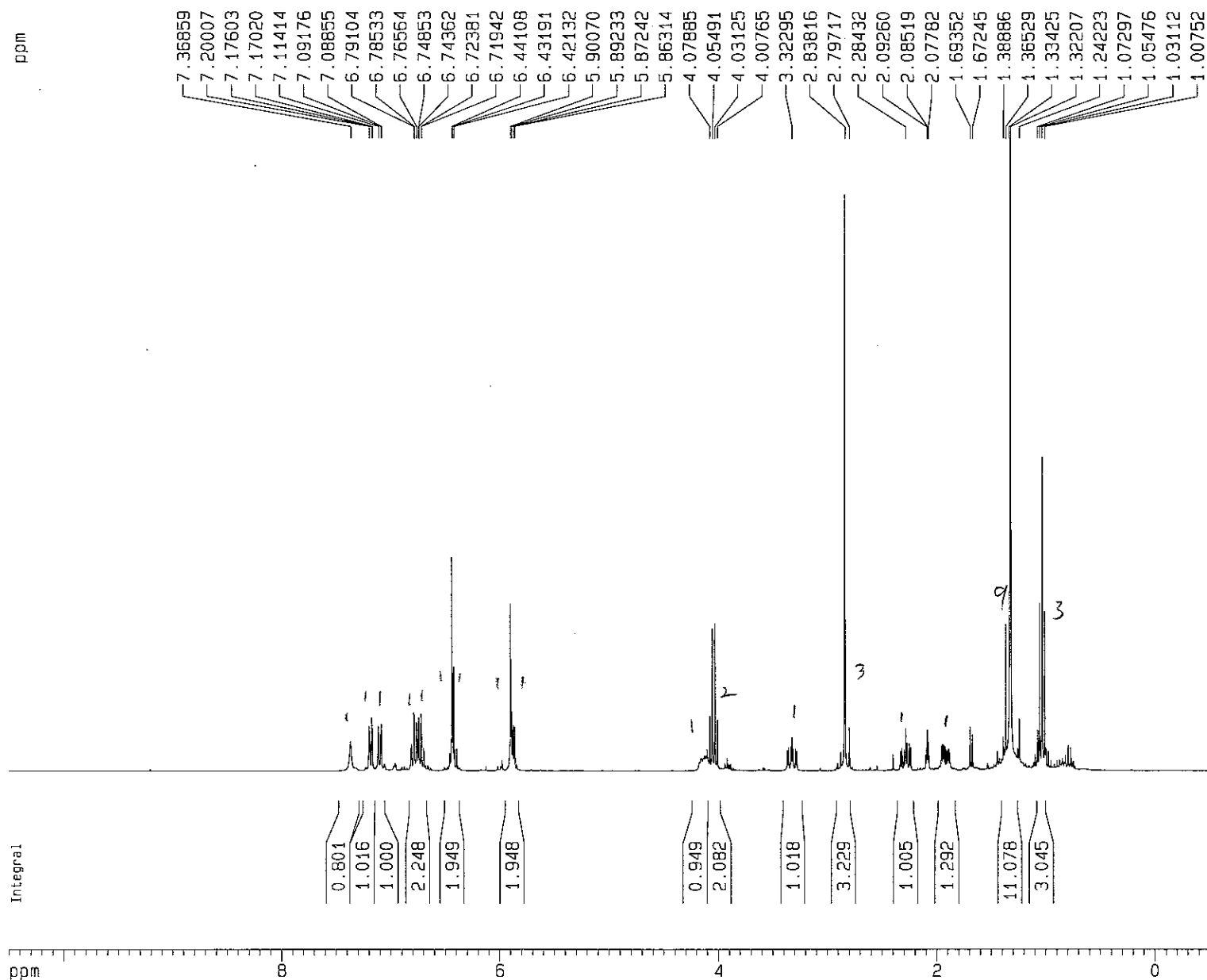
F2 - Processing parameters  
SI 32768  
SF 100.6127290 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

1D NMR plot parameters  
CX 20.00 cm  
F1P 205.000 ppm  
F1 20625.61 Hz  
F2P -5.000 ppm  
F2 -503.06 Hz  
PPMCM 10.50000 ppm/cm  
HZCM 1056.43372 Hz/cm



in tol at 90 oC 1H

30c



## Current Data Parameters

NAME Apr04-SJH  
EXPNO 3  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20080403  
Time 20.32  
INSTRUM spect  
PROBHD 5 mm GNP 1H/1  
PULPROG zg30  
TD 37036  
SOLVENT Tol  
NS 19  
DS 2  
SWH 6172.839 Hz  
FIDRES 0.166671 Hz  
AQ 2.9999659 sec  
RG 161.3  
DW 81.000 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec

## ===== CHANNEL f1 =====

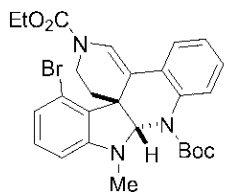
NUC1 1H  
P1 11.70 usec  
PL1 0.00 dB  
SF01 299.8718518 MHz

## F2 - Processing parameters

SI 32768  
SF 299.8700136 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

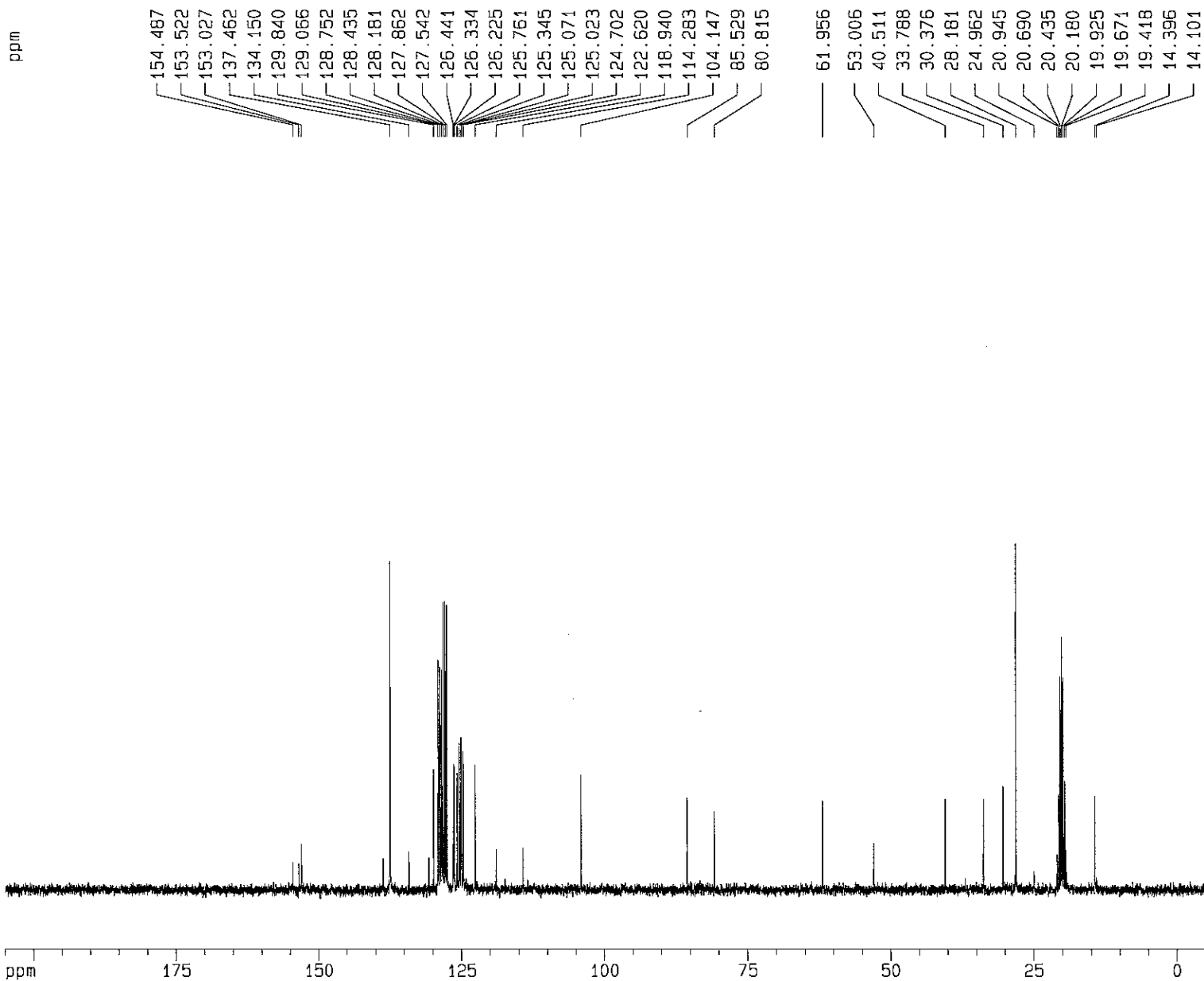
## 1D NMR plot parameters

CX 20.00 cm  
F1P 10.500 ppm  
F1 3148.64 Hz  
F2P -0.500 ppm  
F2 -149.94 Hz  
PPMCM 0.55000 ppm/cm  
HZCM 164.92851 Hz/cm



in tol at 90 oC 13C

30c



Current Data Parameters  
NAME Apr04-SJH  
EXPNO 4  
PROCNO 1

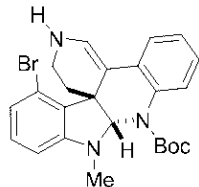
F2 - Acquisition Parameters  
Date\_ 20080403  
Time 20.36  
INSTRUM spect  
PROBHD 5 mm GNP 1H/1  
PULPROG zgpg30  
TD 65536  
SOLVENT Tol  
NS 251  
DS 2  
SWH 18795.992 Hz  
FIDRES 0.286819 Hz  
AQ 1.7433076 sec  
RG 4096  
DW 25.600 usec  
DE 6.00 usec  
TE 300.0 K  
D1 2.0000000 sec  
D11 0.0300000 sec  
D12 0.00002000 sec

===== CHANNEL f1 =====  
NUC1 13C  
P1 5.40 usec  
PL1 -6.00 dB  
SF01 75.4106357 MHz

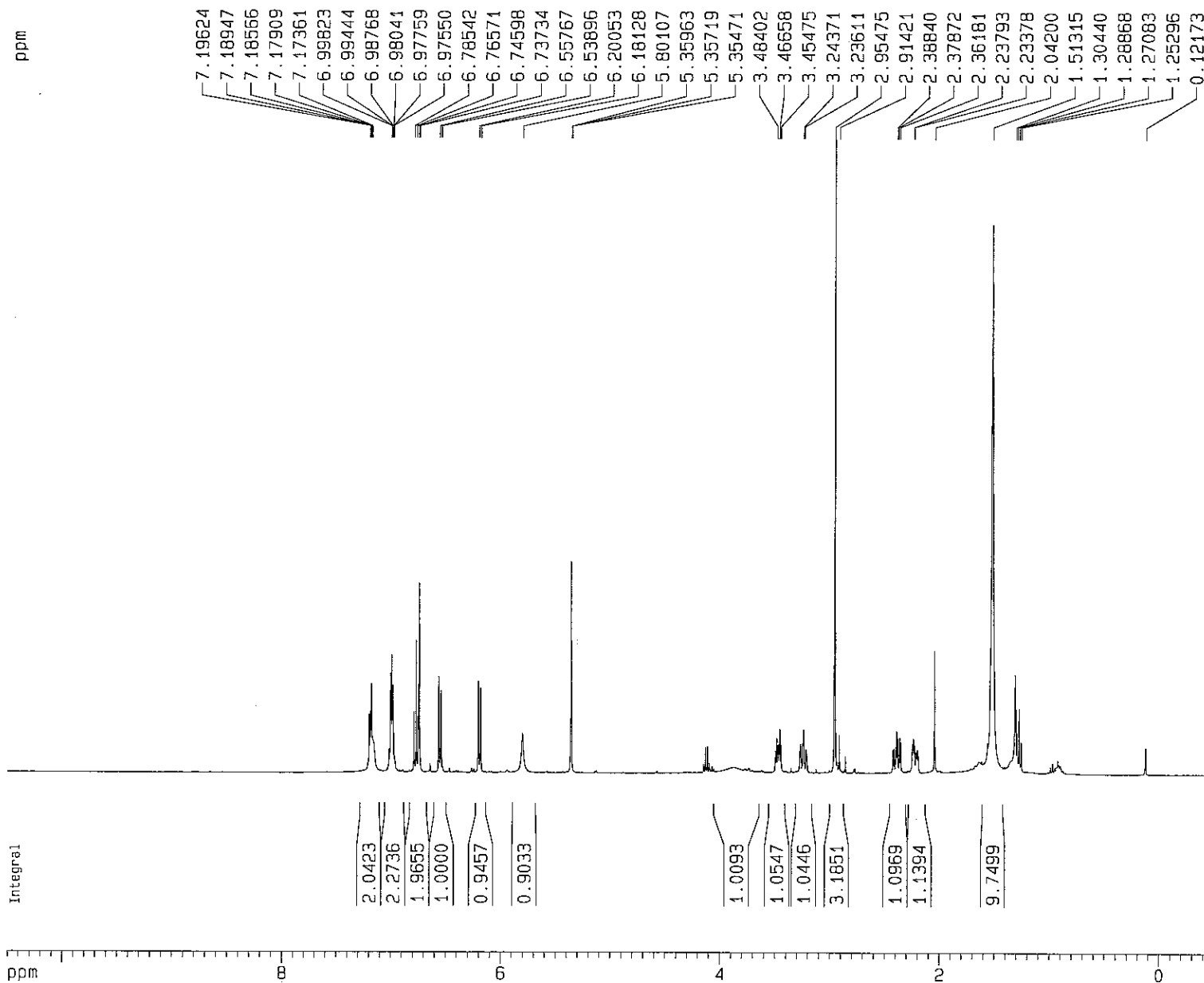
===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 115.00 usec  
PL2 0.00 dB  
PL12 20.00 dB  
PL13 20.00 dB  
SF02 299.8711995 MHz

F2 - Processing parameters  
SI 32768  
SF 75.4023410 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

1D NMR plot parameters  
CX 20.00 cm  
F1P 205.000 ppm  
F1 15457.48 Hz  
F2P -5.000 ppm  
F2 -377.01 Hz  
PPMCM 10.50000 ppm/cm  
HZCM 791.72461 Hz/cm

Crude in CD<sub>2</sub>C<sub>12</sub>

40



## Current Data Parameters

NAME Apr8-SJH  
EXPNO 1  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20080408  
Time 9.01  
INSTRUM spect  
PROBHD 5 mm BBI 1H-B  
PULPROG zg30  
TD 49668  
SOLVENT CD2C12  
NS 16  
DS 2  
SWH 8278.146 Hz  
FIDRES 0.166670 Hz  
AQ 2.9999971 sec  
RG 287.4  
DW 60.400 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec

## ===== CHANNEL f1 =====

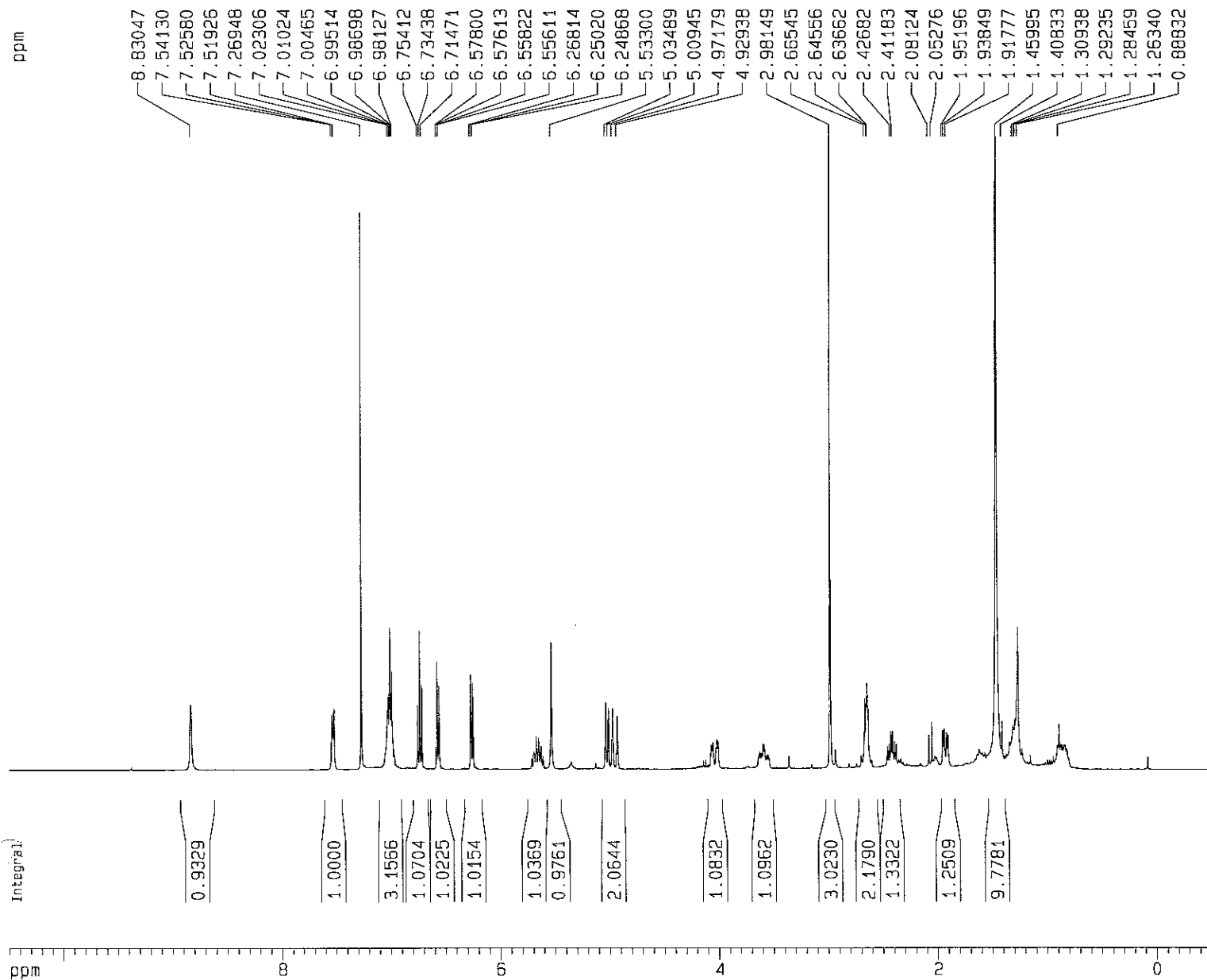
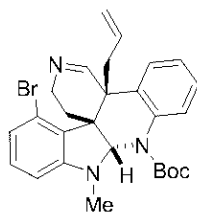
NUC1 1H  
P1 6.45 usec  
PL1 0.00 dB  
SFO1 400.1324710 MHz

## F2 - Processing parameters

SI 32768  
SF 400.1300000 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

## 1D NMR plot parameters

CX 20.00 cm  
F1P 10.500 ppm  
F1 4201.37 Hz  
F2P -0.500 ppm  
F2 -200.07 Hz  
PPMCM 0.55000 ppm/cm  
HZCM 220.07150 Hz/cm



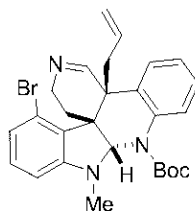
Current Data Parameters  
 NAME Apr25-SJH  
 EXPNO 5  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20080425  
 Time 10.08  
 INSTRUM spect  
 PROBHD 5 mm BBI 1H-8  
 PULPROG zg30  
 TD 49668  
 SOLVENT CDCl3  
 NS 21  
 DS 2  
 SWH 8278.146 Hz  
 FIDRES 0.166670 Hz  
 AQ 2.9999971 sec  
 RG 322.5  
 DW 60.400 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 1.00000000 sec

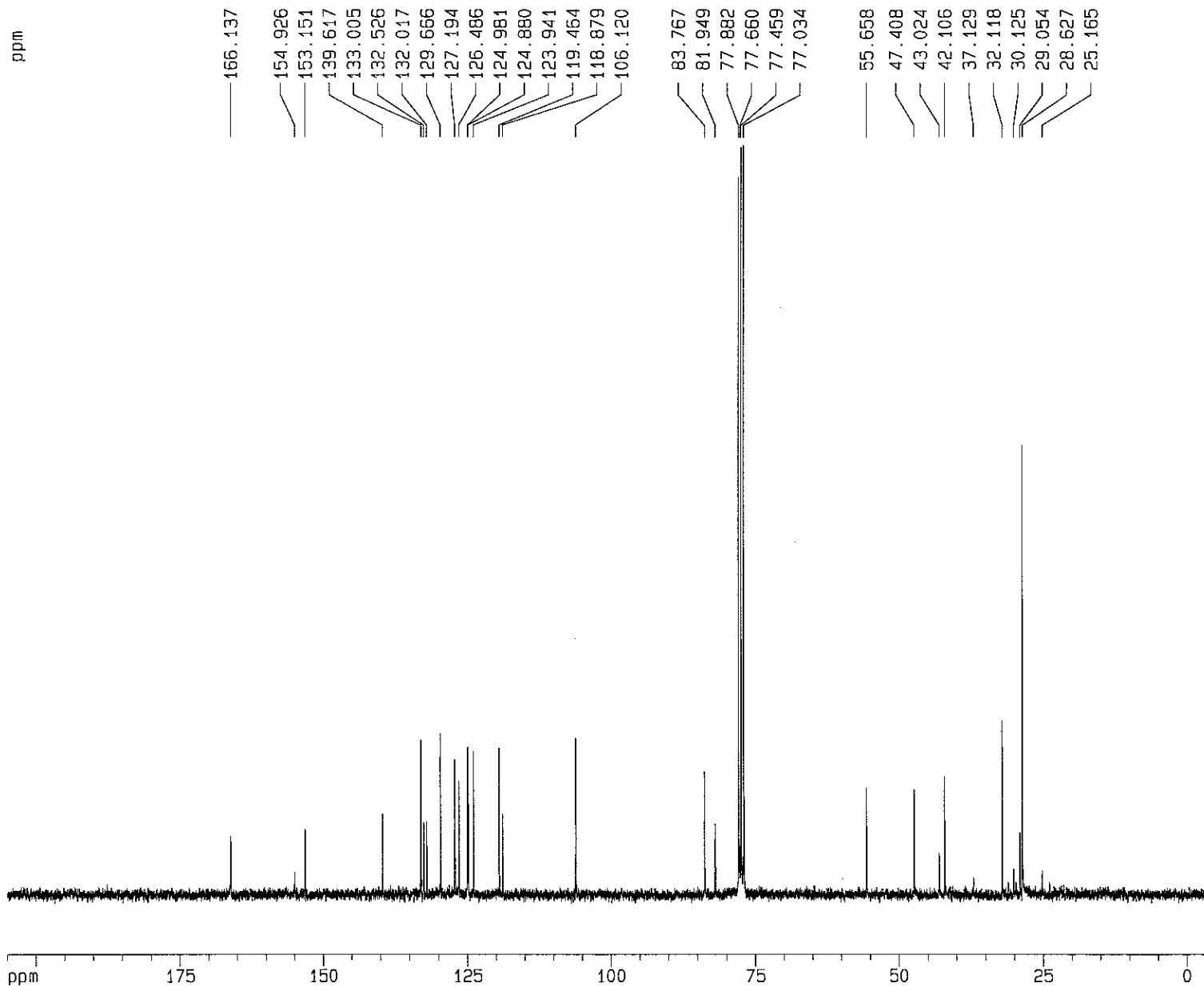
===== CHANNEL f1 =====  
 NUC1 1H  
 P1 6.45 usec  
 PL1 0.00 dB  
 SF01 400.1324710 MHz

F2 - Processing parameters  
 SI 32768  
 SF 400.1300051 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 10.500 ppm  
 F1 4201.37 Hz  
 F2P -0.500 ppm  
 F2 -200.07 Hz  
 PPMCM 0.55000 ppm/cm  
 HZCM 220.07150 Hz/cm



41



Current Data Parameters  
NAME May22-SJH  
EXPNO 4  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20080522  
Time 14.46  
INSTRUM spect  
PROBHD 5 mm QNP 1H/1  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 1126  
DS 2  
SWH 18796.992 Hz  
FIDRES 0.286819 Hz  
AQ 1.7433076 sec  
RG 512  
DW 25.600 usec  
DE 6.00 usec  
TE 300.0 K  
D1 2.0000000 sec  
D11 0.0300000 sec  
D12 0.00002000 sec

===== CHANNEL f1 =====  
NUC1 13C  
P1 5.40 usec  
PL1 -6.00 dB  
SF01 75.4106357 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 115.00 usec  
PL2 0.00 dB  
PL12 20.00 dB  
PL13 20.00 dB  
SF02 299.8711995 MHz

F2 - Processing parameters  
SI 32768  
SF 75.4023410 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

1D NMR plot parameters  
CX 20.00 cm  
F1P 205.000 ppm  
F1 15457.48 Hz  
F2P -5.000 ppm  
F2 -377.01 Hz  
PPMCM 10.50000 ppm/cm  
HZCM 791.72461 Hz/cm

HMOC

Current Data Parameters  
 NAME May23-5JH  
 EXPNO 3  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20080523  
 Time 22.34  
 INSTRUM spect  
 PROBHD 5 mm BBI 1H-3  
 PULPROG inv4gptp  
 TD 2648  
 SOLVENT CDCl3  
 NS 8  
 DS 80  
 SWH 406.410 MHz  
 FIDRES 1.552295 Hz  
 AQ 0.2556404 sec  
 RG 14595.5  
 DM 124.800 usec  
 DE 5.00 usec  
 TE 300.0 K  
 CNST2 145.000000  
 a0 0.0000300 sec  
 a1 1.00031250 sec  
 a2 0.00344628 sec  
 a4 0.00172414 sec  
 a11 0.03000000 sec  
 a13 0.0000300 sec  
 a15 0.0003000 sec  
 a20 0.00032414 sec  
 a21 0.00224228 sec  
 aNO 0.0001308 sec

\*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
 NUCl1 1H  
 P1 5.45 usec  
 P2 12.00 usec  
 PL1 2.00 dB  
 SF01 400.1320007 MHz

\*\*\*\*\* CHANNEL f2 \*\*\*\*\*  
 GRPRG2 gpr2  
 NUCl2 13C  
 P3 17.00 usec  
 P4 34.00 usec  
 PFC02 64.00 usec  
 PL2 -5.00 dB  
 PL12 6.30 dB  
 SF02 100.6222872 MHz

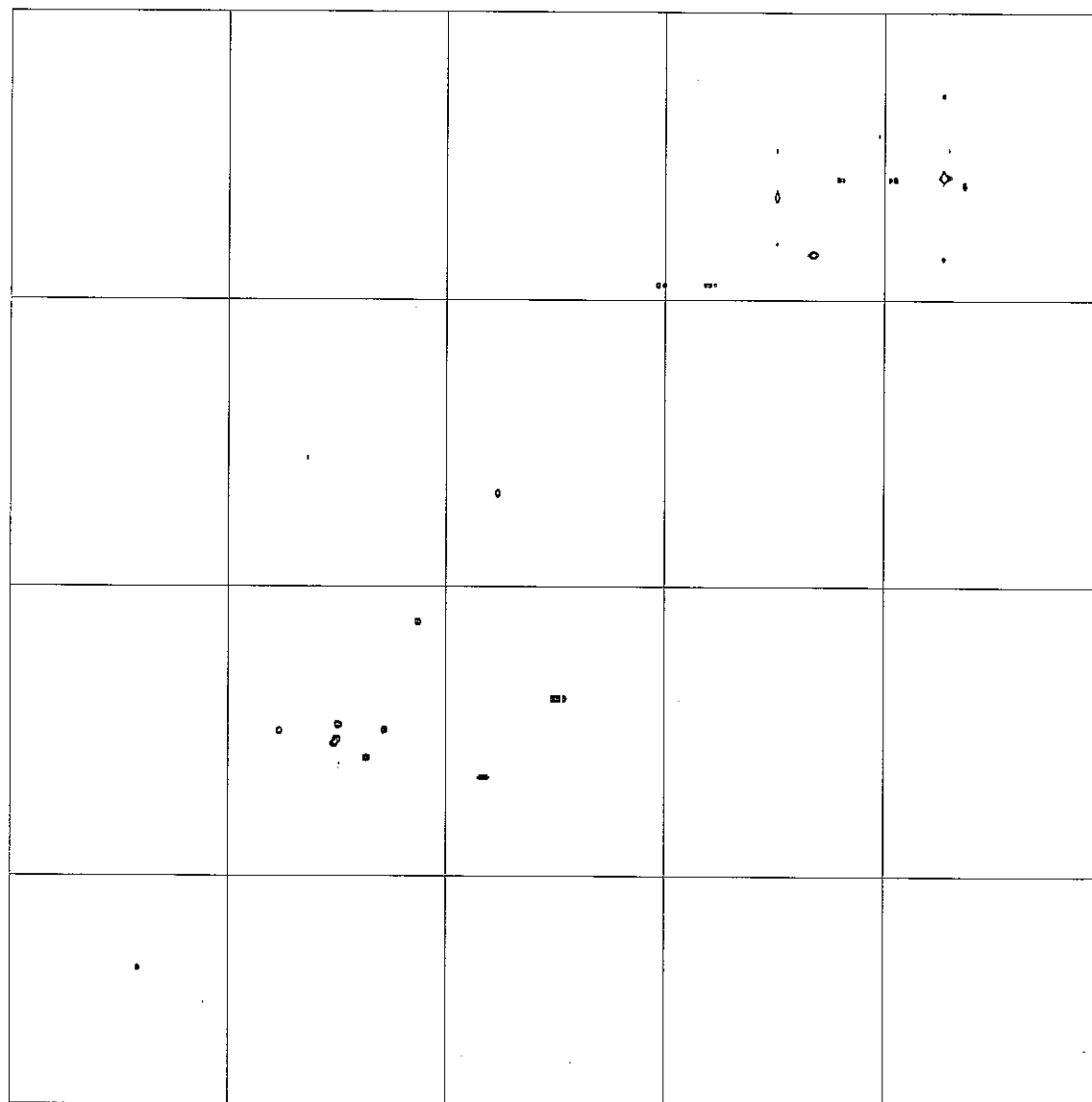
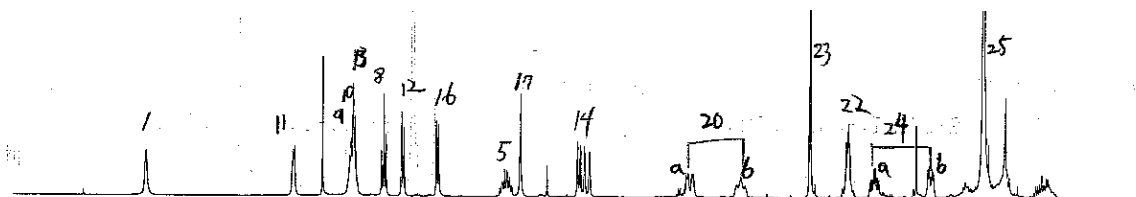
\*\*\*\*\* GRADIENT CHANNEL \*\*\*\*\*  
 GPNM1 SINE\_100  
 GPNM2 SINE\_100  
 GPNM3 SINE\_100  
 GPX1 17.00 X  
 GPX2 20.00 X  
 GPX3 25.00 X  
 GPY1 17.00 X  
 GPY2 20.00 X  
 GPY3 25.00 X  
 GPZ1 17.00 X  
 GPZ2 20.00 X  
 GPZ3 25.00 X  
 P16 1000.00 usec

F1 - Acquisition parameters  
 NDO 4  
 TD 256  
 SF01 100.6223 MHz  
 FIDRES 74.58923 Hz  
 SM 190.022 ppm

F2 - Processing parameters  
 SI 2043  
 SF 400.1300054 MHz  
 WDW GSTH  
 SSB 2  
 LB 0.00 Hz  
 GB 0  
 PC 1.40

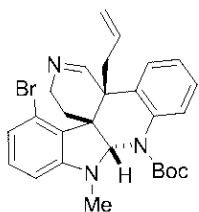
F1 - Processing parameters  
 SI 1024  
 MC2 1PP1  
 SF 100.6127230 MHz  
 WDW GSTH  
 SSB 2  
 LB 0.00 Hz  
 GB 0

2D NMR plot parameters  
 CX2 15.00 cm  
 CX1 15.00 cm  
 F2PLD 9.993 ppm  
 F2L0 3959.47 Hz  
 F2PH 0.00 ppm  
 F2H1 -7.95 Hz  
 F1PLD 190.020 ppm  
 F1L0 19113.44 Hz  
 F1PH 0.00 ppm  
 F1H1 -2.02 Hz  
 F2PPMCH 0.86752 ppm/cm  
 F1MCH 267.03602 Hz/cm  
 F1PPMCH 12.08334 ppm/cm  
 F1MCH 1274.09727 Hz/cm



ppm

ppm



41

25

23

21

20

19

17

18

16

14

15

13

12

11

10

9

8

7

6

5

4

3

2

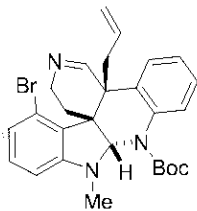
1

50

100

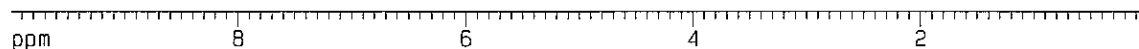
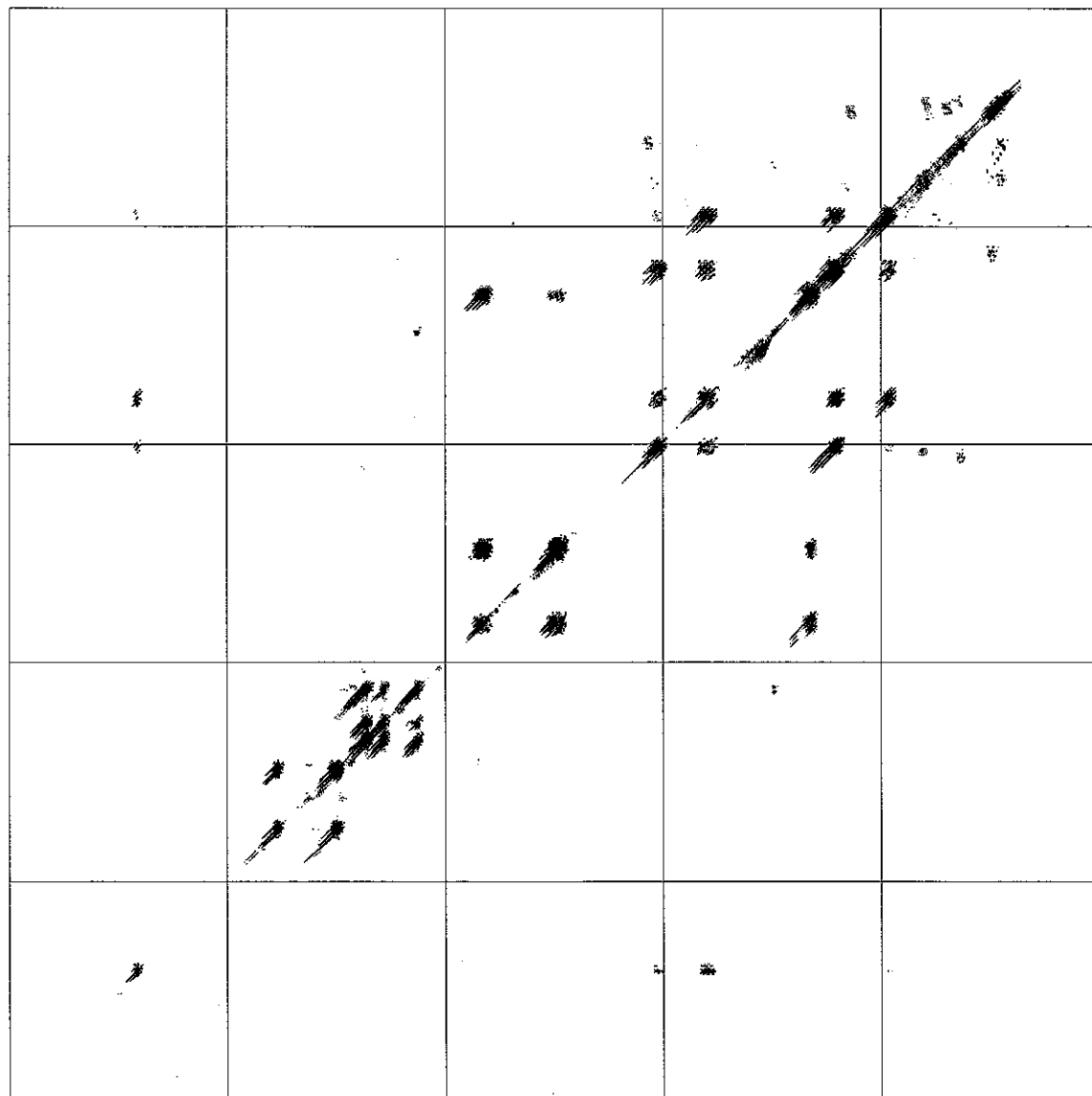
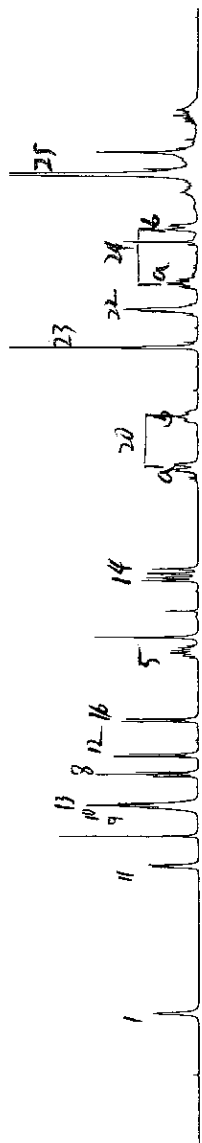
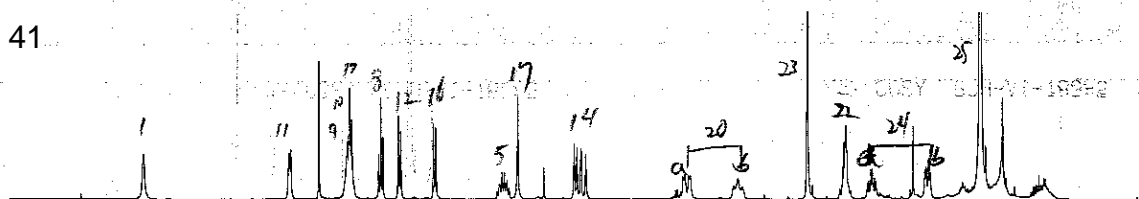
150





S153

41



COSY

Current Data Parameters  
 NAME May23-SJH  
 EXPNO 5  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20080525  
 Time 17.21  
 INSTRUM spect  
 PROBHD 5 mm BBI 1H-8  
 PULPROG ajb.cosygmftp  
 TD 2048  
 SOLVENT CDCl3  
 NS 6  
 DS 8  
 SWH 4006.410 Hz  
 FIDRES 1.955255 Hz  
 AQ 0.2556404 sec  
 RG 842.75  
 DW 124.800 usec  
 DE 5.00 usec  
 TE 300.0 K  
 D0 0.0000300 sec  
 D1 4.0000000 sec  
 d13 0.0000300 sec  
 D16 0.0020000 sec  
 d20 0.00120300 sec  
 IN0 0.00012496 sec

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 6.45 usec  
 P2 12.90 usec  
 PL1 0.00 dB  
 SFO1 400.1320007 MHz

===== GRADIENT CHANNEL =====  
 P16 1000.00 usec

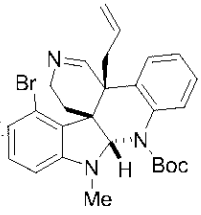
F1 - Acquisition parameters  
 NDD 2  
 TD 650  
 SFO1 400.132 MHz  
 FIDRES 6.155693 Hz  
 SW 10.000 ppm

F2 - Processing parameters  
 SI 2048  
 SF 400.1300054 MHz  
 NDD SINE  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 PC 1.40

F1 - Processing parameters  
 SI 1024  
 MC2 TPPI  
 SF 400.1300054 MHz  
 NDD SINE  
 SSB 0  
 LB 0.00 Hz  
 GB 0

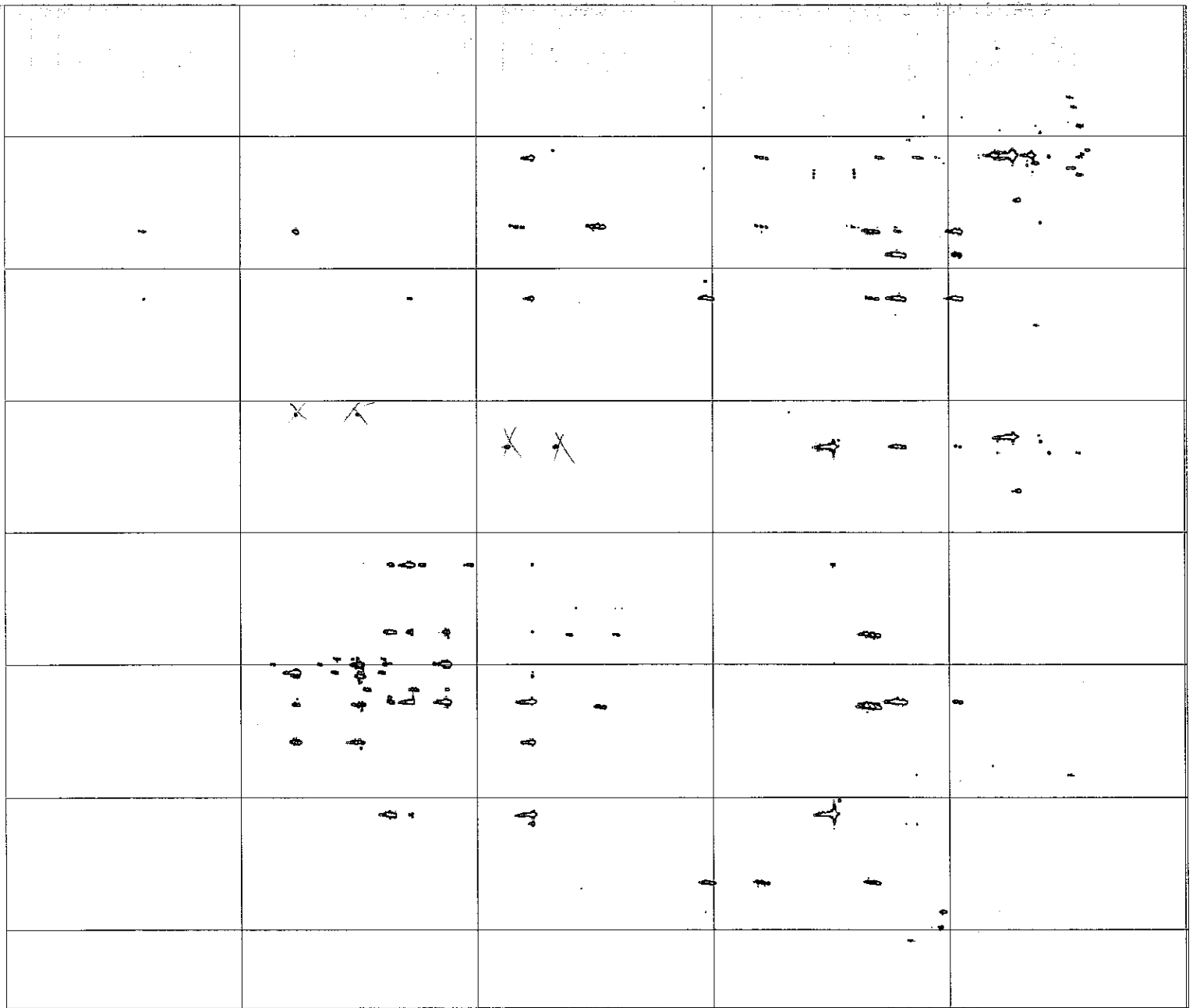
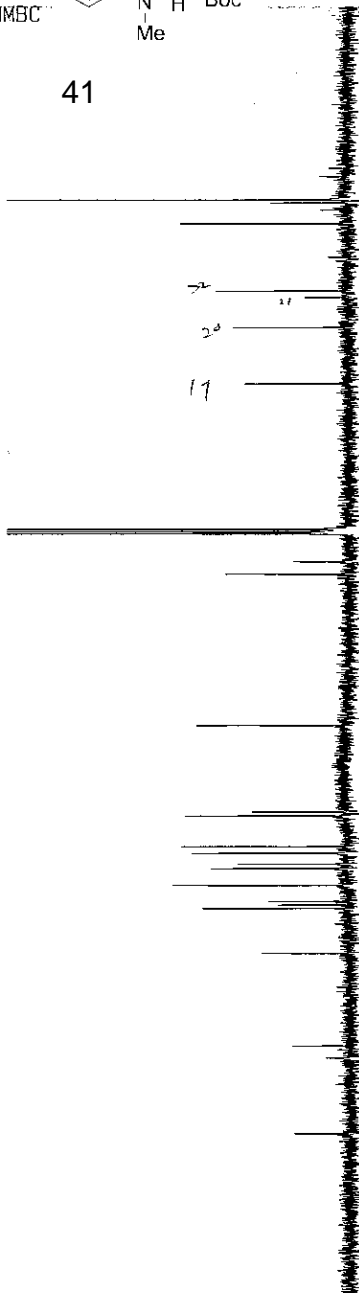
2D NMR plot parameters  
 CX2 15.00 cm  
 CX1 15.00 cm  
 F2PLO 10.000 ppm  
 F2LO 4001.30 Hz  
 F2PHI 0.000 ppm  
 F2HI 0.00 Hz  
 F1PLO 10.000 ppm  
 F1LO 4001.30 Hz  
 F1PHI 0.000 ppm  
 F1HI 0.00 Hz  
 F2PPMCM 0.65667 ppm/cm  
 F2HZCM 266.75333 Hz/cm  
 F1PPMCM 0.65667 ppm/cm  
 F1HZCM 266.75333 Hz/cm





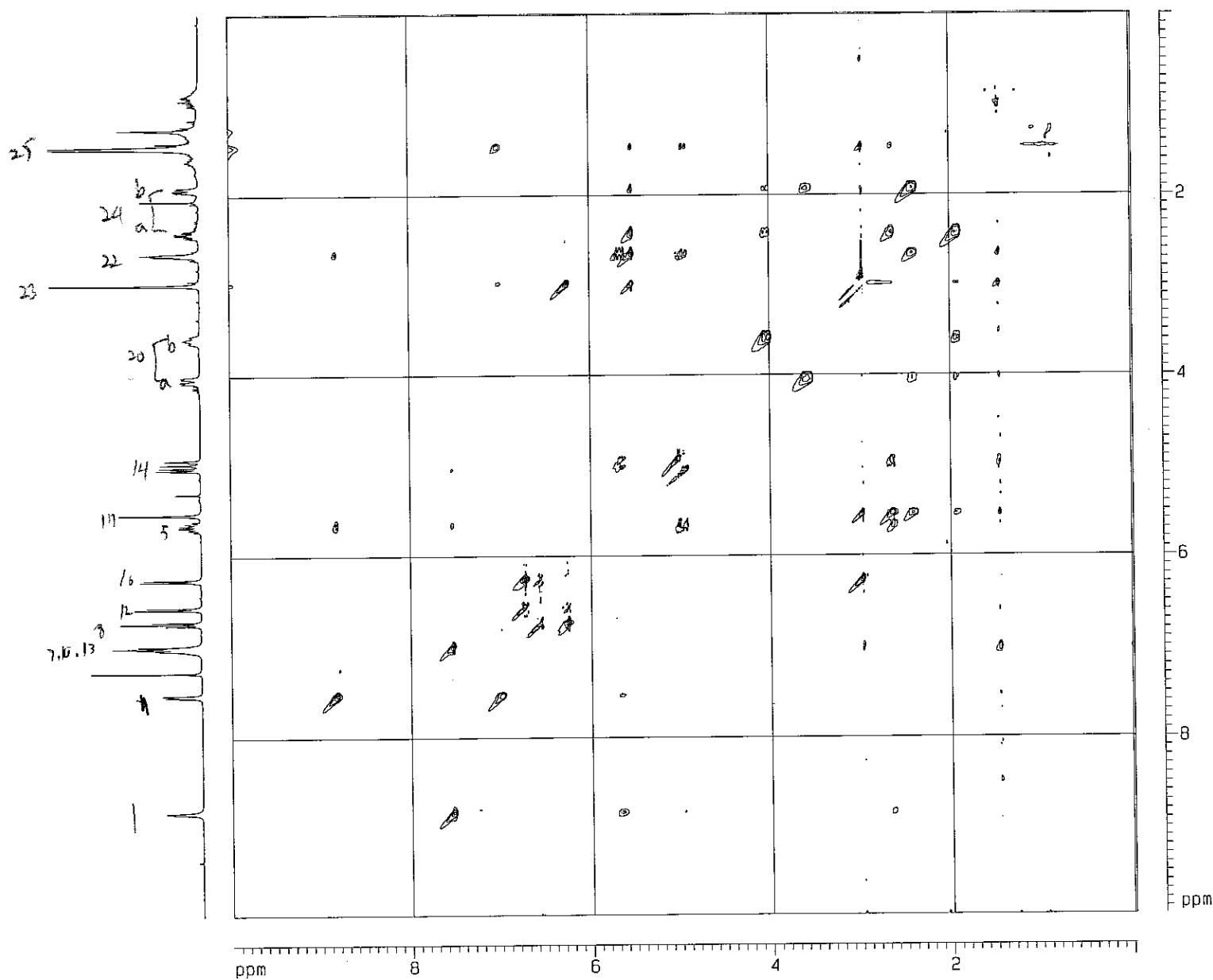
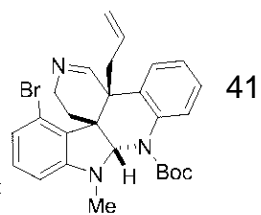
HMBC

41



ppm 8 6 4 2

NOESY



Current Data Parameters  
 NAME May23-5JH  
 EXPNO 6  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20080525  
 Time 22.02  
 INSTRUM spect  
 PROBHD 5 mm BBI 1H-B  
 PULPROG noesygptp  
 TO 2048  
 SOLVENT CDCl3  
 NS 12  
 DS 8  
 SWH 4005.410 Hz  
 FIDRES 1.956295 Hz  
 AQ 0.2555404 sec  
 RG 22.5  
 OW 124.800 usec  
 DE 6.00 usec  
 TE 300.0 K  
 d0 0.0000300 sec  
 d1 4.0000000 sec  
 dB 0.8000001 sec  
 D16 0.0020000 sec  
 d20 0.3988002 sec  
 INO 0.0012496 sec

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 8.45 usec  
 P2 12.90 usec  
 PL1 0.00 dB  
 SF01 400.132007 MHz

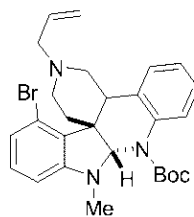
===== GRADIENT CHANNEL =====  
 GPNAM1 sine.100  
 GPNAM2 sine.100  
 GPX1 0.00 %  
 GPX2 0.00 %  
 GPY1 0.00 %  
 GPY2 0.00 %  
 GPF1 40.00 %  
 GPF2 -40.00 %  
 P16 1000.00 usec

F1 - Acquisition parameters  
 ND0 2  
 TD 512  
 SF01 400.132 MHz  
 FIDRES 7.814845 Hz  
 SN 10.000 ppm

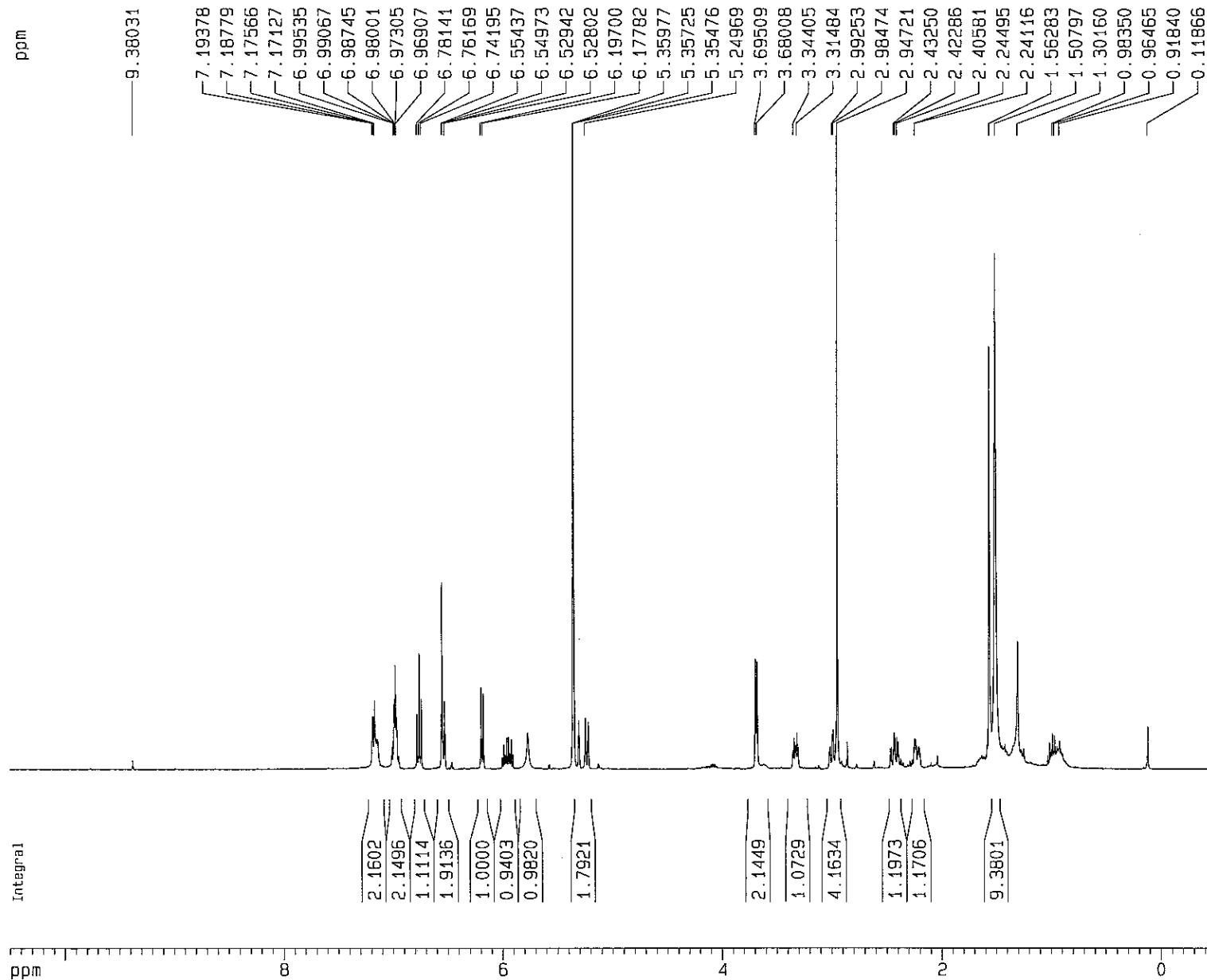
F2 - Processing parameters  
 SI 2048  
 SF 400.1300054 MHz  
 MDW 0SINE  
 SSB 2  
 LB 0.00 Hz  
 GB 0  
 PC 1.40

F1 - Processing parameters  
 SI 1024  
 MC2 TPP1  
 SF 400.1300054 MHz  
 MDW 0SINE  
 SSB 2  
 LB 0.00 Hz  
 GB 0

2D NMR plot parameters  
 CX2 15.00 cm  
 CX1 15.00 cm  
 F2PLD 9.993 ppm  
 F2L0 3998.47 Hz  
 F2PHI -0.020 ppm  
 F2H1 -7.95 Hz  
 F1PLD 9.986 ppm  
 F1L0 3995.86 Hz  
 F1PHI -0.013 ppm  
 F1H1 -5.34 Hz  
 F2PPMCH 0.86752 ppm/cm  
 F2HZCM 267.09402 Hz/cm  
 F1PPMCH 0.66865 ppm/cm  
 F1HZCM 266.74867 Hz/cm



66



Current Data Parameters  
 NAME Apr09-SJH  
 EXPNO 1  
 PROCNO 1

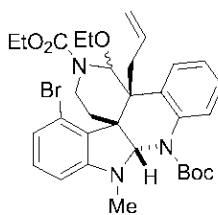
F2 - Acquisition Parameters  
 Date\_ 20080409  
 Time 16.26  
 INSTRUM spect  
 PROBHD 5 mm BBI 1H-8  
 PULPROG zg30  
 TD 49668  
 SOLVENT CD2Cl2  
 NS 26  
 DS 2  
 SWH 8278.146 Hz  
 FIDRES 0.166670 Hz  
 AQ 2.9999971 sec  
 RG 512  
 DW 60.400 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 1.00000000 sec

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 6.45 usec  
 PL1 0.00 dB  
 SF01 400.1324710 MHz

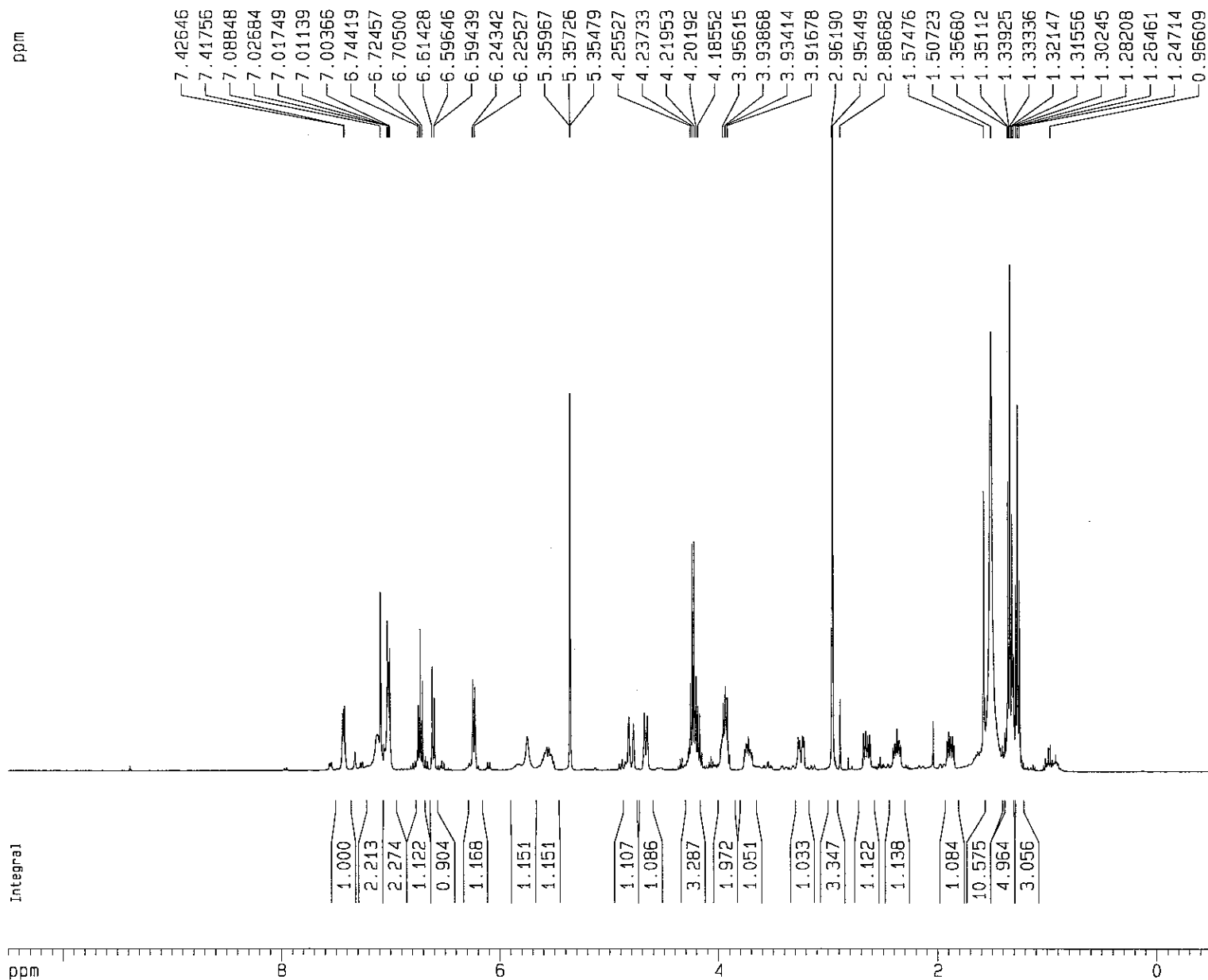
F2 - Processing parameters  
 SI 32768  
 SF 400.1300000 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 10.500 ppm  
 F1 4201.37 Hz  
 F2P -0.500 ppm  
 F2 -200.07 Hz  
 PPMCM 0.55000 ppm/cm  
 HZCM 220.07150 Hz/cm

CD2C12



42



## Current Data Parameters

NAME Apr10-SJH  
EXPNO 1  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20080410  
Time 9.09  
INSTRUM spect  
PROBHD 5 mm BBI 1H-B  
PULPROG zg30  
TD 49658  
SOLVENT CD2C12  
NS 15  
DS 2  
SWH 8278.146 Hz  
FIDRES 0.166670 Hz  
AQ 2.9999971 sec  
RG 203.2  
DW 60.400 usec  
DE 6.00 usec  
TE 300.0 K  
D1 1.00000000 sec

## ===== CHANNEL f1 =====

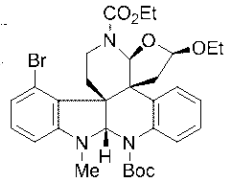
NUC1 1H  
P1 6.45 usec  
PL1 0.00 dB  
SF01 400.1324710 MHz

## F2 - Processing parameters

SI 32768  
SF 400.1300000 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

## 1D NMR plot parameters

CX 20.00 cm  
F1P 10.500 ppm  
F1 4201.37 Hz  
F2P -0.500 ppm  
F2 -200.07 Hz  
PPMCM 0.55000 ppm/cm  
HZCM 220.07150 Hz/cm

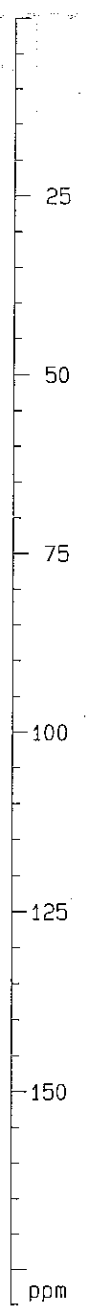
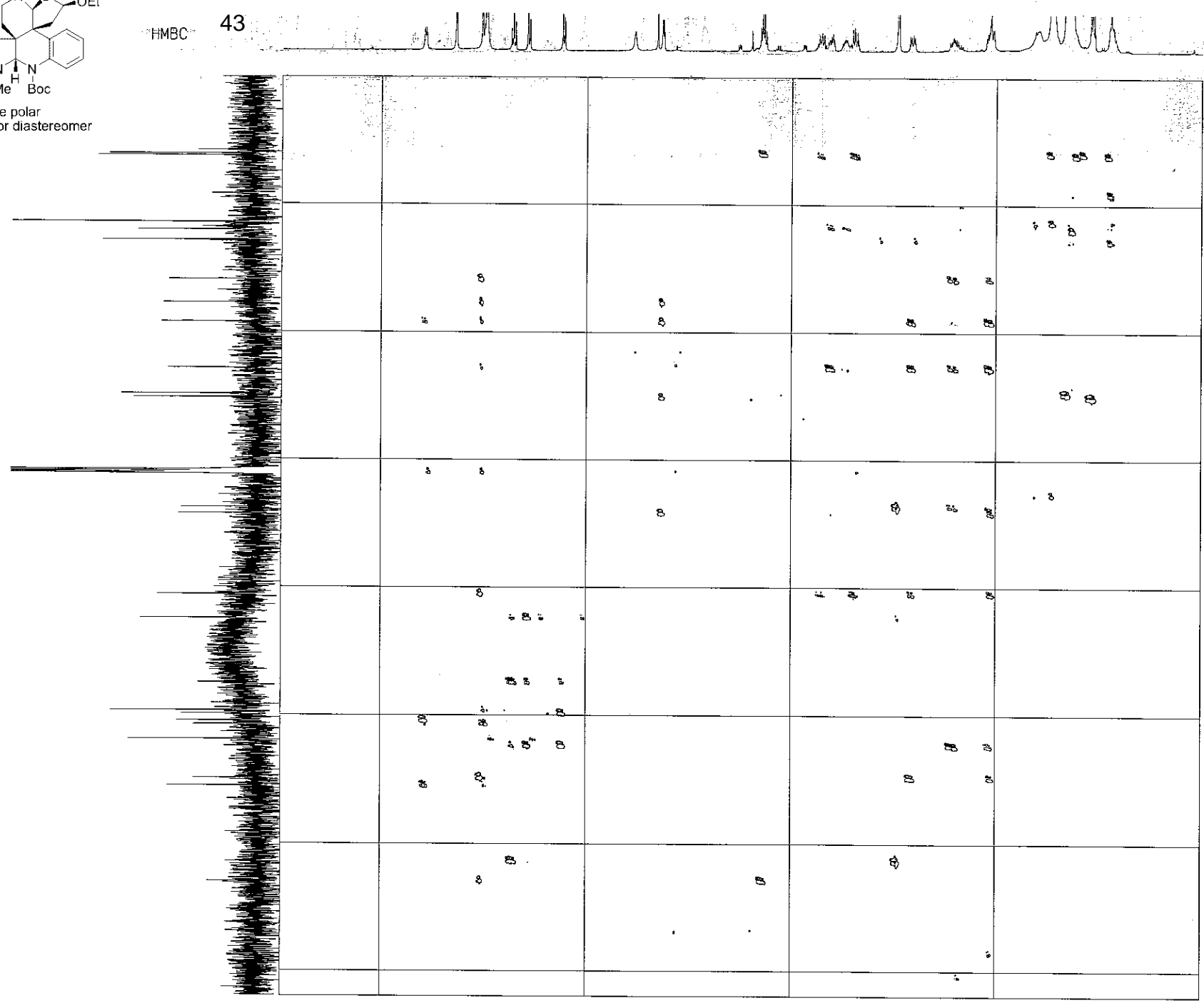


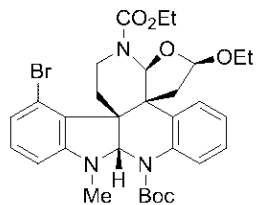
HMBC

43

S158

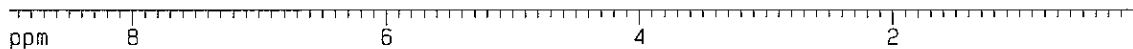
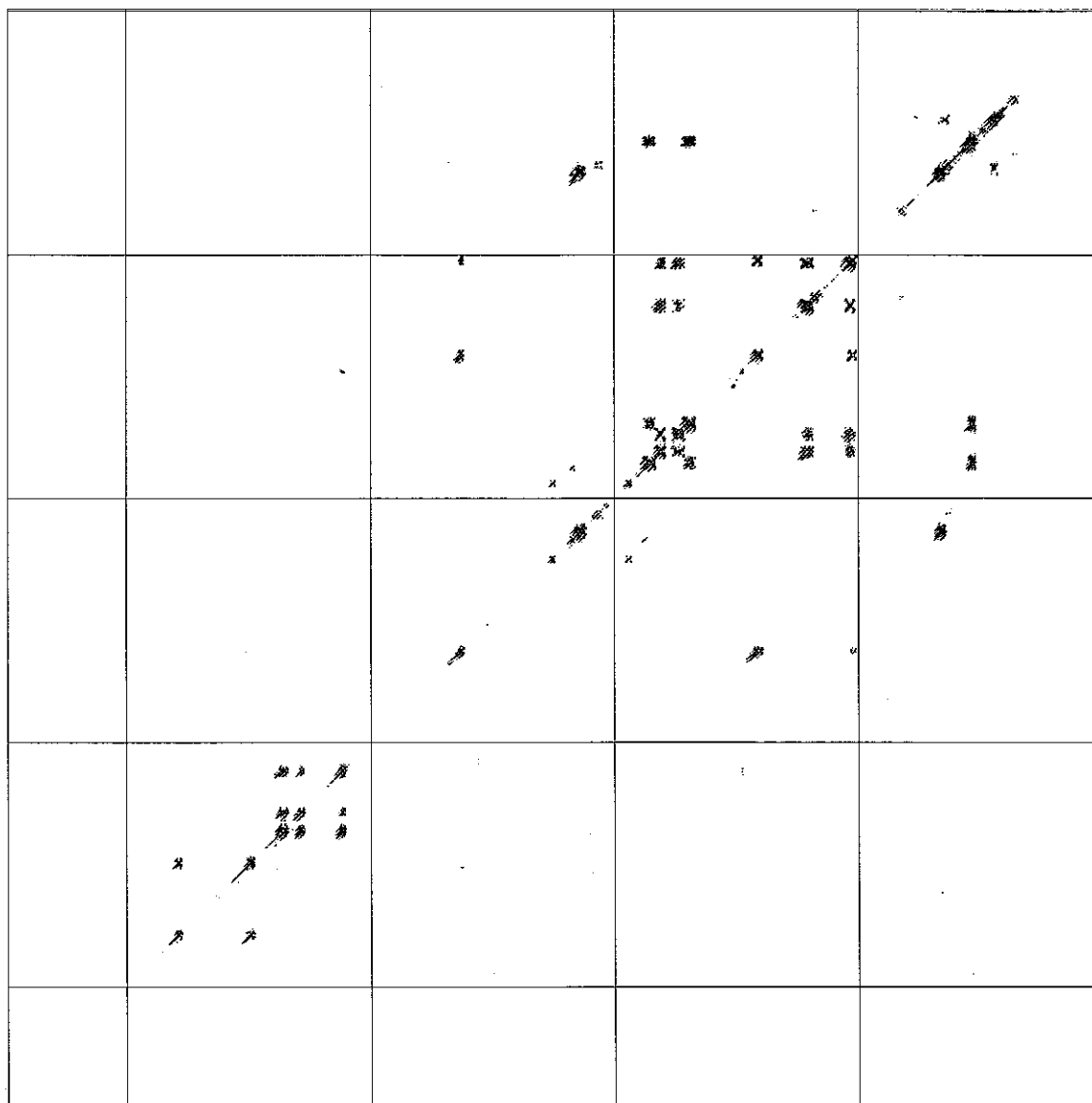
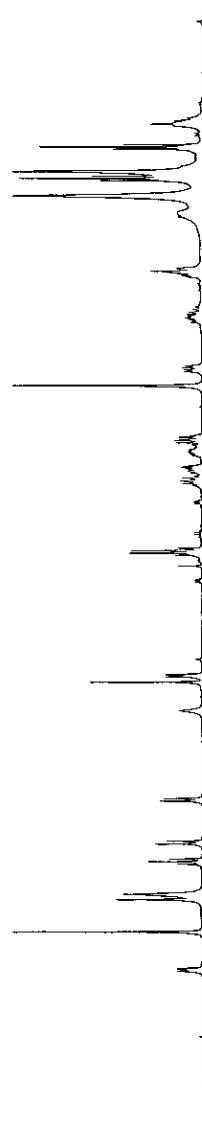
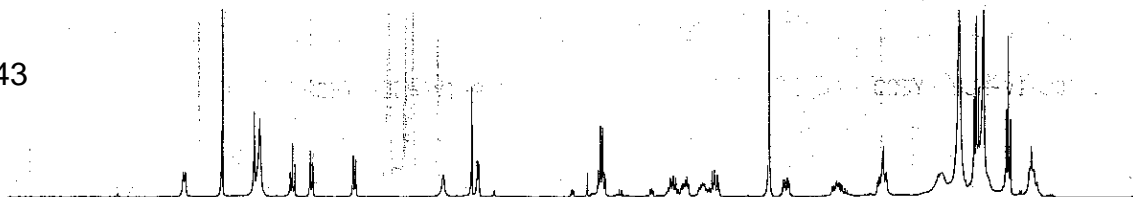
more polar  
major diastereomer





43

more polar  
major diastereomer



S159

COSY

Current Data Parameters  
NAME May28-SJH  
EXPNO 5  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20080529  
Time 5.29  
INSTRUM spect  
PROBHD 5 mm BBI 1H-9  
PULPROG ajb.cosygamfz  
TO 2048  
SOLVENT DDC13  
NS 6  
DS 8  
SWH 3591.954 Hz  
FIDRES 1.753884 Hz  
AQ 0.2851316 sec  
RG 1625.5  
DW 139.200 usec  
DE 6.00 usec  
TE 300.0 K  
D0 0.0000300 sec  
D1 4.0000000 sec  
d13 0.0000300 sec  
D16 0.0020000 sec  
d20 0.00120300 sec  
IN0 0.00013884 sec

===== CHANNEL f1 =====  
NUC1 1H  
P1 7.13 usec  
P2 14.30 usec  
PL1 0.00 dB  
SFO1 400.1318006 MHz

===== GRADIENT CHANNEL =====  
P16 1000.00 usec

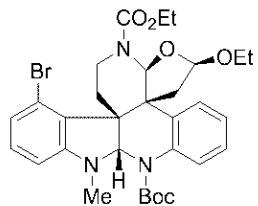
F1 - Acquisition parameters  
NDD 2  
TD 650  
SFO1 400.1318 MHz  
FIDRES 5.540512 Hz  
SN 9.000 ppm

F2 - Processing parameters  
SI 2048  
SF 400.1300056 MHz  
WDW SINE  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.40

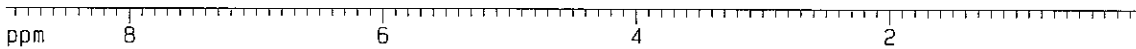
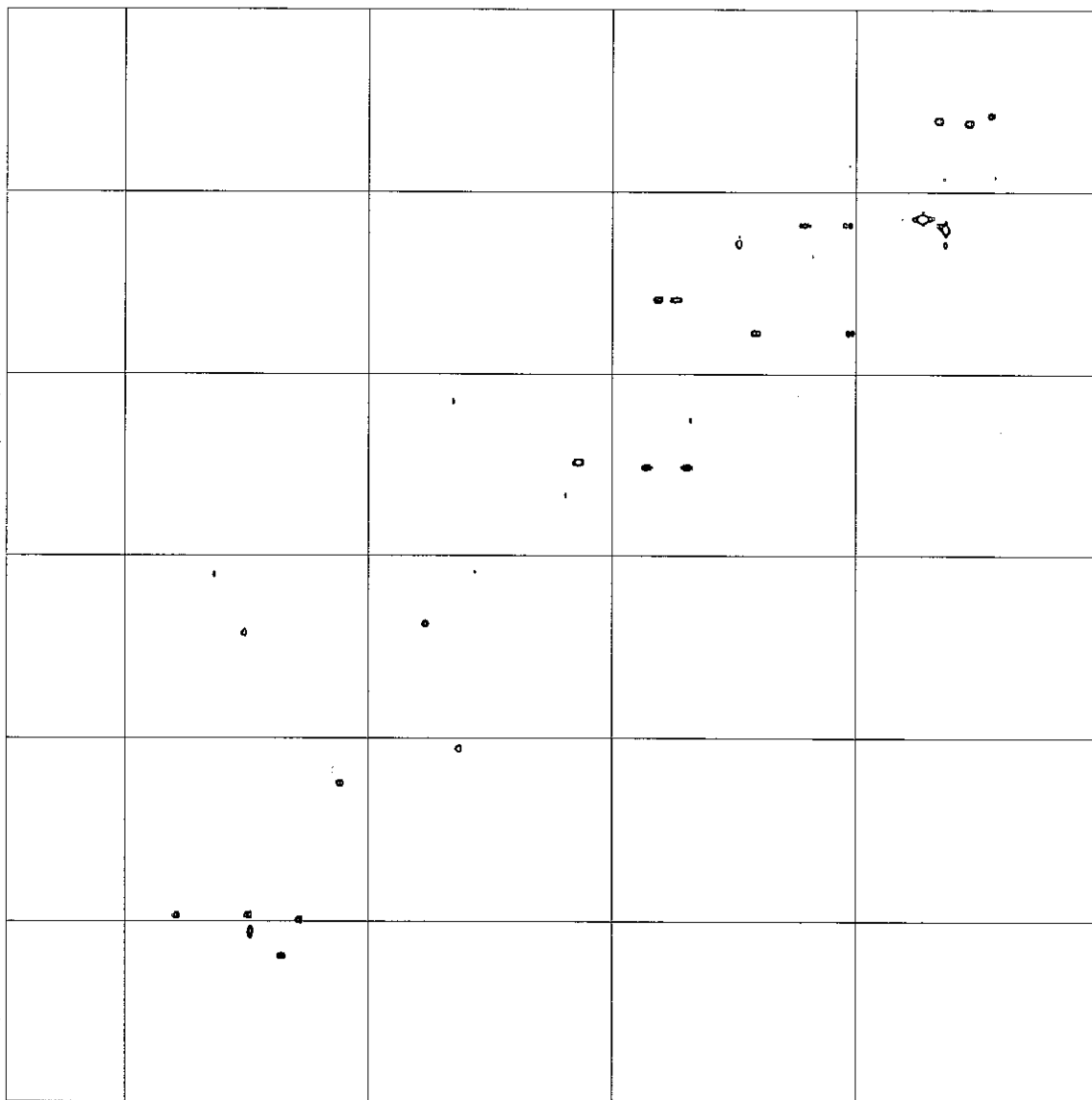
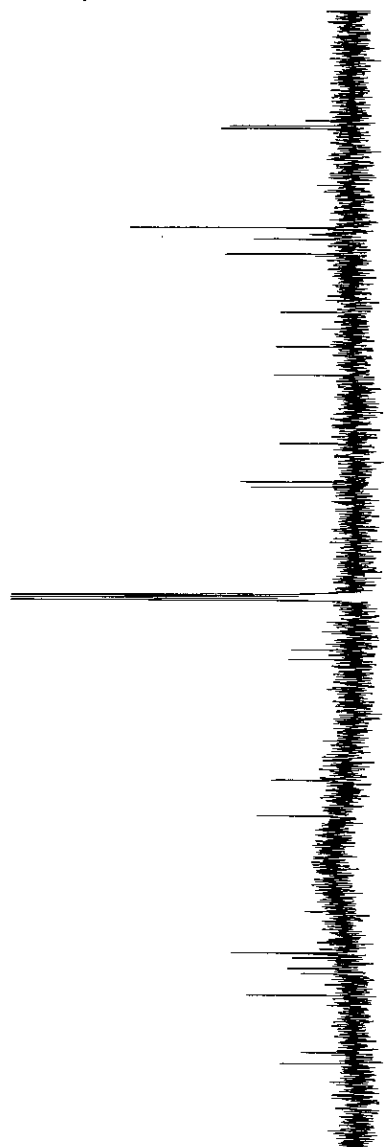
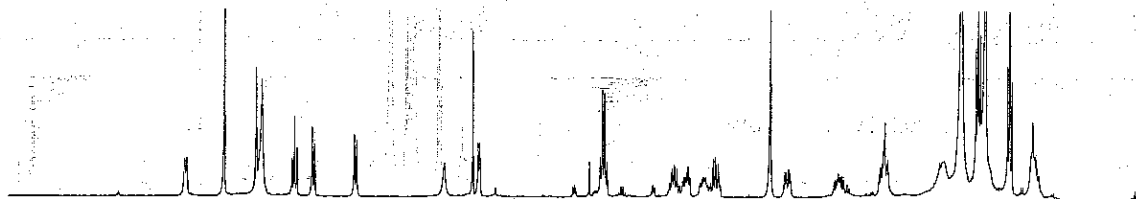
F1 - Processing parameters  
SI 1024  
MC2 TPPI  
SF 400.1300056 MHz  
WDW SINE  
SSB 0  
LB 0.00 Hz  
GB 0

2D NMR plot parameters  
CX2 15.00 cm  
CX1 15.00 cm  
F2PL0 8.974 ppm  
F2L0 3590.92 Hz  
F2PHI -0.003 ppm  
F2HT -1.03 Hz  
F4PL0 8.985 ppm  
F4L0 3595.61 Hz  
F4PHI -0.014 ppm  
F4HT -5.72 Hz  
F2PRMCM 0.59845 ppm/cm  
F2HZCM 239.46361 Hz/cm  
F1PRMCM 0.50003 ppm/cm  
F1HZCM 240.08882 Hz/cm





more polar  
major diastereomer



S160

HMQC

Current Data Parameters  
 NAME May23-SJM  
 EXPNO 3  
 PRGNO 1

F2 - Acquisition Parameters  
 Date\_ 20060528  
 Time 20.26  
 INSTRUM spect  
 PROBHD 5 mm BBI 2H-5  
 PULPROG invgpgtp  
 ID 2048  
 SOLVENT CDCl3  
 NS 16  
 DS 80  
 SWH 3391.954 Hz  
 FIDRES 1.733884 Hz  
 AQ 0.2651316 sec  
 RG 23193  
 DM 139.400 usec  
 DE 6.00 usec  
 TE 300.0 K  
 CHST2 145.000000  
 d0 0.0000000 sec  
 D1 1.99631200 sec  
 D2 0.00344529 sec  
 D4 0.00172414 sec  
 d13 0.03000000 sec  
 dJ3 0.00000000 sec  
 D16 0.00020000 sec  
 d59 0.00020414 sec  
 dE1 0.00234200 sec  
 INO 0.00001655 sec

\*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
 NU31 2H  
 P1 7.15 usec  
 p2 14.30 usec  
 PL1 0.00 dB  
 SFO1 400.1318006 MHz

\*\*\*\*\* CHANNEL f2 \*\*\*\*\*  
 CPDPR22 gprp  
 MUC2 13C  
 P3 17.00 usec  
 p4 34.00 usec  
 PCPD2 64.00 usec  
 PL2 -6.00 dB  
 PL3 6.30 dB  
 SFO2 100.6202750 MHz

\*\*\*\*\* GRADIENT CHANNEL \*\*\*\*\*  
 GPMH1 SINE.100  
 GPMH2 SINE.100  
 GPMH3 SINE.100  
 GPX1 17.00 %  
 GPX2 20.00 %  
 GPX3 25.00 %  
 GPY1 17.00 %  
 GPY2 20.00 %  
 GPY3 25.00 %  
 GPZ1 17.00 %  
 GPZ2 20.00 %  
 GPZ3 25.00 %  
 P16 1000.00 usec

F1 - Acquisition parameters  
 ND0 4  
 TD 256  
 SFO1 100.6203 MHz  
 FIDRES 58.952265 Hz  
 SN 150.013 ppm

F2 - Processing parameters  
 SI 2048  
 SF 400.1300556 MHz  
 WDW 651NE  
 SSB 2  
 LB 0.00 Hz  
 GB 0  
 PC 1.40

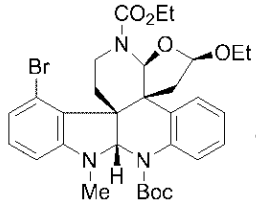
F1 - Processing parameters  
 SI 1024  
 HCF 1P1  
 SF 100.6127250 MHz  
 WDW 651NE  
 SSB 2  
 LB 0.00 Hz  
 GB 0

2D NMR plot parameters  
 CX2 15.00 cm  
 CX1 15.00 cm  
 F2PLO 8.374 ppm  
 F2LO 9590.90 Hz  
 F2PH1 -0.003 ppm  
 F2H1 -1.03 Hz  
 F1PLO 150.012 ppm  
 F1LO 15093.13 Hz  
 F1PH1 -0.012 ppm  
 F1H1 -1.21 Hz  
 F2FWMH 0.98245 ppm/cm  
 F2HCCA 233.46361 Hz/cm  
 F1FWMH 10.00161 ppm/cm  
 F1HCCA 1006.28931 Hz/cm

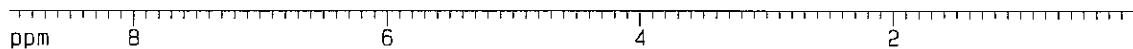
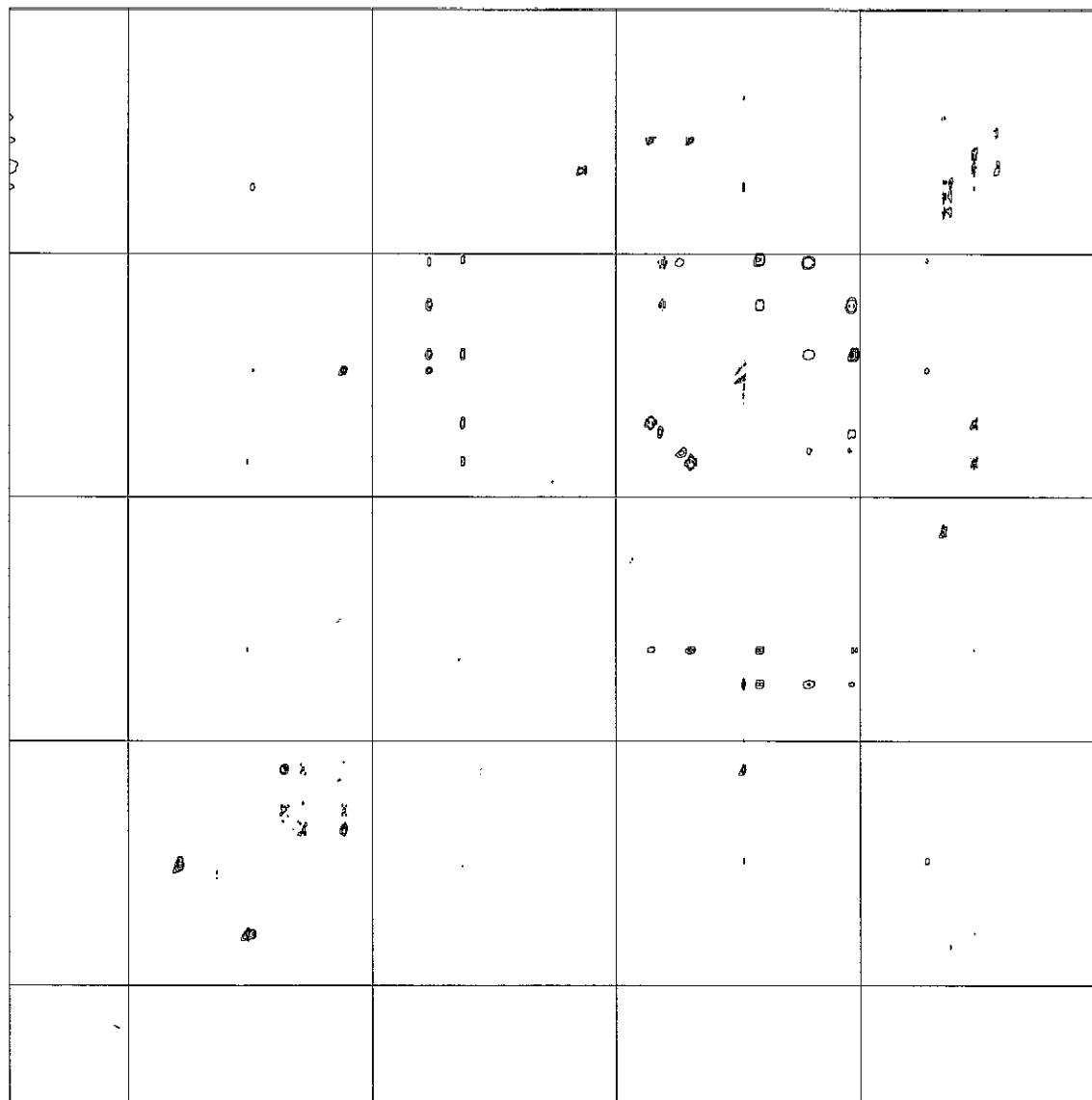
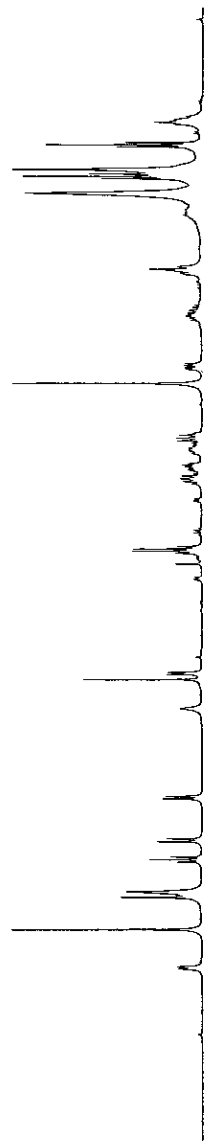
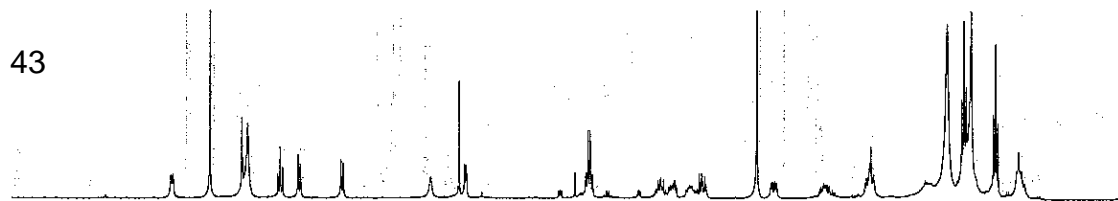
25  
50  
75  
100  
125  
ppm



NOESY



more polar  
major diastereomer



Current Data Parameters  
NAME May28-SJH  
EXPNO 7  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20080529  
Time 21.20  
INSTRUM spect  
PROBHD 5 mm BBI 1H-5  
PULPROG noesygpgp  
TD 2048  
SOLVENT CDCl3  
NS 16  
DS 8  
SWH 3591.954 Hz  
FIDRES 1.753884 Hz  
AQ 0.2851318 sec  
RG 50.64  
DM 139.200 usec  
DE 6.00 usec  
TE 300.0 K  
dD 0.0000300 sec  
D1 4.0000000 sec  
DB 0.8000001 sec  
D18 0.0020000 sec  
d20 0.3980002 sec  
IN0 0.0013884 sec

----- CHANNEL f1 -----  
NUC1 1H  
P1 7.15 usec  
P2 14.30 usec  
PL1 0.00 dB  
SFO1 400.1318006 MHz

----- GRADIENT CHANNEL -----  
GPMAX1 sine.100  
GPMAX2 sine.100  
GPK1 0.00 %  
GPK2 0.00 %  
GPH1 0.00 %  
GPH2 0.00 %  
GPT1 40.00 %  
GPT2 -40.00 %  
PI6 1000.00 usec

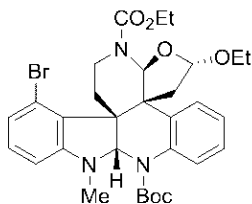
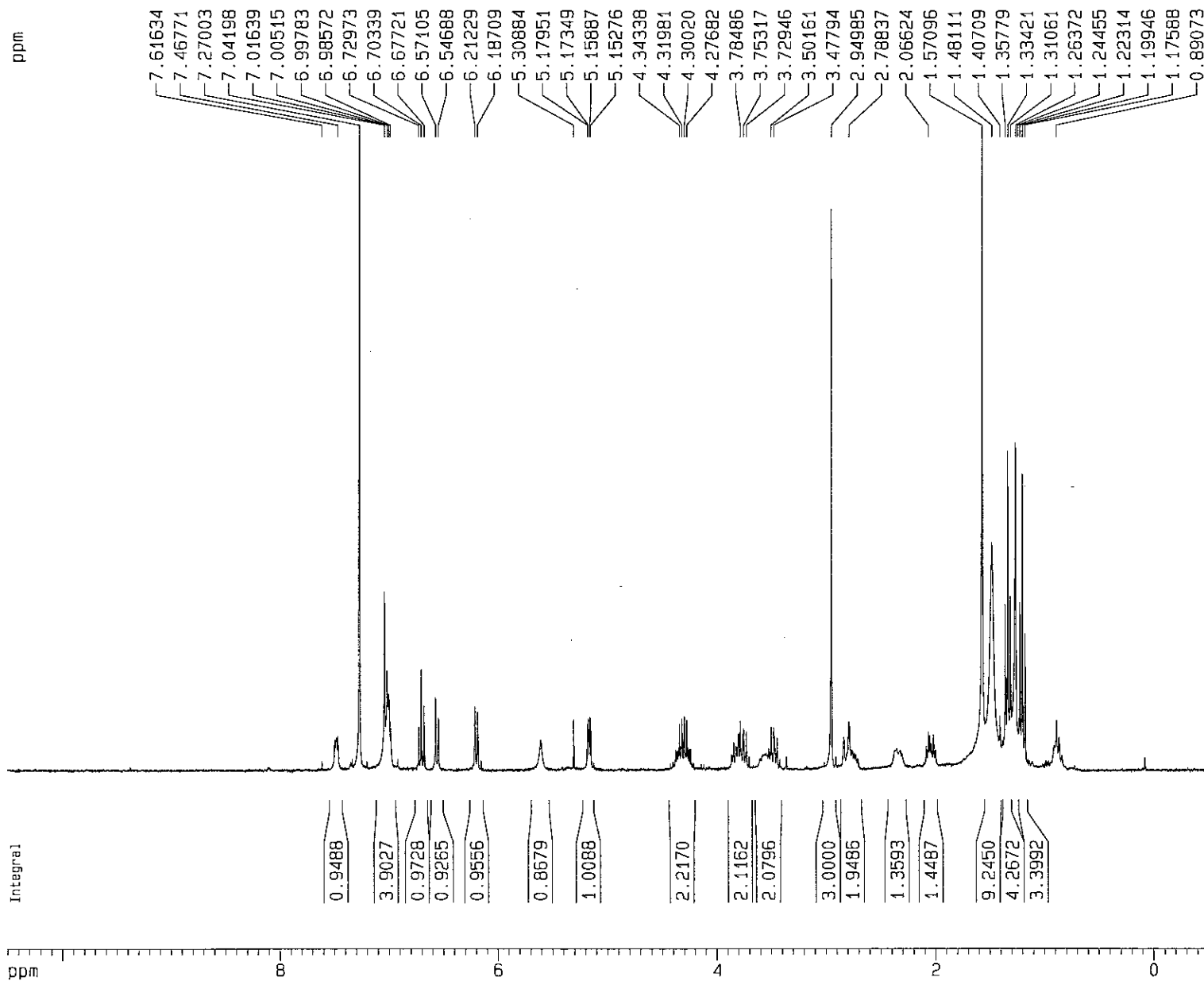
F1 - Acquisition parameters  
ND0 2  
TD 512  
SFO1 400.1318 MHz  
FIDRES 7.033853 Hz  
SW 8.000 ppm

F2 - Processing parameters  
SI 2048  
SF 400.1300051 MHz  
WDW OSINE  
SSB 2  
LB 0.00 Hz  
GB 0  
PC 1.00

F1 - Processing parameters  
SI 1024  
MC2 TPP1  
SF 400.1300051 MHz  
WDW OSINE  
SSB 2  
LB 0.00 Hz  
GB 0

2D NMR plot parameters  
CX2 15.00 cm  
CX1 15.00 cm  
F2PL0 8.976 ppm  
F2L0 3591.43 Hz  
F2PH1 -0.001 ppm  
F2H1 -0.52 Hz  
F1PL0 8.997 ppm  
F1L0 3596.12 Hz  
F1PH1 -0.013 ppm  
F1H1 -5.21 Hz  
F2PPMCM 0.59846 ppm/cm  
F2HZCM 239.46361 Hz/cm  
F1PPMCM 0.60003 ppm/cm  
F1HZCM 240.08882 Hz/cm



43  
minor

Current Data Parameters  
 NAME May27-SJH  
 EXPNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20080527  
 Time 22.20  
 INSTRUM spect  
 PROBHD 5 mm QNP 1H/1  
 PULPROG zg30  
 TD 37036  
 SOLVENT CDC13  
 NS 17  
 DS 2  
 SWH 6172.839 Hz  
 FIDRES 0.166671 Hz  
 AQ 2.9999659 sec  
 RG 812.7  
 DW 81.000 usec  
 DE 6.00 usec  
 TE 300.0 K  
 D1 1.00000000 sec

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 11.70 usec  
 PL1 0.00 dB  
 SF01 299.8718518 MHz

F2 - Processing parameters  
 SI 32768  
 SF 299.8700069 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 20.00 cm  
 F1P 10.500 ppm  
 F1 3148.64 Hz  
 F2P -0.500 ppm  
 F2 -149.93 Hz  
 PPMCM 0.55000 ppm/cm  
 HZCM 164.92850 Hz/cm