

Experimental Section

General. All reactions were performed under nitrogen or argon atmosphere with flame-dried glassware. All solvents were distilled prior to use; diethyl ether and THF over sodium and benzophenone. Toluene and dichloromethane (DCM) were dried over calcium hydride. Reactions were monitored by TLC analysis of aliquots, using aluminum sheets precoated (0.2 mm layer thickness) with silica gel 60 F₂₅₄ (E. Merck). Column chromatography was performed with 60 silica gel (230-400 mesh, Merck). ¹H and ¹³C NMR spectra were recorded on Bruker Advance 300 MHz and Bruker AMX 500 MHz spectrometers. IR spectra were recorded with a Bomen instrument. HRMS spectra were obtained on a Kratos Analytical Concept instrument.

5-Hydroxy-3-oxo-tetradec-6-enoic acid methyl ester (8)

To a mixture of sodium hydride (2.33 g, 58.3 mmol) in THF (194 mL), cooled to 0°C, was added methylacetooacetate **7** (4.2 mL, 38.9 mmol) dropwise. After stirring for 20 minutes, *n*-BuLi (2.38 M in hexanes, 17.9 mL, 42.8 mmol) was added dropwise and the resulting orange solution was cooled to -78°C. *trans*-2-Decenal (3.0 g, 19.5 mmol) was then added dropwise and the solution was stirred at -78°C for 20 minutes after which the reaction was quenched with saturated NH₄Cl_(aq) while still at -78°C. After warming to room temperature the layers were separated and the aqueous layer was extracted with ether. The combined organic extracts were dried (MgSO₄), and concentrated to dryness. Flash column chromatography with a gradient elution from 100% hexanes to 30% ethyl acetate/70% hexanes afforded **8** (3.87 g, 74%) as a yellow oil. ¹H NMR (300 MHz, benzene D₆): δ 5.56 – 5.66 (m, 1H), 5.32 – 5.40 (m, 1H), 4.43 (m, 1H), 3.26 (s, 3H), 3.04 – 3.05 (m, 2H), 2.38 – 2.47 (m, 1H), 2.20 – 2.31 (m, 1H), 1.92 (q, J = 6.7 Hz, 2H), 1.23 – 1.34 (m, 10H), 0.87 – 0.92 (m, 3H); ¹³C NMR (75 MHz, benzene D₆): δ 202.0 (C₄), 167.2 (C₄), 131.6 (CH), 131.6 (CH), 68.5 (CH), 51.7 (CH₃), 49.9 (CH₂), 49.6 (CH₂), 32.5 (CH₂), 32.2 (CH₂), 29.6 (CH₂), 29.5 (CH₂), 23.0 (CH₂), 14.3 (CH₃); IR (neat) 3455 (w), 2956 (m), 2926 (s), 2871 (w), 2855 (m), 1748 (s), 1716 (s), 1656 (w), 1629 (w), 1438 (m), 1405 (w), 1322 (m), 1268 (m), 1155 (m), 1095 (w), 1038 (w), 970 (w), 861 (w), 828 (w), 722 (w); HRMS (EI) *m/z* M⁺ – H₂O (C₁₅H₂₄O₃) calculated 252.17255, found 252.17220.

4-Hydroxy-6-non-1-enyl-tetrahydro-pyran-2-one (9)

To a mixture of acetonitrile (16 mL) and acetic acid (16 mL) cooled to 0°C was added Me₄NBH₄ (1.46 g, 16.4 mmol). The resulting mixture was left to stir at 0°C for 20 minutes and then cooled to -78°C. A solution of **8** (1.09 g, 4.03 mmol) in acetonitrile (2 mL) was then added to the frozen mixture via canula with a rinse of three 2 mL portions of acetonitrile. The reaction was then placed in a refrigerator set to -25°C and left for 48 hours with occasional swirling. The reaction was then quenched while still at -25°C with 33 mL of 0.5 N sodium potassium tartrate. The resulting mixture was warmed to room temperature, diluted with dichloromethane, and washed with saturated NaHCO_{3(aq)}. The layers were separated and the aqueous layer was extracted with dichloromethane. The

combined organic extracts were washed again with saturated NaHCO_3 _(aq) and the layers separated. The aqueous layer was extracted with dichloromethane. The combined organic extracts were dried (MgSO_4), and concentrated to dryness. The crude product was then dissolved in 36 mL of dichloromethane and to this was added TFA (0.06 mL measured from a needleless syringe, 0.7 mmol). The resulting solution was left to stir at room temperature for 48 hours. Solid sodium carbonate was then added to the reaction until the pH was non-acidic as indicated by pH paper. The mixture was filtered to remove the solid particles and the filtrate was dried (MgSO_4), and concentrated to dryness. Flash column chromatography with 55% ethyl acetate/45% hexanes afforded **9** (0.536 g, 55%) as a clear oil. ^1H NMR (300 MHz, CDCl_3): δ 5.78 (dt, J = 15.4, 6.7 Hz, 1H), 5.48 (dd, J = 15.4, 6.9 Hz, 1H), 4.58 – 4.65 (m, 1H), 4.25 (m, 1H), 2.90 (ddd, J = 17.2, 5.9, 1.3 Hz, 1H), 2.45 (dd, J = 17.2, 8.1 Hz, 1H), 2.22 – 2.30 (m, 1H), 2.18 (br s, 1H), 2.00 – 2.07 (m, 2H), 1.62 – 1.73 (m, 2H), 1.24 – 1.37 (m, 10H), 0.83 – 0.88 (m, 3H); ^{13}C NMR (75 MHz, CDCl_3): δ 170.3 (C₄), 135.6 (CH), 127.0 (CH), 77.8 (CH), 63.9 (CH), 39.5 (CH₂), 38.4 (CH₂), 32.1 (CH₂), 31.8 (CH₂), 29.1 (CH₂), 29.1 (CH₂), 28.7 (CH₂), 22.6 (CH₂), 14.1 (CH₃); IR (neat) 3423 (w), 2956 (m), 2926 (s), 2855 (m), 1731 (s), 1461 (w), 1359 (w), 1242 (m), 1164 (w), 1064 (m), 1034 (m), 968 (m), 950 (w); HRMS (EI) m/z $\text{M}^+ - \text{H}_2\text{O}$ ($\text{C}_{14}\text{H}_{22}\text{O}_2$) calculated 222.16198, found 222.16111.

6-Non-1-enyl-4-triethylsilyloxy-tetrahydro-pyran-2-one (10)

To a solution of **9** (0.536 g, 2.23 mmol), triethylamine (1.9 mL, 13.4 mmol), and DMAP (23.5 mg, 0.19 mmol), in THF (22 mL) at room temperature was added chlorotriethylsilane (1.1 mL, 6.69 mmol). The reaction was left to stir overnight at room temperature. The resulting mixture was then quenched with saturated NH_4Cl _(aq). The layers were separated and the aqueous layer was extracted with ether. The combined organic extracts were dried (MgSO_4), and concentrated to dryness. Flash column chromatography with 10% ethyl acetate/90% hexanes as eluent afforded **10** (0.783 g, 99%) as a clear oil. ^1H NMR (300 MHz, benzene D₆): δ 5.58 – 5.68 (m, 1H), 5.35 (ddt, J = 15.3, 6.7, 1.4 Hz, 1H), 4.12 (ddd, J = 10.7, 6.7, 3.2 Hz, 1H), 3.61 – 3.70 (m, 1H), 2.55 (ddd, J = 16.9, 5.8, 1.5 Hz, 1H), 2.28 (dd, J = 16.9, 7.9 Hz, 1H), 1.91 (q, J = 6.7 Hz, 2H), 1.77 (dddd, J = 13.5, 5.1, 3.2, 1.5 Hz, 1H), 1.44 (ddd, J = 13.6, 11.6, 9.1 Hz, 1H), 1.23 – 1.34 (m, 10H), 0.86 – 0.92 (m, 12H), 0.45 (q, J = 8.0 Hz, 8H); ^{13}C NMR (75 MHz, benzene D₆): δ 168.7 (C₄), 134.0 (CH), 128.5 (CH), 76.6 (CH), 64.7 (CH), 40.6 (CH₂), 39.6 (CH₂), 32.4 (CH₂), 32.1 (CH₂), 29.5 (CH₂), 29.5 (CH₂), 29.2 (CH₂), 23.0 (CH₂), 14.3 (CH₃), 6.9 (CH₃), 5.0 (CH₂); IR (neat) 2956 (s), 2927 (s), 2876 (s), 2856 (s), 2734 (w), 1743 (s), 1675 (w), 1459 (m), 1416 (w), 1380 (w), 1349 (w), 1284 (w), 1234 (s), 1164 (w), 1137 (w), 1084 (s), 1044 (m), 1016 (m), 1009 (m), 968 (m), 950 (w), 927 (w), 887 (w), 849 (w), 747 (s), 729 (m), 673 (w); HRMS (EI) m/z $\text{M}^+ - \text{C}_2\text{H}_5$ ($\text{C}_{18}\text{H}_{33}\text{O}_3\text{Si}$) calculated 325.21990, found 325.21829.

6-(1-Ethoxy-vinyl)-2-non-1-enyl-3,4-dihydro-2H-pyran-4-ol (4a)

To a solution of THF (0.53 mL) in ethyl vinyl ether (4.6 mL, 1.7 mmol) cooled to -78°C was added *t*-BuLi (1.7 M in pentane, 14.3 mL, 24.3 mmol) dropwise. The reaction was warmed to 0°C and left to stir at this temperature for 45 minutes. The resulting mixture was cooled to -78°C and to this was added a solution of **10** (0.861 g, 2.43 mmol) in THF (6 mL) via canula with a rinse of three 6 mL portions of THF. The solution was left to stir at -78°C for 20 minutes. Following this, the reaction was quenched with saturated NH₄Cl_(aq) while still at -78°C. The resulting mixture was warmed to room temperature and left to stir for 30 minutes. The layers were then separated and the aqueous layer was extracted with ether. The combined organic extracts were dried (MgSO₄), and concentrated to dryness. The crude product was then dissolved in dichloromethane (6 mL) and added via canula to a flask containing DMAP (27.2 mg, 0.223 mmol) with a rinse of three 6 mL portions of dichloromethane. The resulting solution was cooled to 0°C. Thionyl chloride (0.17 mL, 2.3 mmol) was then added and left to stir for 5 minutes at 0°C. The reaction was quenched with saturated NH₄Cl_(aq) while still at 0°C and the resulting mixture was warmed to room temperature. The layers were separated and the aqueous layer was extracted with dichloromethane. The combined organic extracts were dried (MgSO₄), and concentrated to dryness. Flash column chromatography with 2% ethyl acetate/98% hexanes as eluent afforded 0.513 g of a clear oil product mixture as indicated by TLC. The oil was dissolved in THF (12.6 mL) and to the resulting solution was added TBAF (1 M in THF, 1.9 mL) dropwise. Immediately after addition, the reaction was concentrated to dryness and flash column chromatography with 20% ethyl acetate/80% hexanes as eluent afforded **4a** (0.310 g, 42% from **10**) as a yellow solid. ¹H NMR (300 MHz, benzene D₆): δ 5.62 – 5.72 (m, 2H), 5.53 (dd, *J* = 15.6, 6.0 Hz, 1H), 5.10 (s, 1H), 4.25 – 4.29 (m, 2H), 4.13 (s, 1H), 3.46 (q, *J* = 7.0 Hz, 2H), 1.86 – 1.95 (m, 3H), 1.52 – 1.64 (m, 1H), 1.22 – 1.27 (m, 10H), 1.06 (t, *J* = 7.0 Hz, 3H), 0.88 – 0.92 (m, 3H); ¹³C NMR (75 MHz, benzene D₆): δ 155.3 (C₄), 148.9 (C₄), 132.8 (CH), 129.6 (CH), 102.6 (CH), 83.5 (CH₂), 75.8 (CH), 63.5 (CH), 63.1 (CH₂), 38.3 (CH₂), 32.6 (CH₂), 32.2 (CH₂), 29.5 (CH₂), 29.5 (CH₂), 29.4 (CH₂), 23.0 (CH₂), 14.4 (CH₃), 14.3 (CH₃); IR (neat) 3310 (m), 3210 (w), 2980 (w), 2954 (m), 2921 (s), 2852 (m), 1596 (m), 1467 (w), 1455 (w), 1448 (w), 1380 (w), 1355 (w), 1328 (w), 1297 (m), 1274 (w), 1245 (w), 1195 (w), 1156 (w), 1136 (w), 1091 (w), 1048 (w), 1014 (w), 973 (w), 963 (w), 909 (w), 853 (w), 820 (w), 803 (m), 720 (w); HRMS (EI) *m/z* M⁺ (C₁₈H₃₀O₃) calculated 294.21950, found 294.21827; mp = 60.4 – 61.9°C.

2-Non-1-enyl-6-vinyl-3,4-dihydro-2*H*-pyran-4-ol (**4b**)

To a solution of **10** (0.427 g, 1.20 mmol) in THF (8 mL) cooled to -78°C was added vinylmagnesium bromide (0.9 M in THF, 4.0 mL, 3.6 mmol) dropwise. The resulting mixture was left to stir for 20 minutes at -78°C. Following this, the reaction was quenched with saturated NH₄Cl_(aq) while still at -78°C. The resulting mixture was warmed to room temperature and left to stir for 30 minutes. The layers were then separated and the aqueous layer was extracted with ether. The combined organic extracts were dried (MgSO₄), and concentrated to dryness. The crude product was then dissolved in dichloromethane (2.9 mL) and added via canula to a flask containing DMAP (0.417 g,

3.41 mmol) with a rinse of three 2.9 mL portions of dichloromethane. The resulting solution was cooled to 0°C. Thionyl chloride (0.08 mL 1.1 mmol) was then added dropwise and left to stir for 5 minutes at 0°C. The reaction was quenched with saturated $\text{NH}_4\text{Cl}_{(\text{aq})}$ while still at 0°C and the resulting mixture was warmed to room temperature. The layers were separated and the aqueous layer was extracted with dichloromethane. The combined organic extracts were dried (MgSO_4), and concentrated to dryness. Flash column chromatography with 5% ethyl acetate/95% hexanes as eluent afforded 0.163 g of a clear oil product mixture as indicated by TLC. The oil was dissolved in THF (3.7 mL) and to the resulting solution was added TBAF (1 M in THF, 0.64 mL) dropwise. Immediately after addition, the reaction was concentrated to dryness and flash column chromatography with 20% ethyl acetate/80% hexanes as eluent afforded **4b** (99.8 mg, 35% from **10**) as a clear oil. ^1H NMR (300 MHz, benzene D_6): δ 6.00 (dd, $J = 17.2, 10.5$ Hz, 1H), 5.84 (dd, $J = 17.2, 2.2$ Hz, 1H), 5.61 – 5.71 (m, 1H), 5.48 – 5.56 (m, 1H), 5.04 (dd, $J = 10.5, 2.2$ Hz, 1H), 4.72 (s, 1H), 4.19 – 4.26 (m, 2H), 1.83 – 1.95 (m, 3H), 1.48 – 1.58 (m, 1H), 1.22 – 1.32 (m, 10H), 0.87 – 0.92 (m, 3H); ^{13}C NMR (75 MHz, benzene D_6): δ 151.7 (C₄), 132.8 (CH), 132.7 (CH), 129.6 (CH), 114.5 (CH₂), 107.1 (CH), 73.4 (CH), 63.6 (CH), 38.4 (CH₂), 32.6 (CH₂), 32.2 (CH₂), 29.5 (CH₂), 29.5 (CH₂), 29.4 (CH₂), 23.0 (CH₂), 14.3 (CH₃); IR (neat) 3327 (m), 3101 (w), 3023 (w), 2956 (m), 2925 (s), 2871 (m), 2855 (s), 1654 (w), 1600 (m), 1465 (w), 1457 (w), 1438 (w), 1409 (w), 1378 (w), 1360 (w), 1336 (w), 1304 (w), 1284 (m), 1245 (w), 1202 (w), 1174 (w), 1143 (w), 1094 (w), 1047 (m), 1008 (w), 980 (m), 967 (m), 913 (m), 883 (w), 790 (w), 723 (w); HRMS (EI) m/z M⁺ ($\text{C}_{16}\text{H}_{26}\text{O}_2$) calculated 250.19328, found 250.19553.

7-Ethoxy-5-non-1-enyl-1,2,4,5,8,8a,8b,8c-octahydro-3aH-3,6-dioxa-cyclopenta[bc]acenaphthylen-2a-ol (11) (Method B)

Dichloromethane (0.9 mL) was added to a flask containing $\text{MgBr}_2\text{-OEt}_{2(\text{s})}$ (90.8 mg, 0.352 mmol). Triethylamine (0.10 mL, 0.70 mmol) was also added and the resulting mixture was left to stir at room temperature for 15 minutes. A solution of **4a** (51.6 mg, 0.175 mmol) in dichloromethane (0.3 mL) was then added via canula with a rinse of three 0.2 mL portions of dichloromethane. To this was added 2-cyclopenten-1-one (0.06 mL, 0.716 mmol). The reaction was left to stir for 24 hours at room temperature. It was then quenched with saturated $\text{NH}_4\text{Cl}_{(\text{aq})}$. The layers were separated and the aqueous layer was extracted with dichloromethane. The combined organic extracts were dried (MgSO_4), and concentrated to dryness. Flash column chromatography with 30% ethyl acetate/70% hexanes as eluent afforded **11** (14.9 mg, 23%) as a clear oil. ^1H NMR (300 MHz, benzene D_6): δ 5.77 (dt, $J = 15.4, 6.4$ Hz, 1H), 5.59 (dd, $J = 15.6, 6.2$ Hz, 1H), 4.33 – 4.41 (m, 1H), 4.11 – 4.16 (m, 1H), 3.92 – 4.07 (m, 2H), 2.79 – 2.85 (m, 1H), 2.61 (t, $J = 9.9$ Hz, 1H), 2.31 – 2.39 (m, 1H), 2.27 (br s, 1H), 2.03 – 2.18 (m, 2H), 1.89 – 2.00 (m, 4H), 1.76 – 1.85 (m, 2H), 1.22 – 1.53 (m, 13H), 0.87 – 0.91 (m, 3H); ^{13}C NMR (75 MHz, benzene D_6): δ 132.7 (C₄), 132.6 (CH), 131.6 (C₄), 130.7 (CH), 117.7 (C₄), 74.9 (CH), 72.2 (CH), 65.5 (CH₂), 50.4 (CH), 39.4 (CH), 38.6 (CH₂), 36.5 (CH), 35.5 (CH₂), 32.6 (CH₂), 32.2 (CH₂), 32.0 (CH₂), 29.5 (CH₂), 29.4 (CH₂), 23.0 (CH₂), 16.2

(CH₃), 14.3 (CH₃); IR (neat) 3401 (w), 2954 (m), 2926 (s), 2855 (m), 1455 (w), 1440 (w), 1378 (w), 1332 (w), 1304 (w), 1249 (w), 1184 (m), 1167 (m), 1129 (m), 1079 (m), 1044 (m), 965 (m), 927 (w), 862 (w); HRMS (EI) *m/z* M⁺ (C₂₃H₃₆O₄) calculated 376.26136, found 376.26326.

5-Ethoxy-7-non-1-enyl-2-oxo-2,2a,3,4,7,8,8a,8b-octahydro-1,6-dioxa-acenaphthylene-3-carboxylic acid phenylamide (12) (Method B)

Dichloromethane (0.4 mL) was added to a flask containing MgBr₂-OEt_{2(s)} (39.2 mg, 0.152 mmol). Triethylamine (0.04 mL, 0.29 mmol) was also added and the resulting mixture was left to stir at room temperature for 15 minutes. A solution of **4a** (21.5 mg, 73.0 μ mol) in dichloromethane (0.1 mL) was then added via canula with a rinse of three 0.1 mL portions of dichloromethane. Following this, a solution of *N*-phenylmaleimide (26.2 mg, 0.151 mmol) in dichloromethane (0.1 mL) was added via canula with rinse of three 0.1 mL portions of dichloromethane. The reaction was left to stir for 24 hours at room temperature. It was then quenched with saturated NH₄Cl_(aq). The layers were separated and the aqueous layer was extracted with dichloromethane. The combined organic extracts were dried (MgSO₄), and concentrated to dryness. Flash column chromatography with 40% ethyl acetate/60% hexanes as eluent afforded **12** (27.3 mg, 80%) as a white solid. ¹H NMR (300 MHz, CDCl₃): δ 9.68 (s, 1H), 7.55 – 7.58 (m, 2H), 7.27 – 7.33 (m, 2H), 7.06 – 7.11 (m, 1H), 5.74 (dt, *J* = 15.3, 6.8 Hz, 1H), 5.47 (dd, *J* = 15.4, 7.2 Hz, 1H), 4.91 (ddd, *J* = 7.9, 6.1, 6.1 Hz, 1H), 4.43 (ddd, *J* = 9.8, 7.1, 2.4 Hz, 1H), 3.82 – 3.97 (m, 2H), 3.45 (dd, *J* = 6.6, 2.8 Hz, 1H), 3.31 – 3.36 (m, 1H), 2.91 (ddd, *J* = 10.2, 7.3, 2.9 Hz, 1H), 2.53 – 2.66 (m, 2H), 2.38 (ddd, *J* = 14.8, 8.1, 2.8 Hz, 1H), 2.03 (q, *J* = 6.9 Hz, 2H), 1.80 (ddd, *J* = 14.8, 10.4, 6.2 Hz, 1H), 1.19 – 1.39 (m, 13H), 0.83 – 0.88 (m, 3H); ¹³C NMR (75 MHz, CDCl₃): δ 177.0 (C₄), 169.9 (C₄), 138.0 (C₄), 135.3 (C₄), 135.0 (CH), 130.1 (C₄), 128.9 (CH), 127.8 (CH), 124.3 (CH), 120.0 (CH), 75.9 (CH), 73.5 (CH), 65.6 (CH₂), 42.1 (CH), 41.7 (CH), 38.3 (CH), 34.8 (CH₂), 32.2 (CH₂), 31.8 (CH₂), 29.1 (CH₂), 29.1 (CH₂), 28.9 (CH₂), 26.8 (CH₂), 22.6 (CH₂), 15.5 (CH₃), 14.1 (CH₃); 3334 (w), 3204 (w), 3141 (w), 3057 (w), 2957 (m), 2926 (s), 2871 (m), 2855 (m), 1770 (s), 1714 (s), 1685 (m), 1600 (m), 1541 (m), 1500 (m), 1466 (w), 1456 (w), 1442 (s), 1378 (m), 1354 (w), 1337 (w), 1322 (m), 1239 (w), 1199 (s), 1129 (m), 1081 (w), 1054 (w), 1019 (m), 969 (w), 946 (w), 898 (w), 877 (w), 836 (w), 788 (w), 754 (m), 719 (w), 693 (m); HRMS (EI) *m/z* M⁺ (C₂₈H₃₇NO₅) calculated 467.26717, found 467.26698; mp = 165.4 – 167.2°C.

7-Non-1-enyl-2-oxo-2,2a,3,4,7,8,8a,8b-octahydro-1,6-dioxa-acenaphthylene-3-carboxylic acid phenylamide (13) (Method B)

Dichloromethane (0.8 mL) was added to a flask containing MgBr₂-OEt_{2(s)} (86.0 mg, 0.333 mmol). Triethylamine (0.09 mL, 0.65 mmol) was also added and the resulting mixture was left to stir at room temperature for 15 minutes. A solution of **4b** (41.4 mg, 0.165 mmol) in dichloromethane (0.2 mL) was then added via canula with a rinse of two 0.2 mL portions of dichloromethane. Following this, a solution of *N*-phenylmaleimide

(59.8 mg, 0.345 mmol) in dichloromethane (0.1 mL) was added via canula with rinse of two 0.1 mL portions of dichloromethane. The reaction was left to stir for 24 hours at room temperature. It was then quenched with saturated $\text{NH}_4\text{Cl}_{(\text{aq})}$. The layers were separated and the aqueous layer was extracted with dichloromethane. The combined organic extracts were dried (MgSO_4), and concentrated to dryness. Flash column chromatography with 40% ethyl acetate/60% hexanes as eluent afforded the Diels-Alder adduct **13** (40.6 mg, 58%) as a white solid. ^1H NMR (300 MHz, CDCl_3): δ 9.69 (s, 1.59), 7.57 (d, J = 7.6 Hz, 2H), 7.30 (dd, J = 7.6, 7.4 Hz, 2H), 7.08 (t, J = 7.4 Hz, 1H), 5.75 (dt, J = 15.1, 6.8 Hz, 1H), 5.42 (dd, J = 15.3, 7.3 Hz, 1H), 5.27 – 5.28 (m, 1H), 4.91 – 4.98 (m, 1H), 4.38 – 4.44 (m, 1H), 3.53 (dd, J = 7.0, 2.9 Hz, 1H), 3.30 (m, 1H), 2.88 (ddd, J = 11.1, 6.3, 2.8 Hz, 1H), 2.36 – 2.51 (m, 3H), 2.00 – 2.07 (m, 2H), 1.75 – 1.86 (m, 1H), 1.25 – 1.60 (m, 10H), 0.84 – 0.88 (m, 3H); ^{13}C NMR (75 MHz, CDCl_3): δ 177.1 (C₄), 170.8 (C₄), 146.2 (C₄), 138.1 (C₄), 135.6 (CH), 128.9 (CH), 127.5 (CH), 124.2 (CH), 120.0 (CH), 104.0 (CH), 75.6 (CH), 73.3 (CH), 42.6 (CH), 41.9 (CH), 38.7 (CH), 35.0 (CH₂), 32.2 (CH₂), 31.8 (CH₂), 29.1 (CH₂), 28.8 (CH₂), 23.8 (CH₂), 22.6 (CH₂), 14.1 (CH₃); IR (neat) 3357 (m), 2955 (m), 2923 (s), 2854 (s), 1770 (s), 1750 (m), 1712 (s), 1689 (s), 1601 (m), 1639 (s), 1501 (m), 1441 (s), 1386 (s), 1352 (w), 1340 (w), 1322 (w), 1243 (w), 1207 (s), 1179 (m), 1166 (m), 1136 (m), 1079 (w), 1026 (w), 965 (w), 946 (w), 896 (w), 880 (w), 824 (w), 795 (w), 751 (m), 692 (m); HRMS (EI) m/z M^+ ($\text{C}_{26}\text{H}_{33}\text{NO}_4$) calculated 423.24096, found 423.24305; mp = 137.3 – 138.9°C.

2-Benzyl-5-ethoxy-9-hydroxy-7-non-1-enyl-3a,7,8,9,9a,9b-hexahydro-4H-pyranol[3,2-e]isoindole-1,3-dione (14) (Method A)

Vinyl magnesium bromide (0.96 mL, 0.83 mmol) was added to a solution of **4a** (204 mg, 0.69 mmol) in toluene (7 mL) at –78°C and stirred 30 minutes. N-benzylmaleimide (1.30 g, 6.9 mmol) was added to the solution and stirred for 18 hours, letting the mixture to reach room temperature. The reaction was quenched with water. The aqueous layer was extracted 3 times with diethyl ether (3 X 10 mL). The organic layer was dried over anhydrous MgSO_4 , filtrated and the solvent evaporated. The crude product was purified by flash column chromatography (40 % EtOAc/hexanes) to afford **14** (240 mg, 72% yield) as an oil. ^1H NMR (300 MHz, benzene D₆): δ 7.32 (d, J = 7.4 Hz, 2H), 7.14-7.02 (m, 3H), 5.56 (dt, J = 15.5, 7.1 Hz, 1H), 5.33 (dd, J = 15.4, 6.2 Hz, 1H), 4.44-4.33 (m, 3H), 4.30-4.02 (m, 1H), 3.87-3.82 (m, 1H), 3.75-3.54 (m, 2H), 2.85 (dd, J = 6.1, 8.6 Hz, 1H), 2.73 (dd, J = 15.3, 1.4 Hz, 1H), 2.35 (t, J = 8.0 Hz, 1H), 2.18 (t, J = 7.0 Hz, 1H), 1.94-1.85 (m, 3H), 1.81-1.74 (m, 2H), 1.35-1.11 (m, 10H), 1.04 (t, J = 7.0 Hz, 3H), 0.89 (t, J = 6.3 Hz, 3H); ^{13}C NMR (75 MHz, benzene D₆): δ 180.8 (C₄), 178.5 (C₄), 136.2 (C₄), 137.7 (C₄), 132.4 (CH), 130.6 (C₄), 130.2 (CH), 129.0 (CH), 128.9 (CH), 128.6 (CH), 128.4 (CH), 128.3 (CH), 127.1 (CH), 76.1 (CH), 65.9 (CH₂), 65.8 (CH), 42.3 (CH₂), 41.7 (CH), 40.7 (CH), 38.6 (CH₂), 38.4 (CH), 32.2 (CH₂), 31.9 (CH₂), 29.2 (CH₂), 29.1 (CH₂), 27.7 (CH₂), 22.7 (CH₂), 15.6 (CH₃), 14.0 (CH₃); IR (CH₂Cl₂) 3446 (m), 3093 (w), 3065 (w), 3034 (w), 2956 (s), 2926 (s), 2855 (s), 1771 (m), 1689 (s), 1606 (w), 1587 (w), 1497 (m), 1456 (m), 1434 (m), 1405 (m), 1349 (m), 1181 (m), 1102 (m), 1064 (m), 1019 (m); HRMS (EI) m/z M^+ ($\text{C}_{29}\text{H}_{39}\text{NO}_5$) calculated 481.28282, found 481.28236.

2-Benzyl-5-ethoxy-9-hydroxy-7-non-1-enyl-3a,7,8,9,9a,9b-hexahydro-4H-pyranolo[3,2-e]isoindole-1,3-dione (14) (Method B)

Reagents and conditions, see method B above: Diene **4a** (33.5 mg, 0.114 mmol), MgBr₂-Et₂O (58.7 mg, 0.227 mmol), Et₃N (0.063 mL, 0.445 mmol), NBM (85.1 mg, 0.445 mmol), CH₂Cl₂ (1 mL) gave cycloadduct **14** (43.6 mg, 79%).

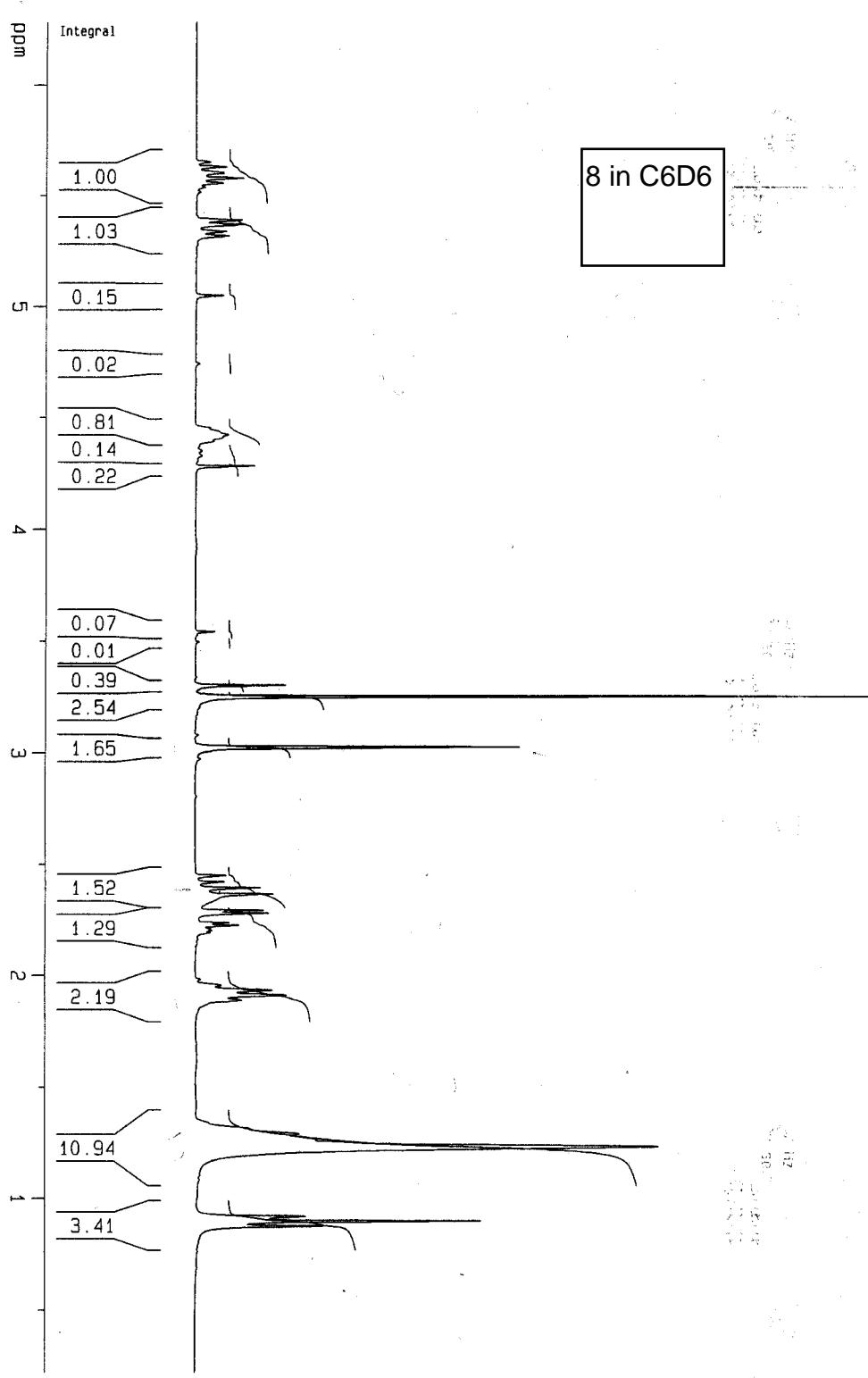
8-Ethoxy-9-heptyl-13-hydroxy-tricyclo[6.5.1.02,6]tetradec-10-ene-3,14-dione (15)

Triethylamine (1 drop) was added to a solution of the **11** (11.8 mg, 31.3 μ mol) in toluene (3 mL) in a sealed tube. The solution was degassed with argon for 15 minutes. After this, the tube was sealed and placed in a wax bath at 160°C and the solution was stirred for 24 hours. The resulting solution was then cooled to room temperature and concentrated to dryness under reduced pressure to give brown oil. Flash column chromatography of the crude product with 30% ethyl acetate/70% hexanes as eluent afforded **15** as a clear oil (8.3 mg, 70%). ¹H NMR (300 MHz, benzene D₆): δ 5.48 – 5.60 (m, 2H), 5.27 (d, J = 4.6 Hz, 1H), 4.25 – 4.33 (m, 1H), 3.56 – 3.66 (m, 1H), 3.15 – 3.25 (m, 1H), 2.69 (dd, J = 11.1, 5.3 Hz, 1H), 2.31 – 2.54 (m, 3H), 2.15 – 2.24 (m, 1H), 1.94 (dd, J = 14.0, 6.4 Hz, 1H), 1.64 – 1.86 (m, 3H), 1.46 – 1.61 (m, 2H), 1.17 – 1.39 (m, 15H), 0.84 – 0.91 (m, 3H), 0.64 – 0.82 (m, 1H); ¹³C NMR (75 MHz, CDCl₃): δ 219.2 (C4), 209.3 (C4), 136.4 (CH), 127.9 (CH), 88.5 (C₄), 77.4 (C₄), 70.1 (CH), 60.9 (CH₂), 55.5 (CH), 50.3 (CH), 46.0 (CH), 39.2 (CH), 37.5 (CH₂), 36.1 (CH₂), 32.7 (CH₂), 31.8 (CH₂), 29.6 (CH₂), 29.3 (CH₂), 28.9 (CH₂), 28.2 (CH₂), 27.2 (CH₂), 22.7 (CH₂), 16.1 (CH₃), 14.1 (CH₃); IR (neat) 3395 (w), 3021 (w), 2926 (s), 2870 (m), 2856 (m), 1718 (s), 1457 (m), 1163 (m), 1089 (s), 964 (w), 885 (w), 784 (w), 695 (m); HRMS (EI) *m/z* M⁺ (C₂₃H₃₆O₄) calculated 376.26136, found 376.26303.

4-Benzyl-8-ethoxy-9-heptyl-13-hydroxy-4-aza-tricyclo[6.5.1.02,6]tetradec-10-ene-3,5,14-trione (16)

Triethylamine (1 drop) was added to a solution of **14** (18 mg, 0.04mmol) in toluene (3 mL) in a sealed tube. The solution was degassed with argon for 15 minutes. The mixture was placed in a wax bath at 160°C and stirred 24 hours and the solvent evaporated. The crude product was purified by gradient flash column chromatography (40-50 % EtOAc/hexanes) to afford **16** (10 mg, 55 % yield) as an oil. ¹H NMR (300 MHz, benzene D₆): δ 7.45 (d, J = 7.4 Hz, 2H), 7.15-6.88 (m, 3H), 5.73 (d, J = 12.7 Hz, 1H), 5.39-5.28 (m, 2H), 4.62 (d, J = 13.7 Hz, 1H), 4.39 (d, J = 13.6 Hz, 1H), 4.26 (tt, J =12.7, 4.0 Hz, 1H), 3.58-3.50 (m, 2H), 3.23 (q, J = 7.8 Hz, 1H), 2.68 (dd, J = 10.6, 12.5 Hz, 1H), 2.57 (d, J = 14.7 Hz, 1H), 2.47-2.36 (m, 1H), 2.26-2.20 (m, 1H), 2.15-2.06 (m, 2H), 1.65-1.48 (m, 3H), 1.27-1.17 (m, 13H), 0.90 (t, J = 7.1Hz, 3H); ¹³C NMR (75 MHz, benzene D₆): δ 205.8 (C₄), 181.2 (C₄), 177.8 (C₄), 136.6 (C₄), 135.4 (C₄), 129.8 (CH), 129.0 (CH), 128.7 (CH), 128.3 (CH), 127.7 (CH), 126.9 (CH), 85.8 (C₄), 74.6 (CH), 60.0 (CH₂), 56.2 (CH), 43.5 (CH₂), 39.6 (CH), 38.9 (CH), 37.8 (CH), 34.1 (CH₂), 32.3 (CH₂), 30.3 (CH₂), 29.6(CH₂), 27.7 (CH₂), 27.4 (CH₂), 26.7 (CH₂), 23.1 (CH₂), 16.2 (CH₃), 14.3

(CH₃); IR (CH₂Cl₂) 3430 (m), 3066 (w), 3027 (w), 2927 (s), 2855 (m), 1771 (m), 1716 (s), 1688 (s), 1605 (w), 1587 (w), 1497 (m), 1456 (m), 1433(m), 1401 (m), 1351 (m), 1125 (m), 1075 (m), 1052 (m); HRMS (EI) *m/z* M⁺ (MC₂₉H₃₉NO₅) calculated 481.28282, found 481.28223.



Current Data Parameters
 NAME pa2-140
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date 2003/03
 Time 18:31
 INSTRUM av300
 PROBHD 5 mm QNP 1H/1
 PULPROG zg30
 TD 30720
 SOLVENT C6D6

NS 16
 DS 0
 SWH 5081.301 Hz
 FIDRES 0.165407 Hz
 AQ 3.022980 sec
 RG 143.7
 DW 98.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.0000000 sec

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

NUC1 1H
 P1 10.50 usec
 PL1 -3.00 dB
 SF01 300.1319477 MHz

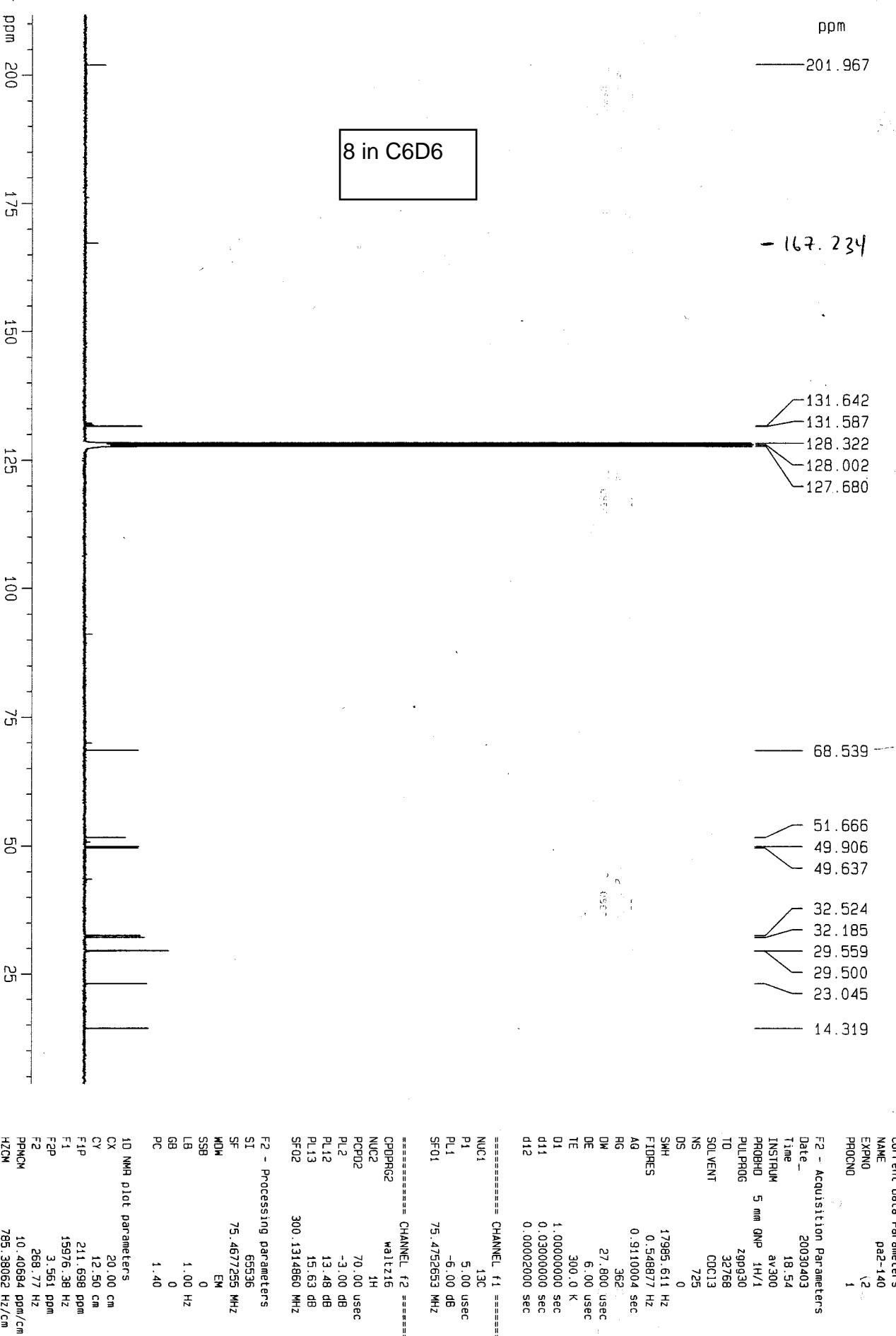
F2 - Processing parameters

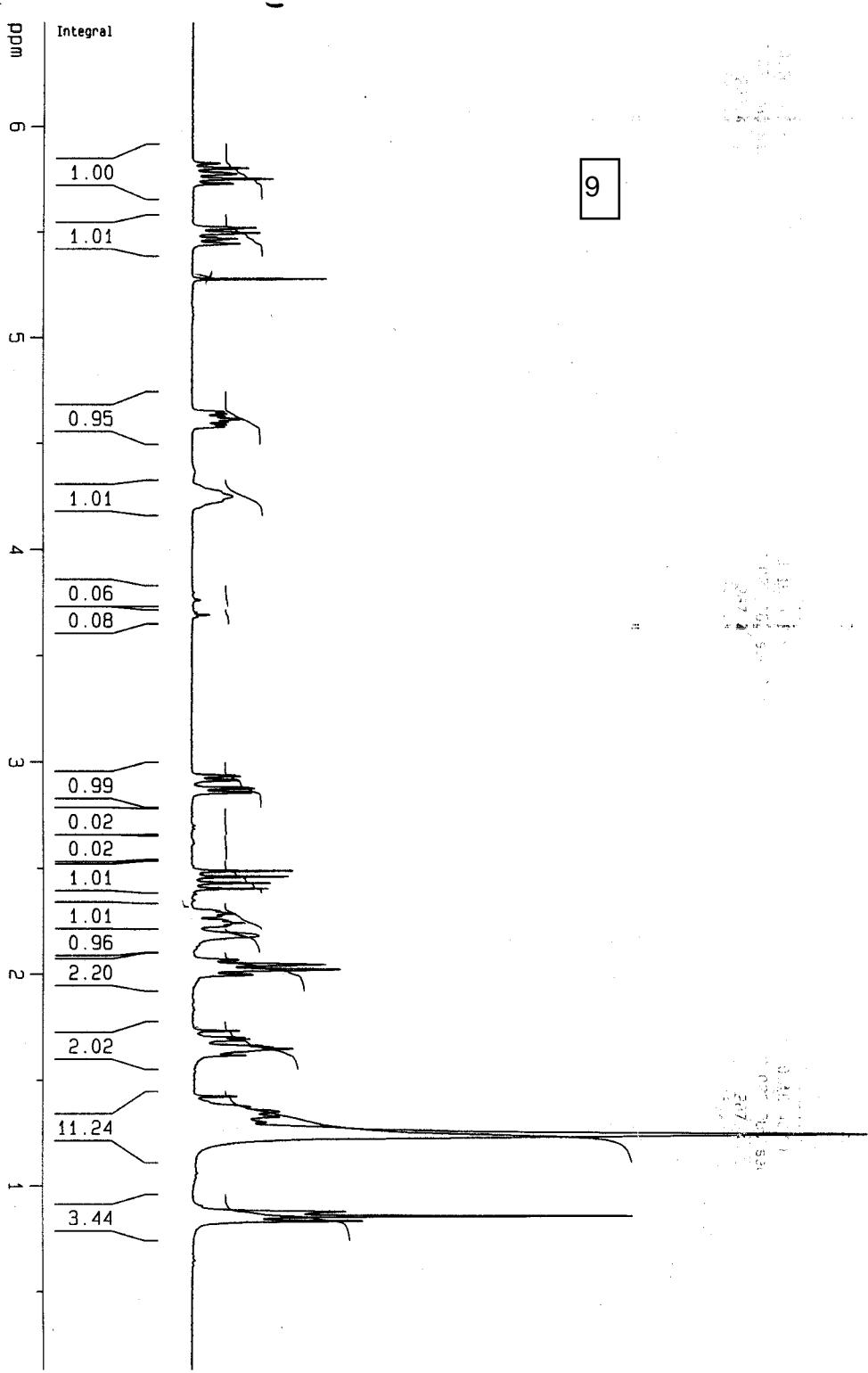
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1D NMR plot parameters

CX 20.00 cm
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 F1P 6.279 ppm
 F1 1884.53 Hz
 F2P 0.220 ppm
 F2 66.04 Hz
 F2PPCM 0.30295 ppm/cm
 HZCM 90.92460 Hz/cm

13C with proton decoupling





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

NUC1 1H
 P1 10.50 usec
 PL1 -3.00 dB
 SF 65536 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

F2 - Processing parameters

SI 300.1300002 MHz
 SF 300.1300002 MHz
 EM 0
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

1D NMR plot parameters

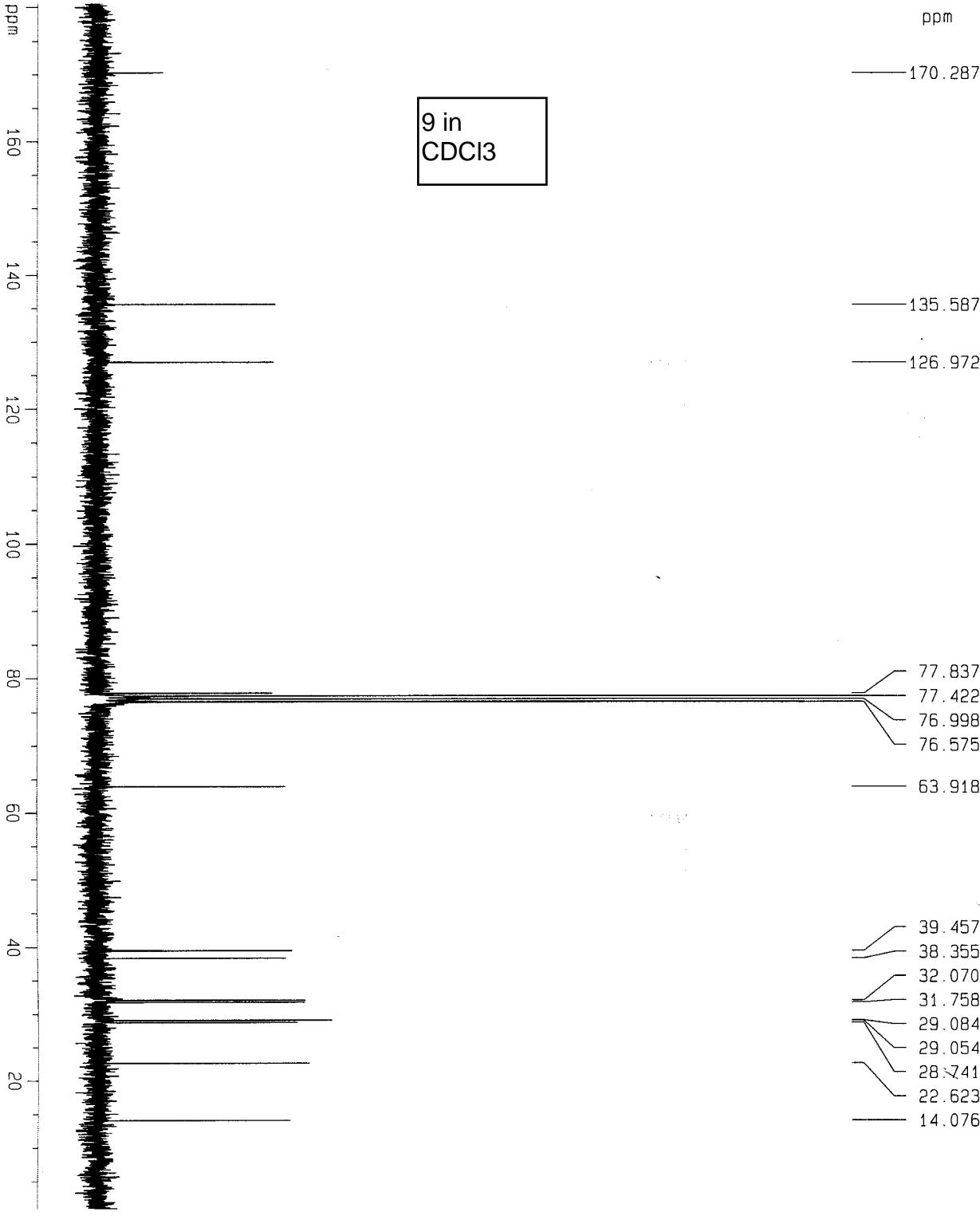
CX 20.00 cm
 CY 10.00 cm
 F1P 6.489 ppm
 F1 19.47.55 Hz
 F2P 0.134 ppm
 F2 40.30 Hz
 F2PPCM 0.31774 ppm/cm
 HZCM 95.36236 Hz/cm

Current Data Parameters
 NAME pa2-158
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date 20030422
 Time 17.06
 INSTRUM av300
 PROBHD 5 mm QNP 1H/1
 PULPROG zg30
 TD 30720
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 5081.301 Hz
 FIDRES 0.165407 Hz
 AQ 3.028880 sec
 R6 287.4
 DW 98.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.0000000 sec

13C with proton decoupling



Current Data Parameters

NAME	pe2-158
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

TD	20480
Time	17.16
INSTRUM	av300
PROBHD	5 mm QNP 1H/1
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	354
DS	0
SWH	17985.611 Hz
FIDRES	0.548877 Hz
AQ	0.9110004 sec
RG	574.7
DW	27.800 usec
DE	6.00 usec
TE	300.0 K
DI	1.0000000 sec
d11	0.03000000 sec
d12	0.00002000 sec

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

NUC1	13C
P1	5.00 usec
PL1	-6.00 dB
SF01	75.4752653 MHz

===== CHANNEL f2 =====

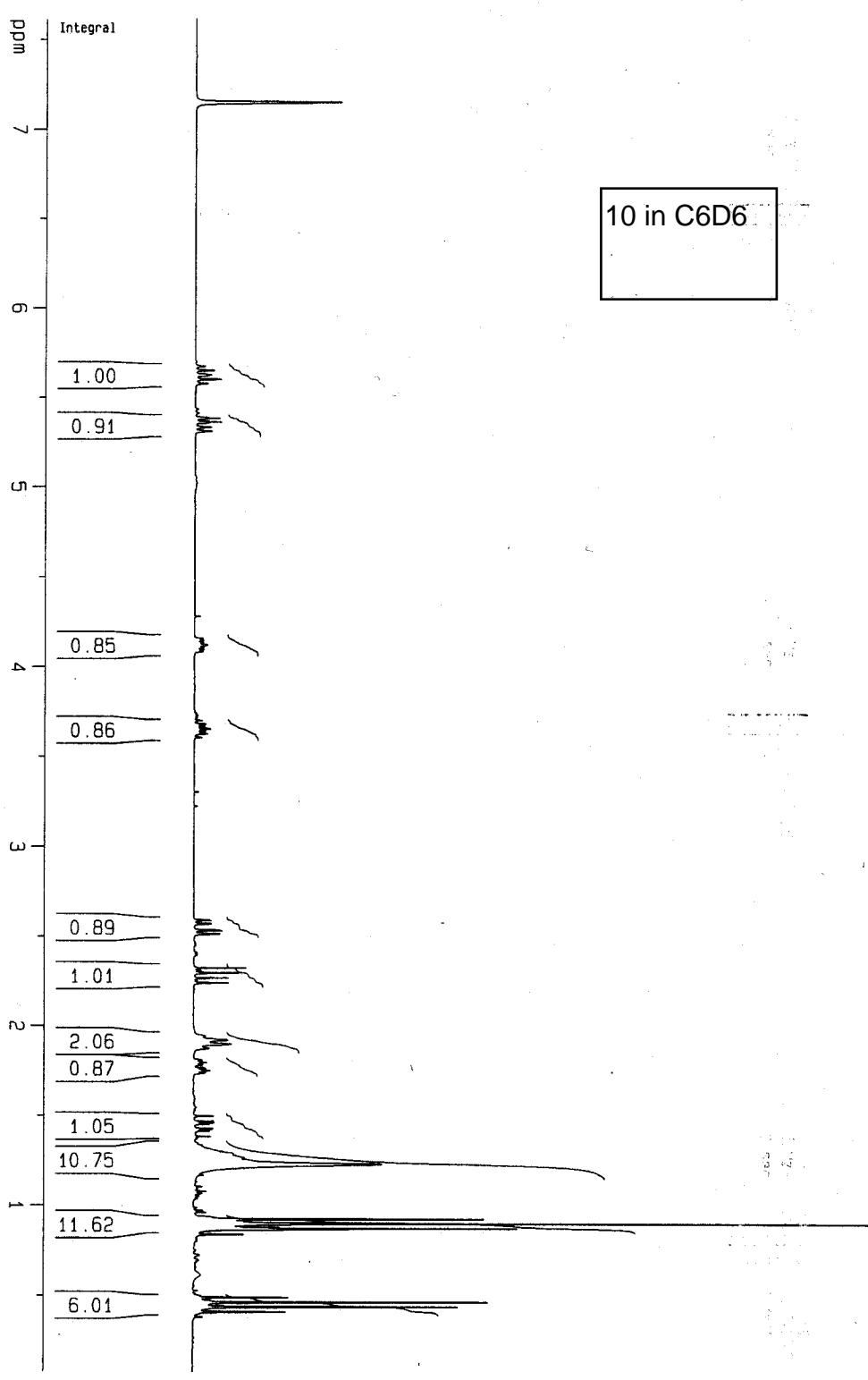
NUC2	1H
PCP02	70.00 usec
PL2	-3.00 dB
PL12	13.48 dB
PL13	15.63 dB
SF02	300.1314860 MHz

F2 - Processing parameters

SI	65536
SF	75.467746 MHz
MDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	4.00 cm
F1P	180.554 ppm
F1	13654.53 Hz
F2P	0.968 ppm
F2	73.04 Hz
PPCM	8.97833 ppm/cm
HZCM	677.57446 Hz/cm



Current Data Parameters

NAME pac-168
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date 20040115
Time 17.59
INSTRUM spect
PROBOD 5 mm QNP 1H/1
PULPROG zg30
TD 30720

SOLVENT C6D6
NS 16
DS 0
SWH 5081.301 Hz
FIDRES 0.165407 Hz
AQ 3.0228980 sec
RG 161.3
DW 98.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.0000000 sec

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

NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SF01 300.1319477 MHz

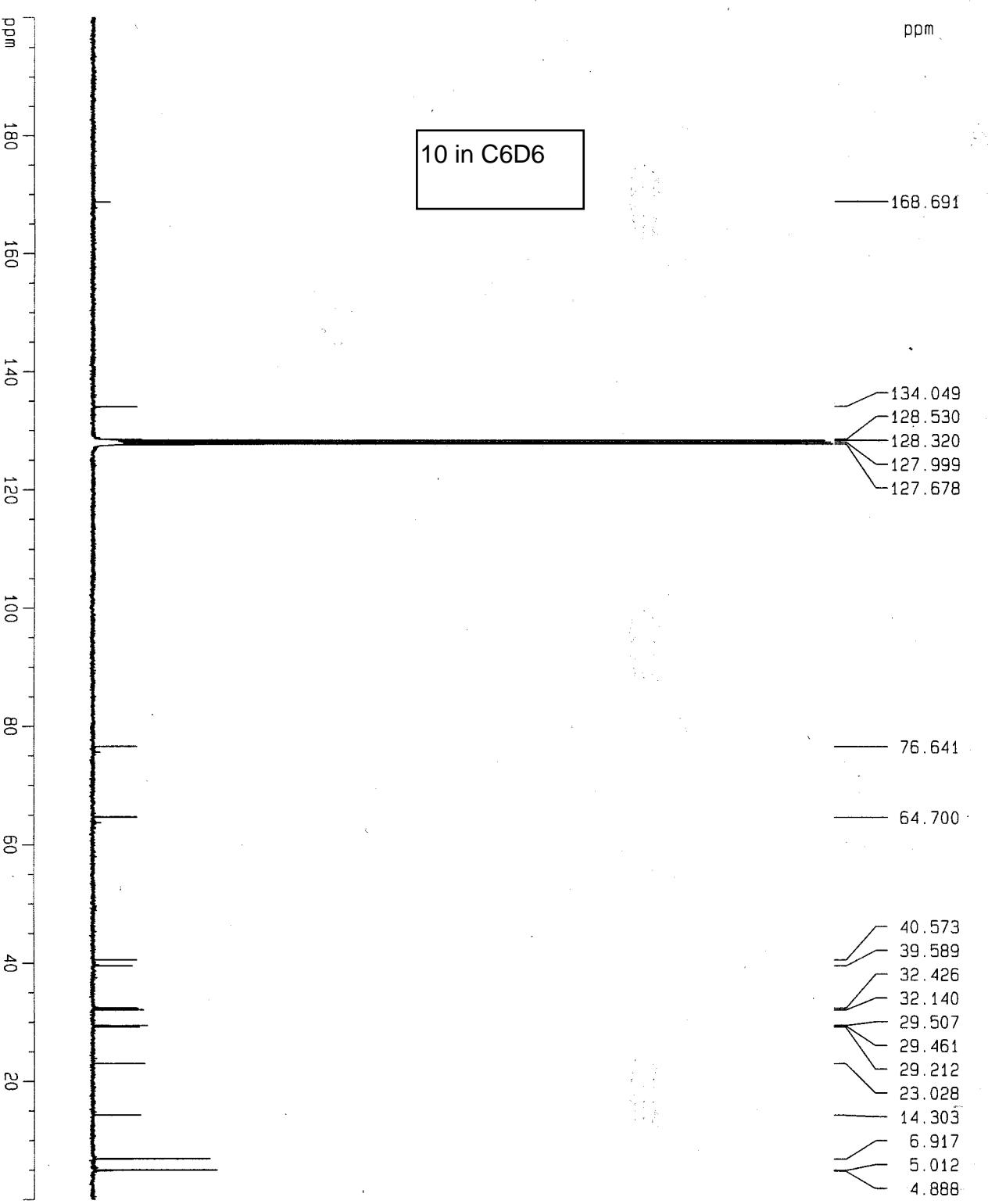
F2 - Processing parameters

SI 65536
SF 300.1299997 MHz
DW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

10 NMR plot parameters

CX 20.00 cm
CY 10.00 cm
F1P 7.613 ppm
F1 2284.78 Hz
F2P 0.075 ppm
F2 22.35 Hz
PPCM 0.37691 ppm/cm
HZCM 113.12118 Hz/cm

13C with proton decoupling



Current Data Parameters
 NAME pa2-168
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date 2004/01/15
 Time 18.13
 INSTRUM av300
 PROBHD 5 mm QNP 1H/1
 PULPROG zgpp30
 TO 32768
 SOLVENT CDCl3
 NS 446
 DS 0
 SWH 17985.611 Hz
 FIDRES 0.5481877 Hz
 AQ 0.9110004 sec
 RG 3649.1
 DW 27.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.0000000 sec
 d11 0.0300000 sec
 d12 0.00002000 sec

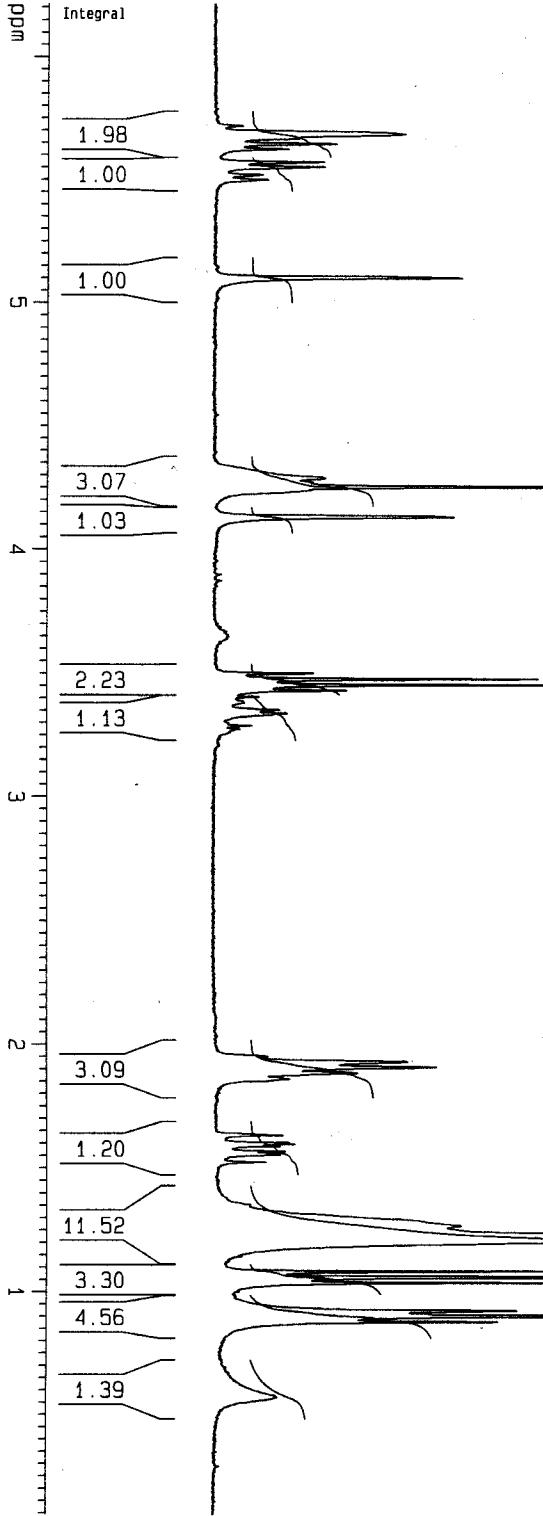
===== CHANNEL f1 =====
 NUC1 13C
 P1 5.00 usec
 PL1 -6.00 dB
 SF01 75.4752653 MHz
 ===== CHANNEL f2 =====
 CPBPRG2 Waltz16
 NUC2 1H
 PCP02 70.00 usec
 PL2 -3.00 dB
 PL12 13.48 dB
 PL13 15.63 dB
 SF02 300.1314960 MHz

F2 - Processing parameters

SI 65536
 SF 75.4677252 MHz
 WM EM
 SSB 0

LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 F1P 200.000 ppm
 F1 15093.55 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPM 10.00000 ppm/cm
 HZCM 754.6725 Hz/cm



4a in C6D6

Current Data Parameters
 NAME pa3-10
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date 2030527

Time 13:45

INSTRUM av300

PROBID 5 mm QNP 1H/1

PULPROG zg30

TD 30720

SOLVENT C6D6

NS 16

DS 0

SWH 0.165407 Hz

FORES 3.0228980 sec

AQ 362

RG 98.400 usec

DW 6.00 usec

DE 300.0 K

TE 1.0000000 sec
 ===== CHANNEL f1 =====

NUC1 1H
 P1 10.50 usec

PL1 -3.00 dB
 SF01 300.1319477 MHz

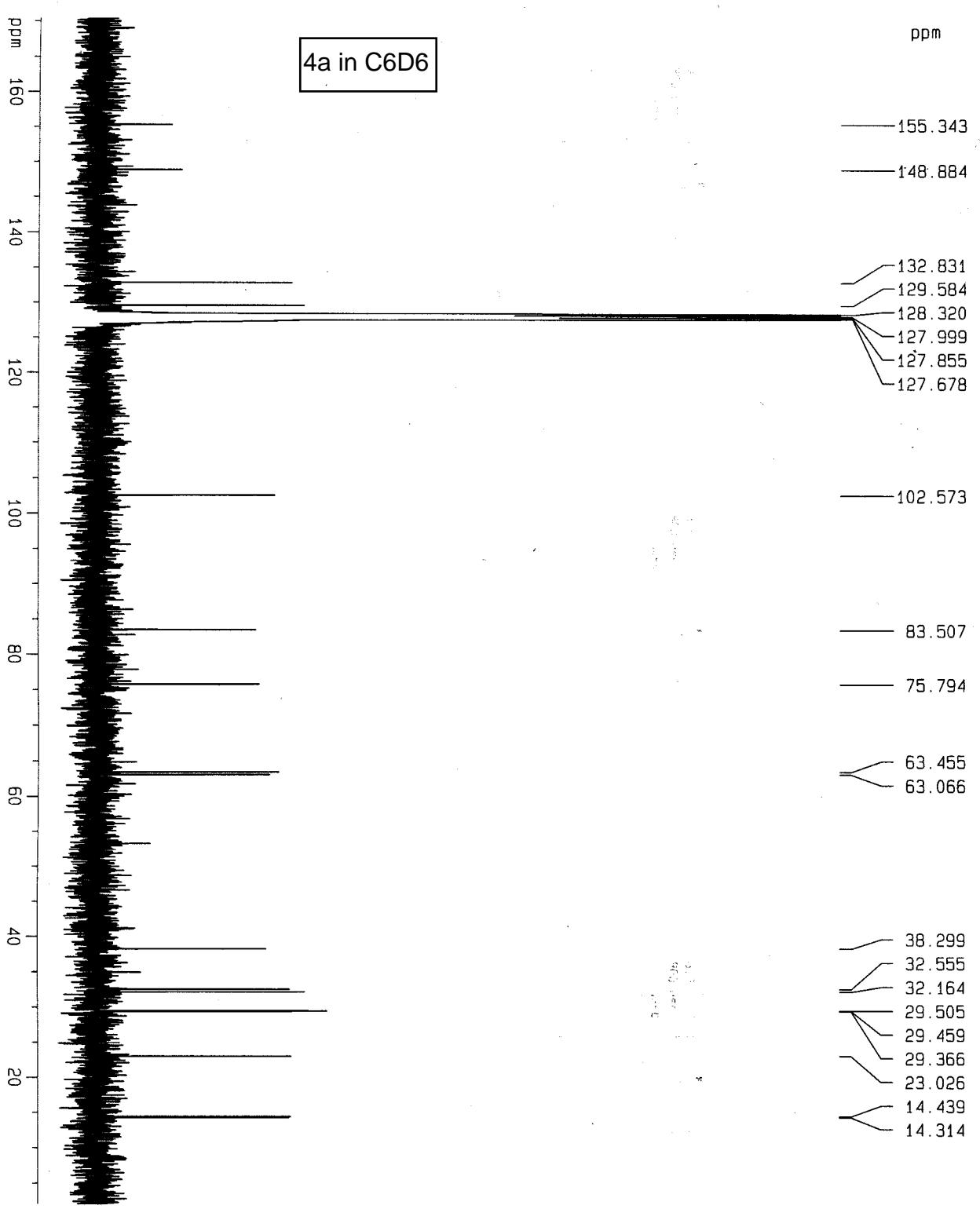
F2 - Processing parameters

SI 66536
 SF 300.1299997 MHz
 MDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

1D NMR plot parameters

CX 20.00 cm
 CY 10.00 cm
 F1P 6.214 ppm
 F1 1865.06 Hz
 F2P 0.092 ppm
 F2 27.66 Hz
 PPMCM 0.30610 ppm/cm
 HZCM 91.86992 Hz/cm

13C with proton decoupling



Current Data Parameters

NAME pa3-10
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date 20030627
Time 13:55
INSTRUM av300
PROBHD 5 mm QNP 1H1
PULPROG zgpp30
TD 32768
SOLVENT CDCl3
NS 802
DS 0
SWH 17955.611 Hz
FIDRES 0.548377 Hz
AQ 0.9110004 sec
RG 2298.8
DM 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.0000000 sec
d1 0.03000000 sec
d12 0.00002000 sec

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

NUC1 13C
P1 5.00 usec
PL1 -6.00 dB
SF01 75.4752653 MHz

===== CHANNEL f2 =====

CPPPG2 w1t16
NUC2 1H
PCP02 70.00 usec
PL2 -3.00 dB
PL12 13.48 dB
PL13 15.63 dB
SF02 300.1314860 MHz

F2 - Processing parameters

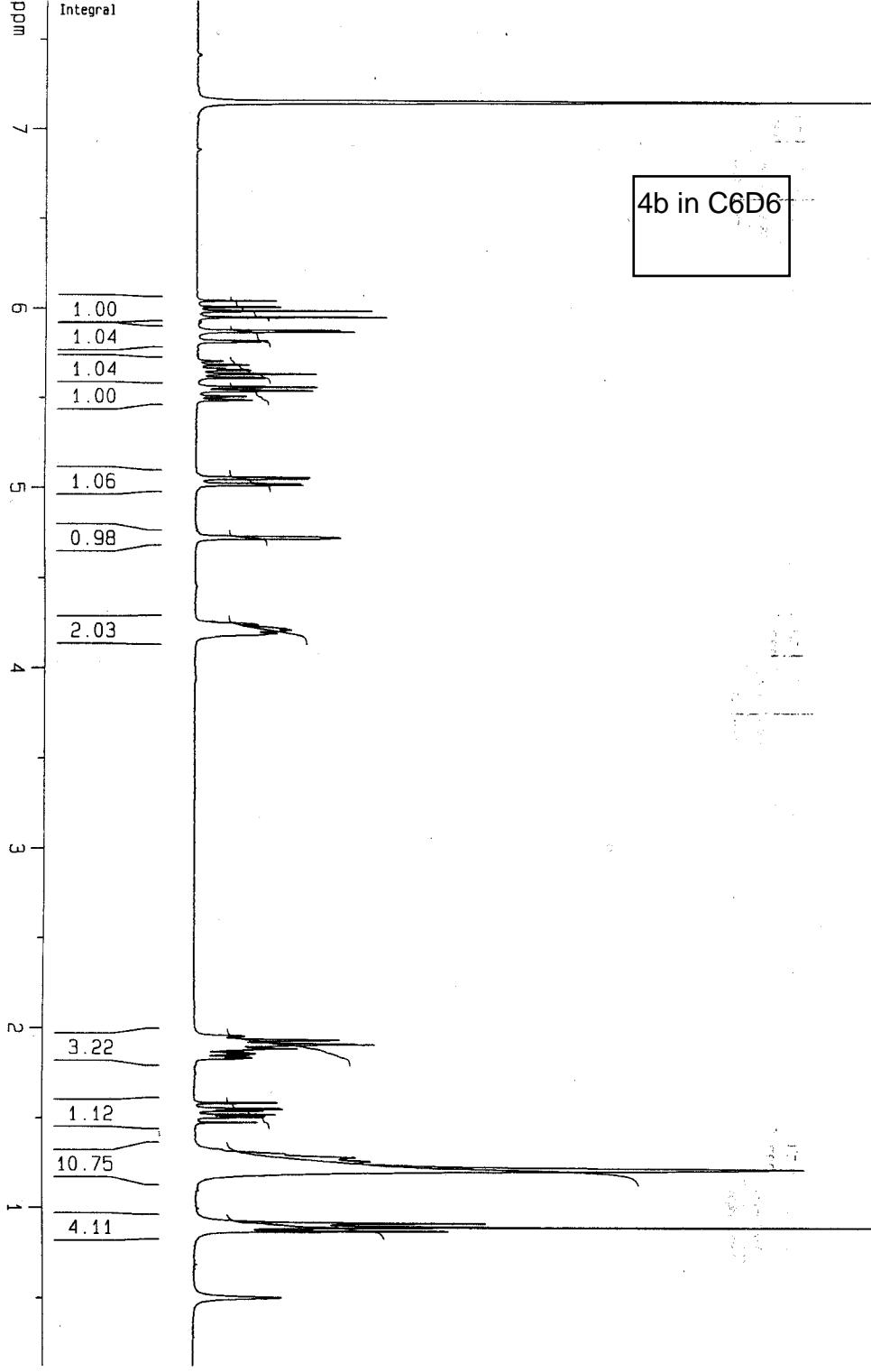
SI 65336
SF 75.4677560 MHz

MDW EM

SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
CY 200.00 cm
F1P 170.444 ppm
F1 12863.02 Hz
F2P 2.030 ppm
F2 153.19 Hz
PPCM 8.4271 ppm/cm
HZCM 635.49158 Hz/cm



Current Data Parameters
 NAME pa3-3
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date 20040115
 Time 18:42
 INSTRUM av300
 PROBHD 5 mm QNP 1H/1
 PULPROG zg30
 TD 30720
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 5081.301 Hz
 FIDRES 0.165407 Hz
 AQ 3.0228980 sec
 RG 287.4
 DW 98.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.0000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.50 usec
 PL1 -3.00 dB
 SF01 300.1319477 MHz
 SF 300.1299997 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

F2 - Processing parameters

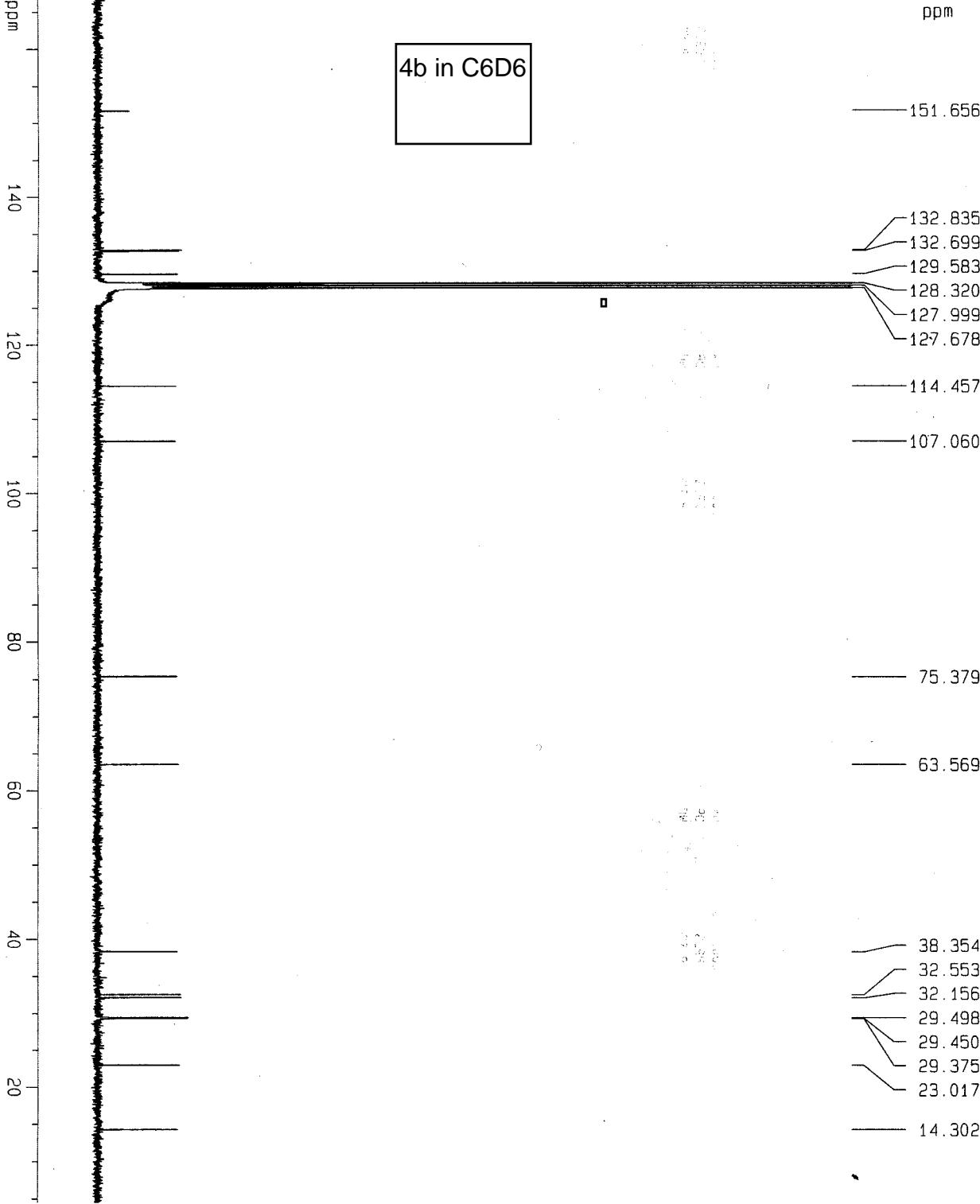
SI 65535
 SF 300.1299997 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 10.00 cm
 F1P 7.710 ppm
 F1 2314.07 Hz
 F2P 0.123 ppm
 F2 37.00 Hz
 PPMCM 0.37935 ppm/cm
 HzCM 113.88335 Hz/cm

13C with proton decoupling

4b in C6D6

ppm



Current Data Parameters
 NAME pa3-3
 EXPNO 2
 PROCNO 1

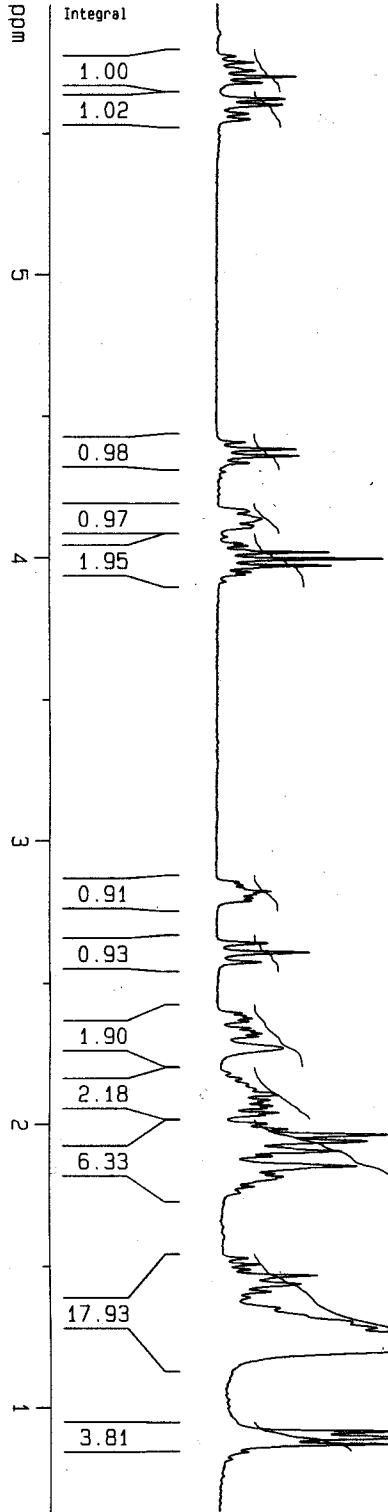
F2 - Acquisition Parameters
 Date 20040115
 Time 19.01
 INSTRUM av300
 PROBHD 5 mm QNP 1H/1
 PULPROG zgpp30
 TD 32768
 SOLVENT CDCl3
 NS 586
 DS 0
 SWH 17955.611 Hz
 FIDRES 0.548977 Hz
 AQ 0.911004 sec
 PG 2298.8
 DW 27.00 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.0000000 sec
 d11 0.0300000 sec
 d12 0.00002000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 5.00 usec
 PL1 -6.00 dB
 SF01 75.4752653 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCP02 70.00 usec
 PL2 -3.00 dB
 PL12 13.48 dB
 PL13 15.63 dB
 SF02 300.1314860 MHz

F2 - Processing parameters
 SI 65536
 SF 75.467755 MHz
 WDM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 25.00 cm
 F1P 166.614 ppm
 F1 12973.96 Hz
 F2P 4.527 ppm
 F2 341.67 Hz
 PPKCM 8.10432 ppm/cm
 HZCM 611.61450 Hz/cm



11 in C6D6

Current Data Parameters
 NAME p83-89
 EXPNO 1
 PROCNO 1

 F2 - Acquisition Parameters
 Date 2003/02/22
 Time 22.54
 INSTRUM av300
 PROBHD 5 mm QNP 1H/1
 PULPROG zg30
 TD 30720
 SOLVENT ~~C6D6~~
 NS 16
 DS 0
 SWH 5081.301 Hz
 FIDRES 0.165407 Hz
 AQ 3.028980 sec
 RG 287.4
 DW 98.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.0000000 sec

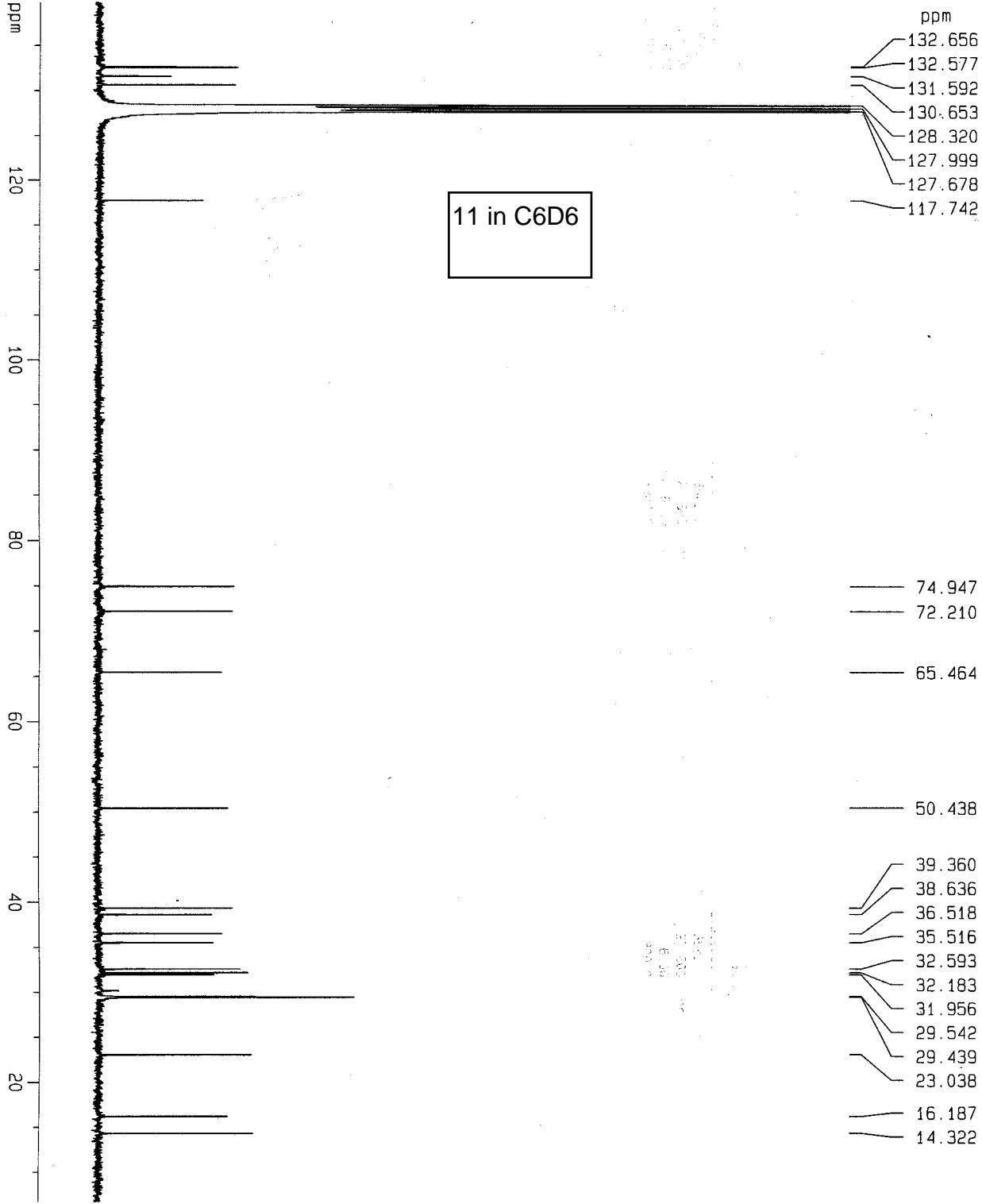
 ===== CHANNEL f1 =====

NUC1 1H
 P1 10.50 usec
 PL1 -3.00 dB
 SF01 300.1319477 MHz

 F2 - Processing parameters
 SI 65536
 SF 300.1299996 MHz
 MDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 10.00 cm
 F1P 5.958 ppm
 F1 1788.24 Hz
 F2P 0.630 ppm
 F2 189.23 Hz
 F2PPCM 0.28639 ppm/cm
 HZCM 79.95053 Hz/cm

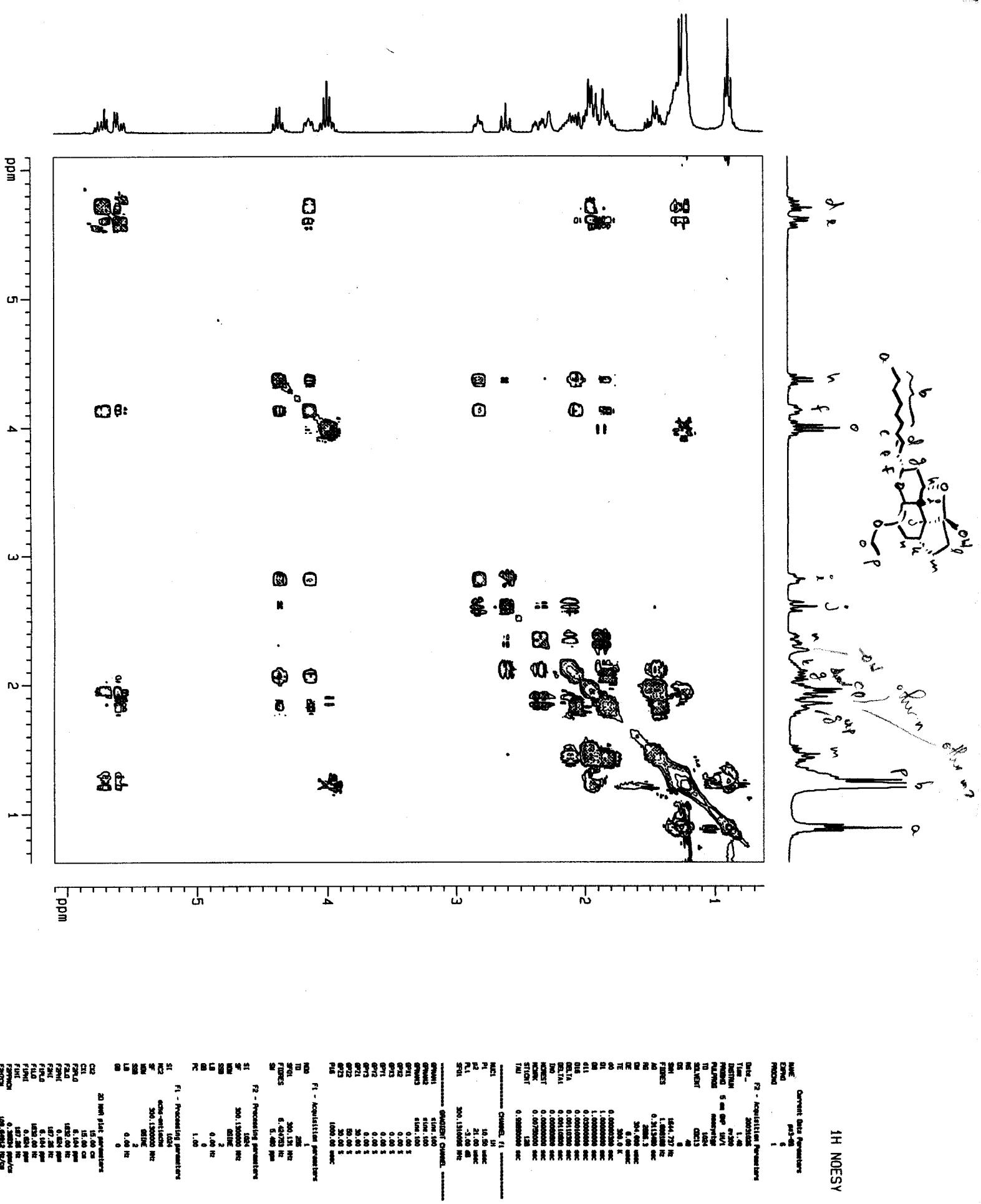
13C with proton decoupling

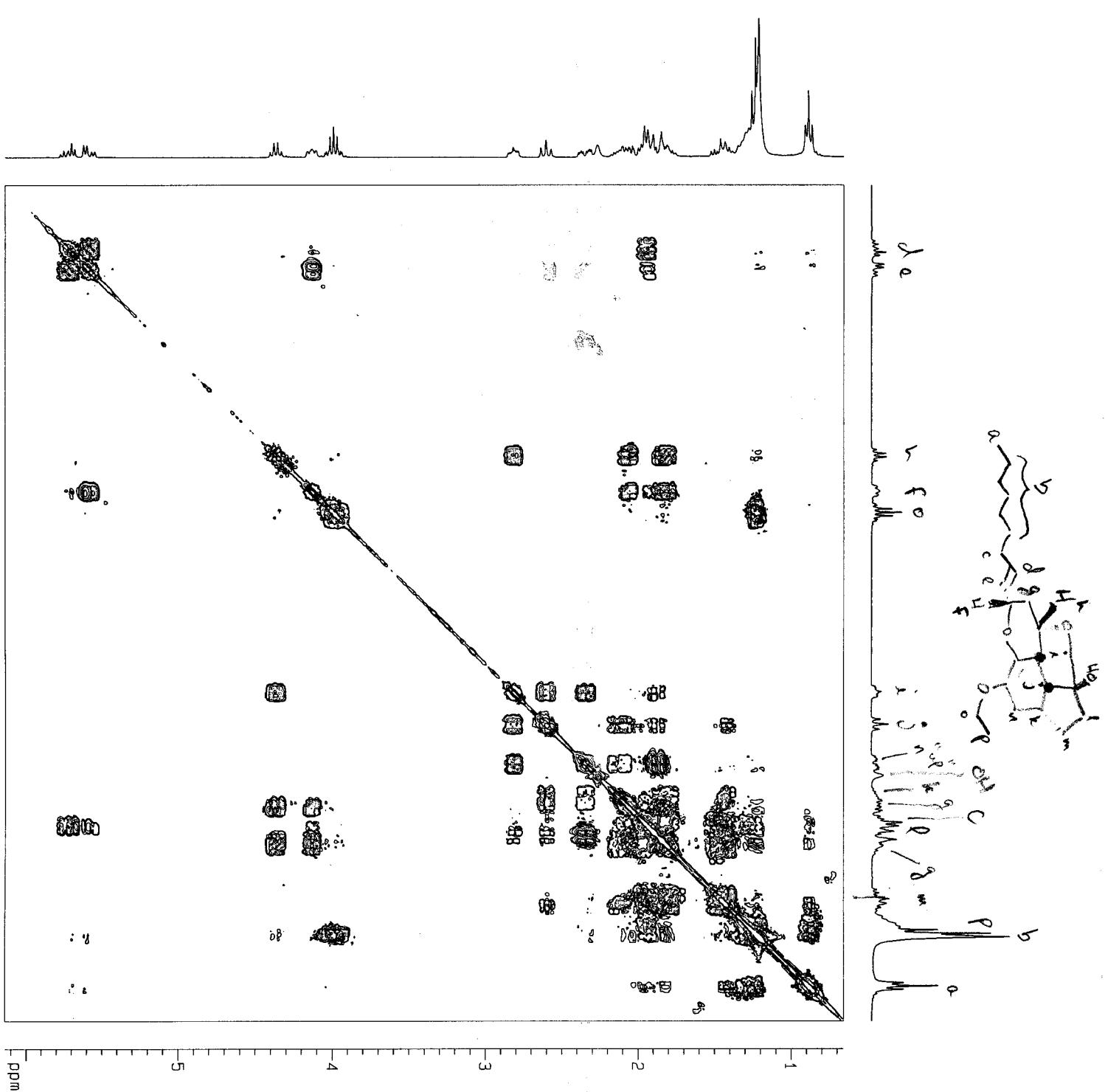


Current	Data	Parameters
NAME	pa3-89	
EXPNO	1	2
PROCNO	1	
F2 - Acquisition Parameters		
DATE	20031023	
TIME	2.54	
INSTRUM	av300	
PROBHD	5 mm GMP 1H/1	
PULPROG	zgpg30	
TD	32768	
SOLVENT	CDCl3	
NS	7168	
DS	0	
SWH	17985.611 Hz	
FOIDRES	0.548877 Hz	
AQ	0.9110004 sec	
RG	3649.1	
DW	27.800 usec	
DE	6.00 usec	
TE	300.0 K	
D1	1.0000000 sec	
d11	0.0300000 sec	
d12	0.00002000 sec	
===== CHANNEL f1 =====		
NUC1	13C	
P1	5.00 usec	
PL1	-6.00 dB	
SF01	75.4752653 MHz	
===== CHANNEL f2 =====		
CPDPRG2	Waltz16	
NUC2	1H	
PCPD2	70.00 usec	
PL2	-3.00 dB	
PL12	13.48 dB	
PL13	15.63 dB	
SF02	300.1314860 MHz	
F2 - Processing parameters		
SI	65536	
SF	75.4677260 MHz	
WDW	EM	
SSB	0	
LB	1.00 Hz	
GB	0	
PC	1.40	

1D NMR plot parameters

CX	20.00	cm
CY	100.00	cm
F1P	139.732	ppm
F1	105.45	-27 Hz
F2P	6.739	ppm
F2	508.54	Hz
PPCM	6.6498	ppm/cm
HCM	501.83624	Hz/cm





Current Data Parameters

NAME: p03-89
EXPO: 4
PROCNO: 1

Date: 2003/02/23
TIME: 4:34
INSTRUM: a1300
PROBOD: 5 mm QNP 1H/1
PULPROG: cosy-seqpr
TD: 1024
SOLVENT: CDCl3
NS: 10
DS: 0
SWH: 164.737 Hz
FIDRES: 1.606188 Hz
AQ: 0.313460 sec
RG: 1824.6
DM: 304.000 usc
DE: 6.00 usc
TE: 300.0 K
d1: 0.0000000 sec
d13: 1.0000000 sec
d15: 0.0001000 sec
TIN: 0.0006000 sec
MCNEST: 0.0000000 sec
MCINCH: 1.0000000 sec

NUC1: 1H
GR1N1: 1H
GR1N2: 1H
S1: sine 100
P0: 5.28 usc
P1: 10.50 usc
PL1: -3.00 dB
SF01: 300.131203 MHz

CHANNEL 1
NUC1: 1H
GR1N1: 1H
GR1N2: 1H
S1: sine 100
P0: 5.28 usc
P1: 10.50 usc
PL1: -3.00 dB
SF01: 300.131203 MHz

GRADIENT CHANNEL

NUC1: 1H
GR1N1: 1H
GR1N2: 1H
S1: sine 100
P0: 5.28 usc
P1: 10.50 usc
PL1: -3.00 dB
SF01: 300.131203 MHz

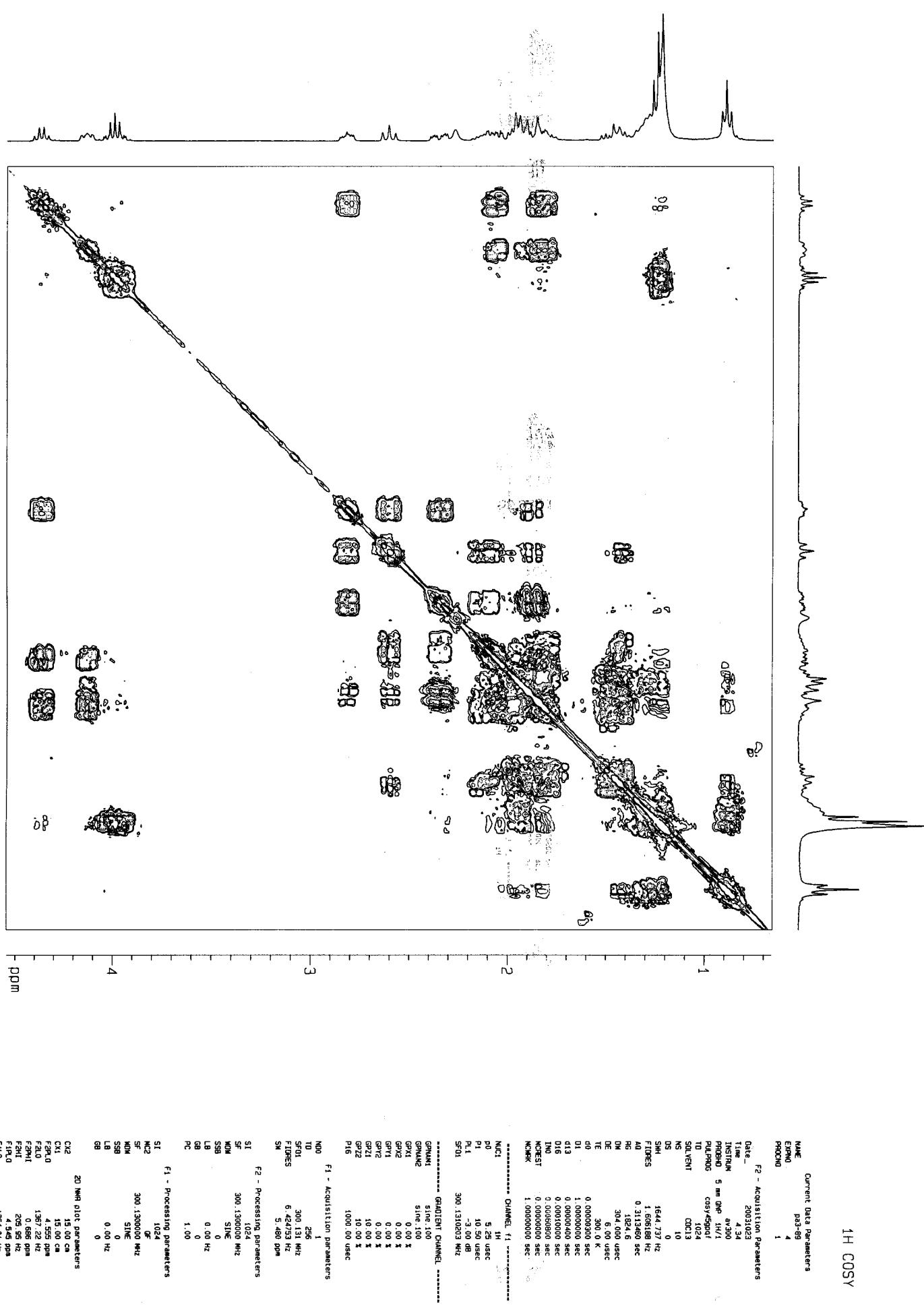
NUC1: 1H
GR1N1: 1H
GR1N2: 1H
S1: sine 100
P0: 5.28 usc
P1: 10.50 usc
PL1: -3.00 dB
SF01: 300.131203 MHz

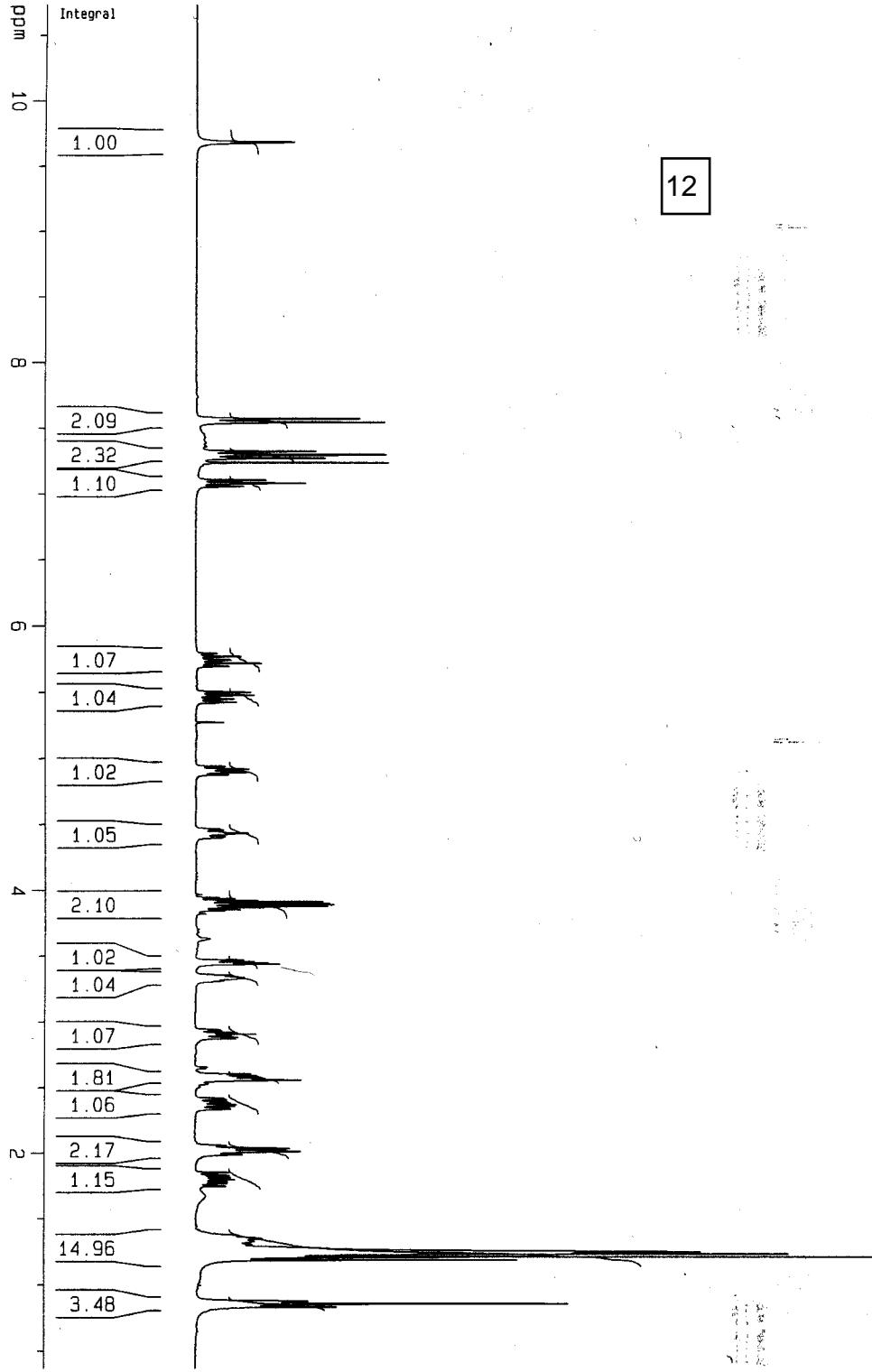
F1 - Acquisition parameters
N0: 1
TD: 256
SF01: 300.131 MHz
FIDRES: 6.42753 Hz
SW: 5.480 ppm
S1: 1024
SF: 300.130000 MHz
HDM: SINE
SSB: 0
LB: 0.00 Hz
GB: 1.00
PC: 1.00

F2 - Processing parameters
S1: 1024
N0: 1
SF: 300.130000 MHz
HDM: SINE
SSB: 0
LB: 0.00 Hz
GB: 0

2D NMR plot parameters
CX1: 15.00 cm
CX2: 15.00 cm
F2P1: 0.3654 ppm/cm
F2P2: 0.3654 ppm/cm
F2D1: 0.3654 ppm/cm
F2D2: 0.3654 ppm/cm
F2H1: 0.3654 ppm/cm
F2H2: 0.3654 ppm/cm
F1P1: 0.3654 ppm/cm
F1P2: 0.3654 ppm/cm
F1D1: 0.3654 ppm/cm
F1D2: 0.3654 ppm/cm
F1H1: 0.3654 ppm/cm
F1H2: 0.3654 ppm/cm
109.6592 Hz/cm
109.6594 Hz/cm
109.6592 Hz/cm
109.6594 Hz/cm

1H COSY





Current Data Parameters

NAME	pa3-74c
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date	2003/06
Time	17.00
INSTRUM	8v300
PROBHD	5 mm QNP 1H/1
PULPROG	zg30
TD	30720
SOLVENT	CCl3
NS	16
DS	0
SWH	5081.301 Hz
FIDRES	0.165407 Hz
AQ	3.0228980 sec
RG	228.1
DW	98.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.0000000 sec

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

NUC1	1H
P1	10.50 usec
PL1	-3.00 dB
SF01	300.131947 MHz

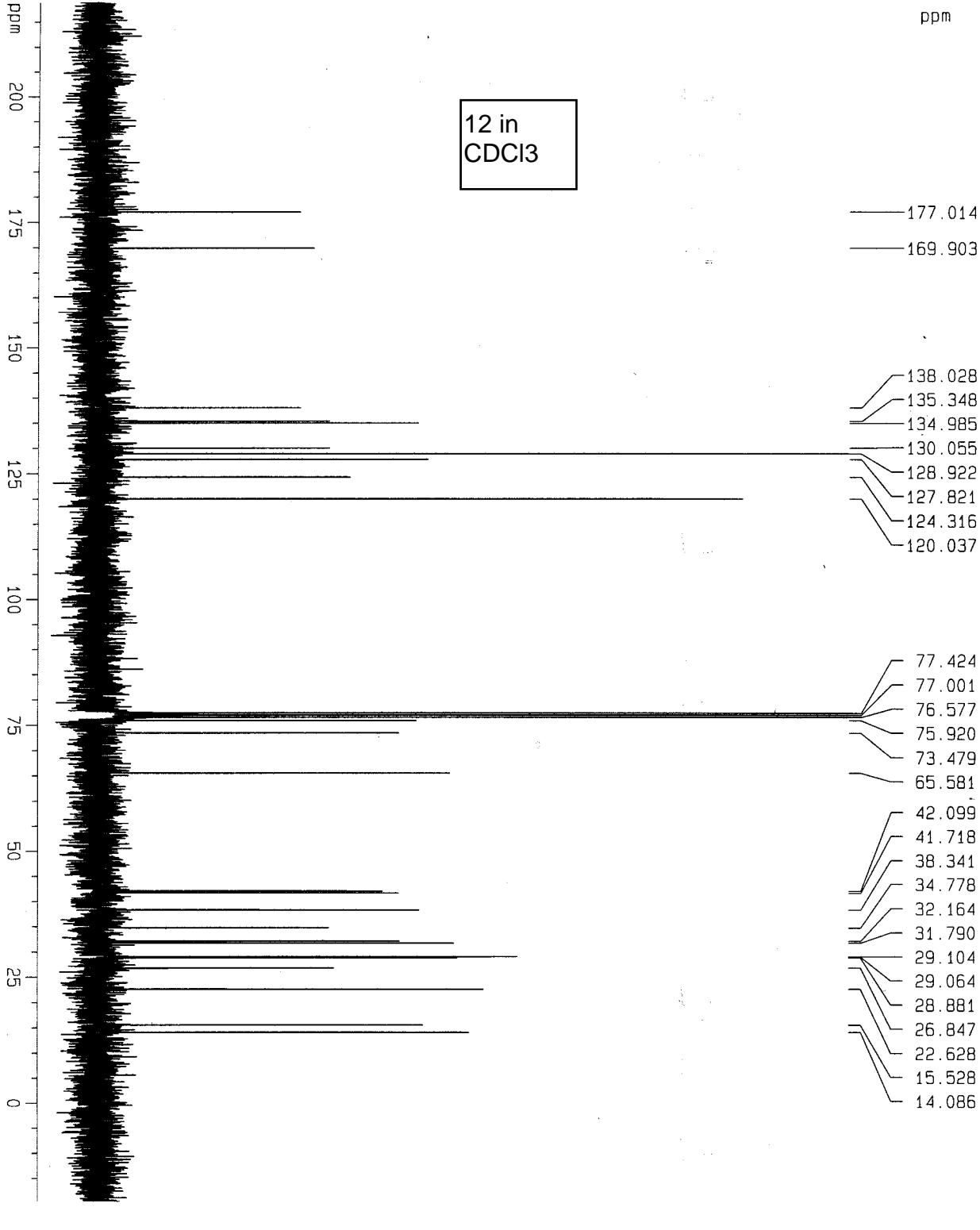
F2 - Processing parameters

SI	66536
SF	300.1300000 MHz
MDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
CY	10.00 cm
F1P	10.728 ppm
F1	3219.94 Hz
F2P	0.367 ppm
F2	110.03 Hz
PPMCM	0.51809 ppm/cm
HZCM	155.49545 Hz/cm

13C with proton decoupling



Current Data Parameters

NAME	pa3-74c
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date	20031006
Time	17.09
INSTRUM	av390
PROBHD	5 mm QNP 1H1
PULPROG	zpg30
TD	32768
SOLVENT	CDCl3
NS	371
DS	0
SWH	17985.611 Hz
FIDRES	0.9548877 Hz
AQ	0.910004 sec
RG	8192
DW	27.800 usec
DE	6.00 usec
TE	300.0 K
D1	1.0000000 sec
d11	0.0300000 sec
d12	0.00002000 sec

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

NUC1	13C
P1	5.00 usec
PL1	-6.00 dB
SF01	75.4752653 MHz

===== CHANNEL f2 =====

CPDPFG2	Walzt16
NUC2	1H
PCPPI2	70.00 usec
PL2	-3.00 dB
PL12	13.48 dB
PL13	15.63 dB
SF02	300.1314860 MHz

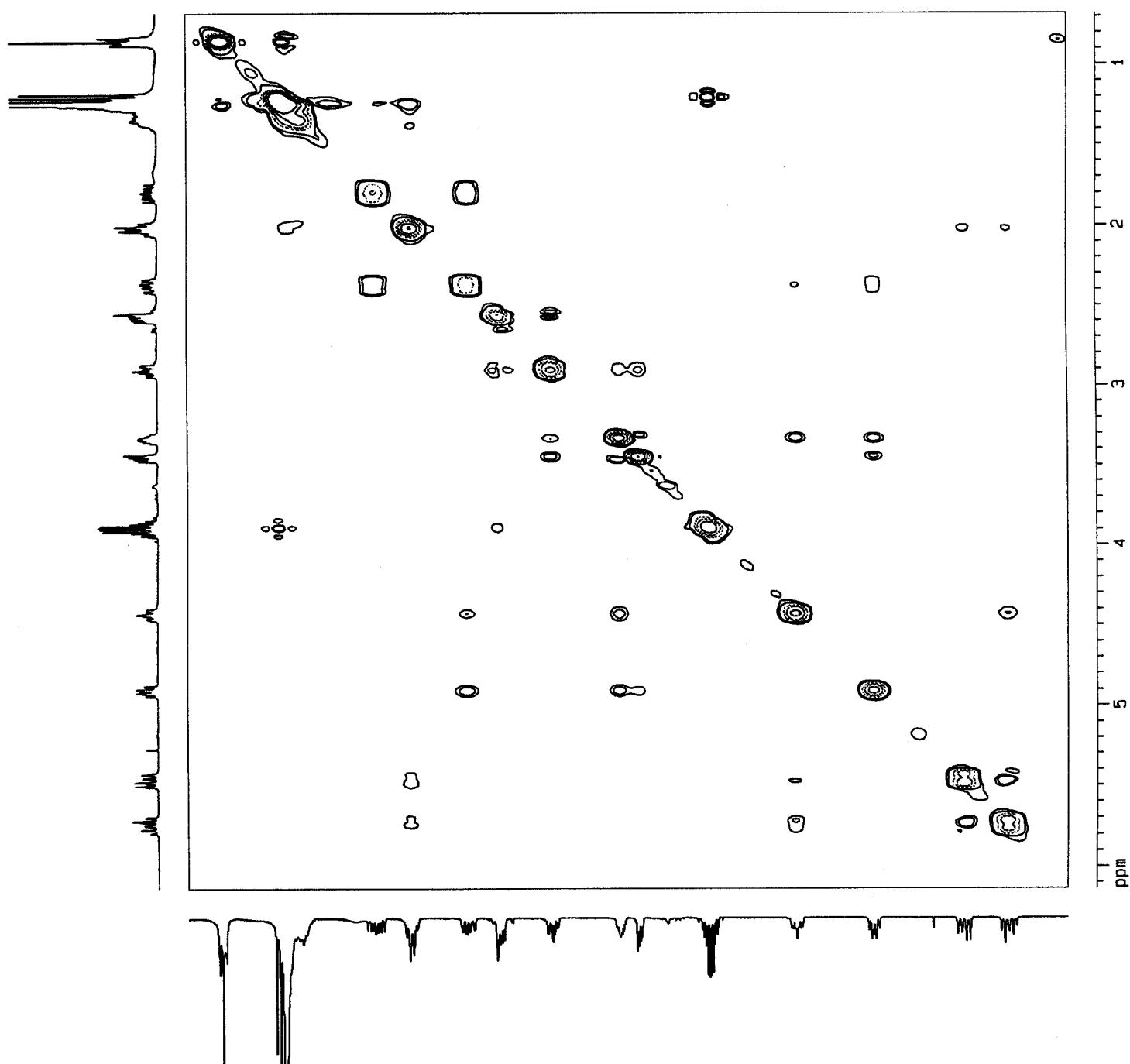
F2 - Processing parameters

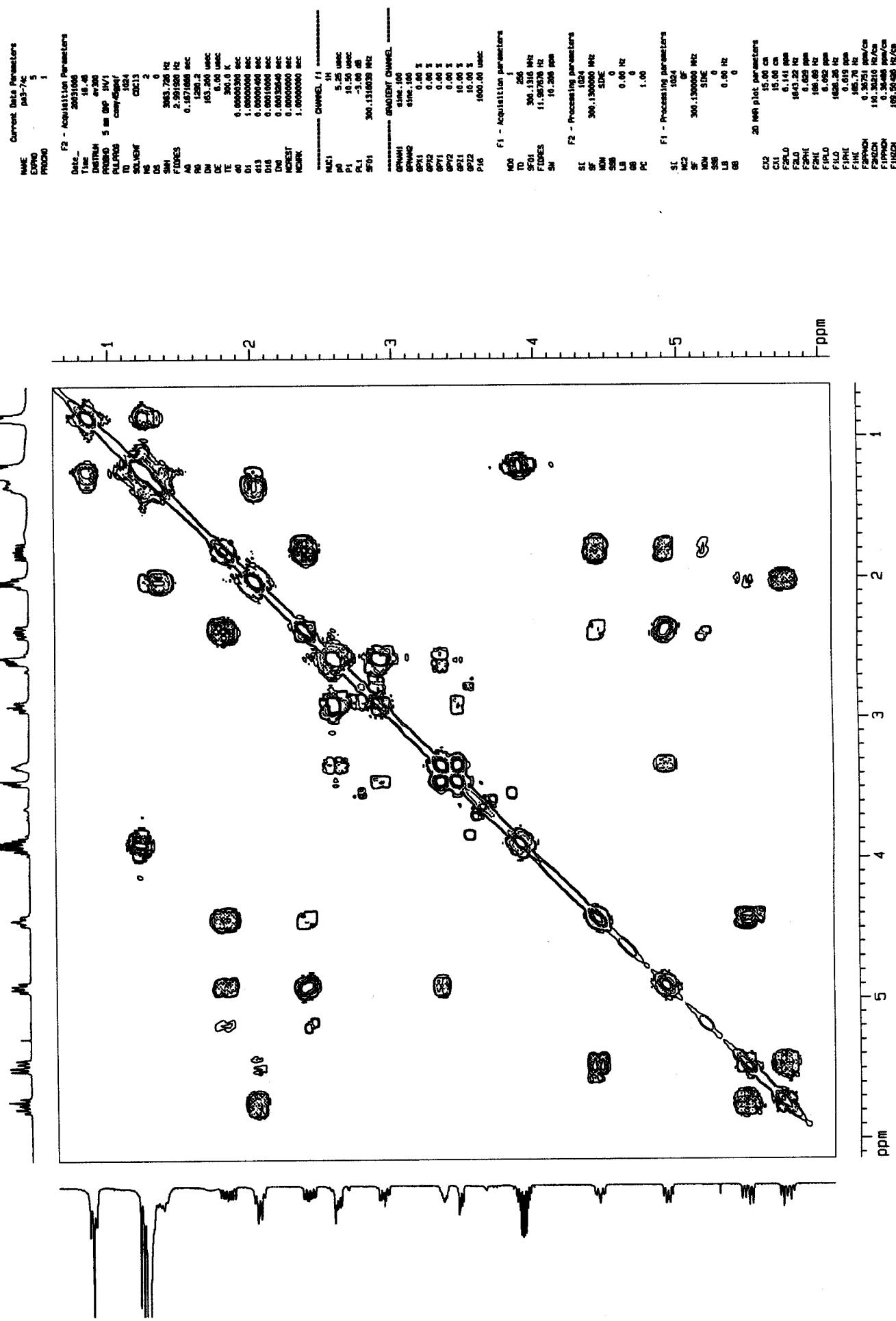
ST	65536
SF	75.4677492 MHz
WDW	EM
SSB	0

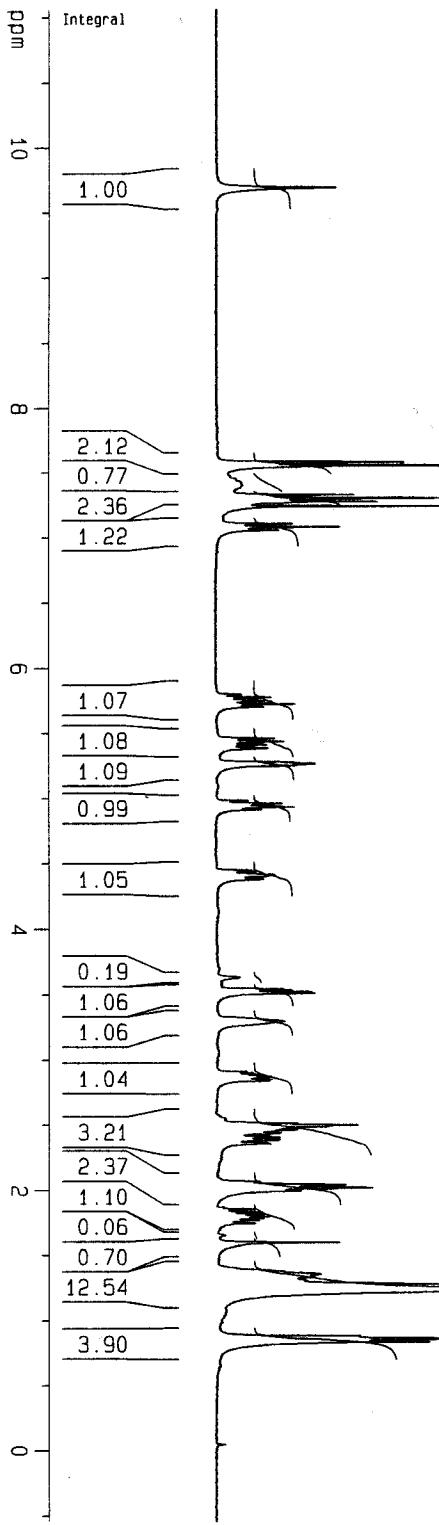
LB	1.00 Hz
GB	0
PC	1.40

10 NMR plot parameters

CX	20.00 cm
CY	12.50 cm
F1P	218.754 ppm
F1	16508.91 Hz
F2P	-19.567 ppm
F2	-1476.70 Hz
PPMCM	11.91609 ppm/cm
HZCM	899.28052 Hz/cm







13

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

NUC1 1H
 P1 10.50 usec
 PL1 -3.00 dB
 SF01 300.1318477 MHz

F2 - Processing parameters

SI 65536
 SF 300.1300001 MHz
 MDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 10.00 cm
 F1P 11.052 ppm
 F1 3320.00 Hz
 F2P -0.545 ppm
 F2 -163.72 Hz
 PPMCM 0.558037 ppm/cm
 HZCM 174.18587 Hz/cm

Current Data Parameters

NAME pa2-164
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date 20030429

Time 14.12

INSTRUM

PROBHD 5 mm QNP 1H/1

PULPROG

zg30

TD 30720

SOLVENT

CCl3

NS 16

DS 0

SWH 5081.301 Hz

FIDRES 0.165407 Hz

AQ 3.0228980 sec

RG 256

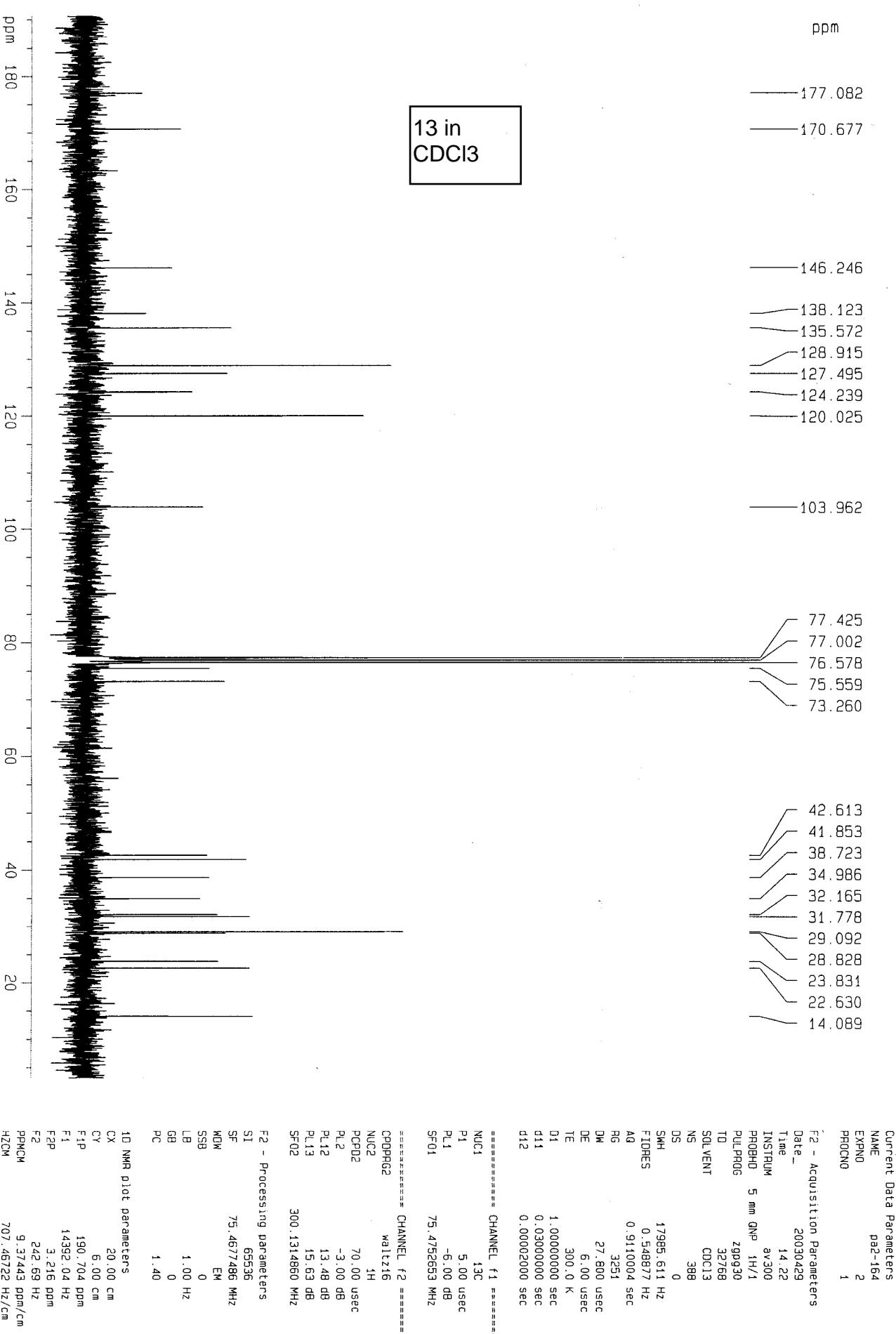
DM 98.400 usec

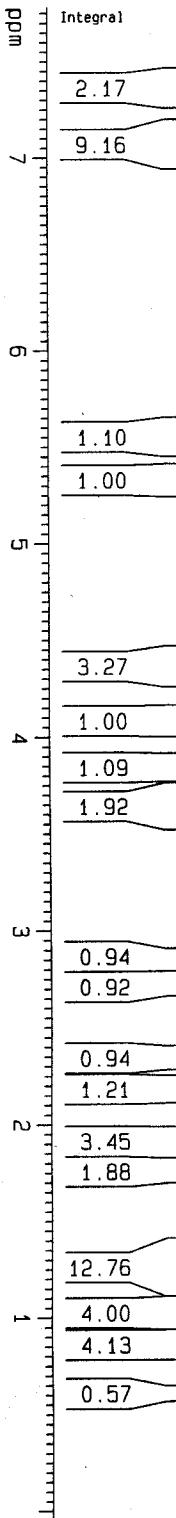
DE 6.00 usec

TE 300.0 K

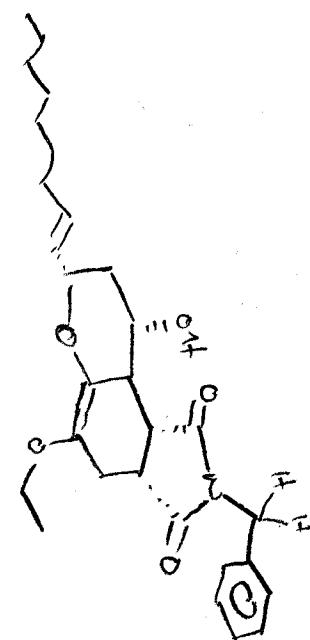
D1 1.0000000 sec

13C with proton decoupling





14 in C6D6



Current Data Parameters

NAME r1-110
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date 2003/02/22
Time 12:56
INSTRUM av300
PROBOD 5 mm QNP 1H/1
PULPROG zg30
TD 30720
SOLVENT CDCl3
NS 16
DS 0
SWH 5081.301 Hz
SF 0.165407 Hz
FIRES 3.0228980 sec
AQ 228.1
RG 98.400 usec
DW 6.00 usec
DE 300.0 K
TE 1.0000000 sec
D1

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

NUC1 1H
SI 65536
SF 300.1300000 MHz
MDW 0
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

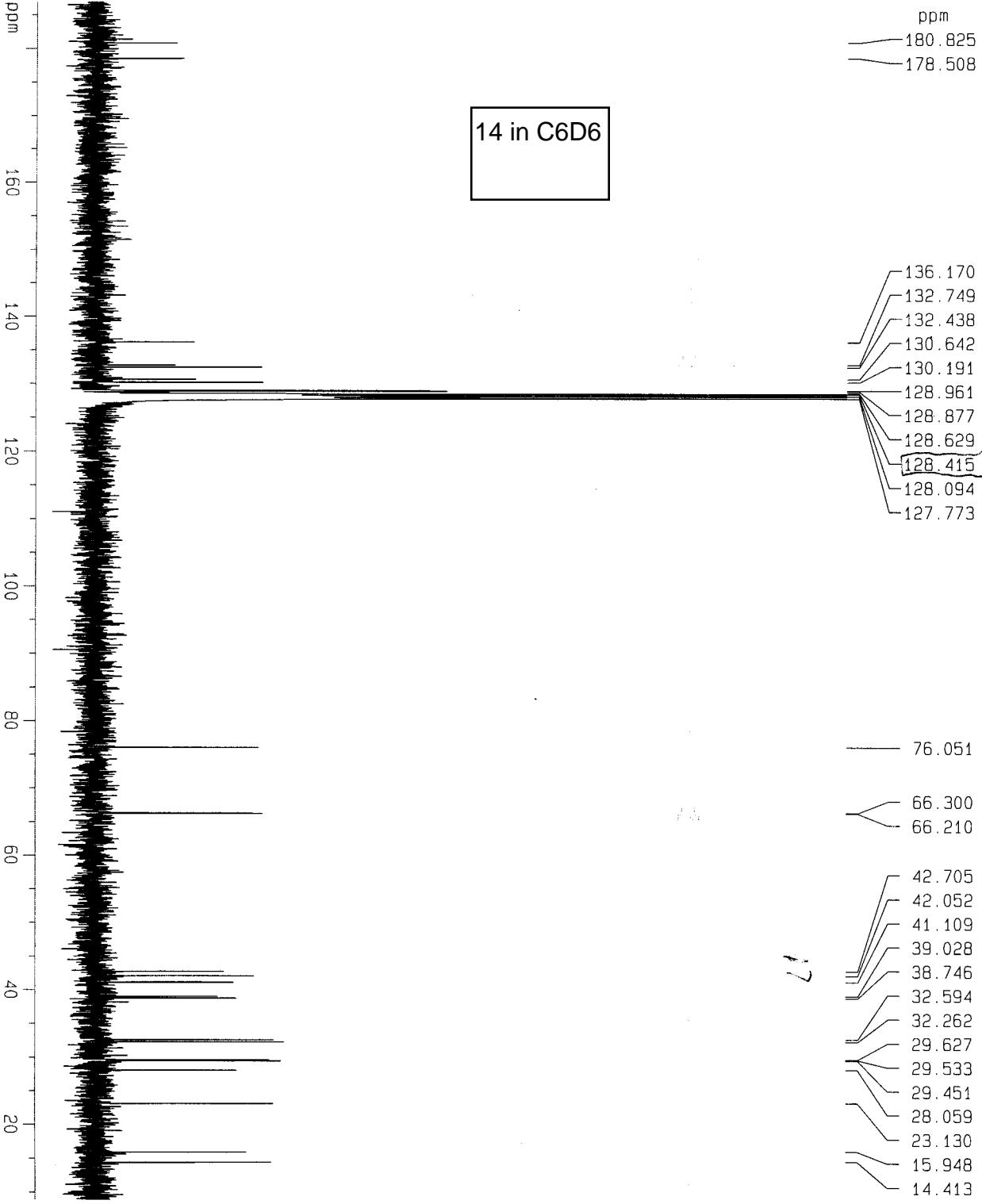
F2 - Processing parameters

ST 65536
SF 300.1300000 MHz
DW 0
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
CY 10.00 cm
F1P 7.780 ppm
F1 2335.03 Hz
F2P -0.034 ppm
F2 -10.19 Hz
PPMCM 0 ppm/cm
HZCM 117.26078 Hz/cm

13C with proton decoupling



Current	Data	Parameters
NAME	r1-110	
EXPNO	2	
PROCNO	1	

F2 - Acquisition Parameters

DATE	20031022	
TIME	13:03	
INSTRUM	av300	
PROBHD	5 mm QNP 1H/1	
PULPROG	zgpg30	
TD	32768	
SOLVENT	CDCl ₃	
NS	27	
DS	0	
SWH	17985.611 Hz	
FIDRES	0.508877 Hz	
AQ	0.9110004 sec	
RG	3649.1	
DH	27.800 usec	
DE	6.00 usec	
TE	300.0 K	
D1	1.00000000 sec	
d11	0.03000000 sec	
d12	0.00002000 sec	

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

NUC1	13C
P1	5.00 usec
PL1	-6.00 dB
SF01	75.4752653 MHz

===== CHANNEL f2 =====

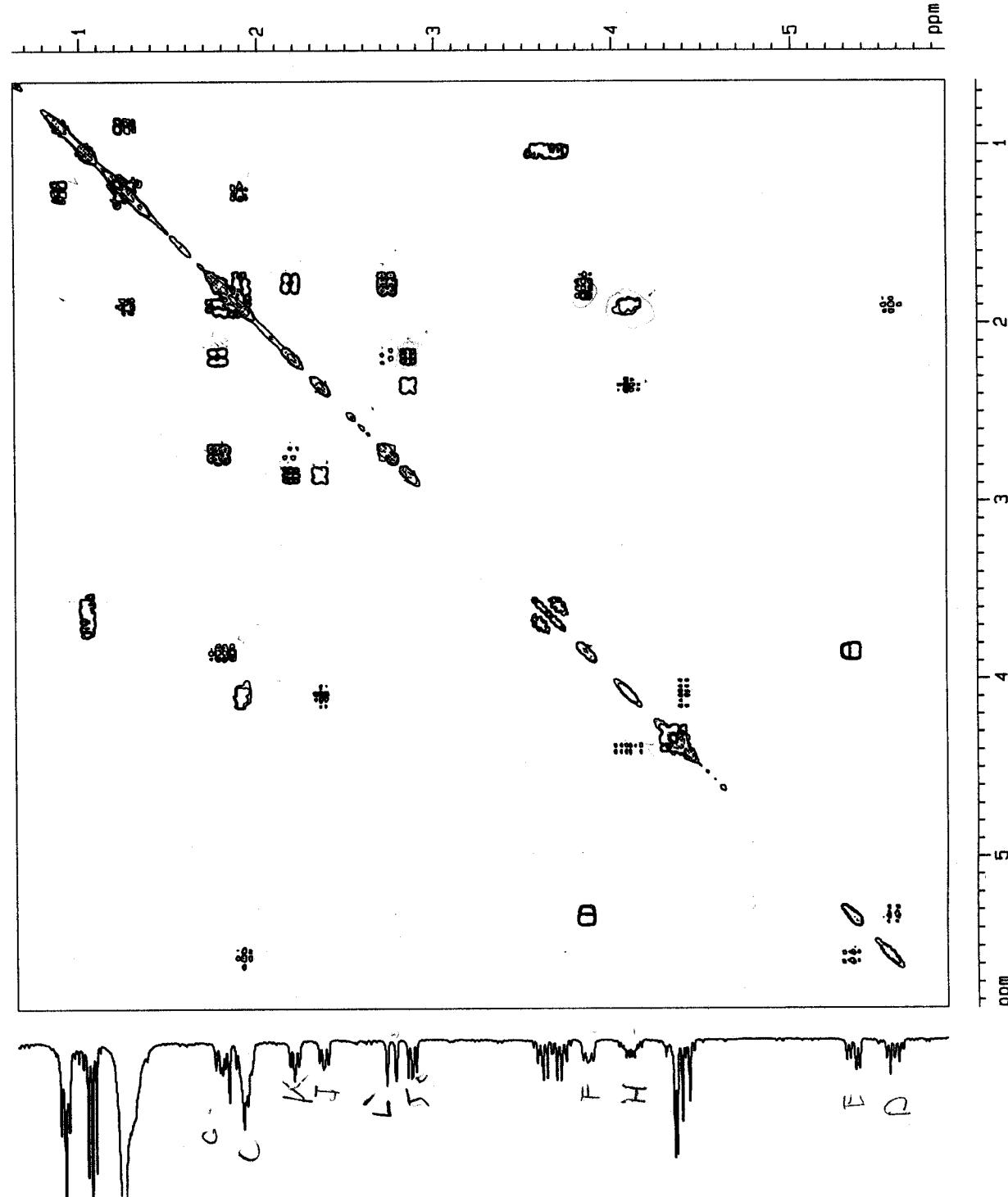
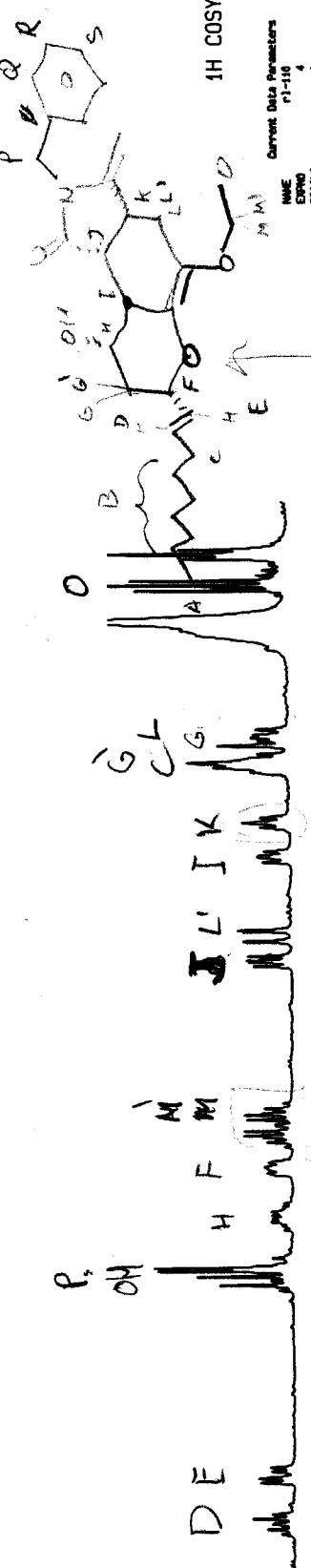
CPDPG2	wait2f6
NUC2	1H
PCP02	70.00 usec
PL2	-3.00 dB
PL12	13.48 dB
PL13	15.63 dB
SFC2	300.1314860 MHz

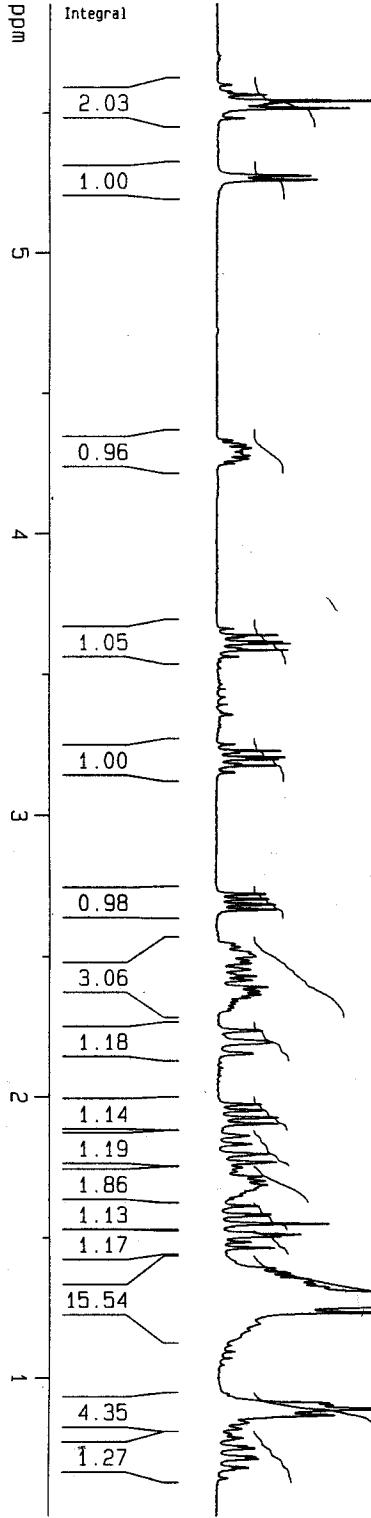
F2 - Processing parameters

SI	6536
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
CY	100.00 cm
F1P	186.823 ppm
F1	14099.08 Hz
F2P	8.831 ppm
F2	666.47 Hz
PPMCM	8.8957 ppm/cm
HZCM	671.63055 Hz/cm





15 in C6D6

Current Data Parameters
 NAME paclainen
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date 20040121
 Time 11.44

INSTRUM av300

PROBID 5 mm QNP 1H/1

PULPROG zg30

TD 30720

SOLVENT CDCl3

NS 128

DS 0

SWH 5081.301 Hz

FIDRES 0.165407 Hz

AQ 3.0228980 sec

RG 645.1

DW 98.400 usec

DE 6.00 usec

TE 300.0 K

D1 1.0000000 sec

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

NUC1 1H

P1 10.50 usec

PL1 -3.00 dB

SF 300.1299998 MHz

MDW EM

SSB 0

LB 0.10 Hz

GB 0

PC 1.00

F2 - Processing parameters

SI 66536

SF 300.1299998 MHz

MDW EM

SSB 0

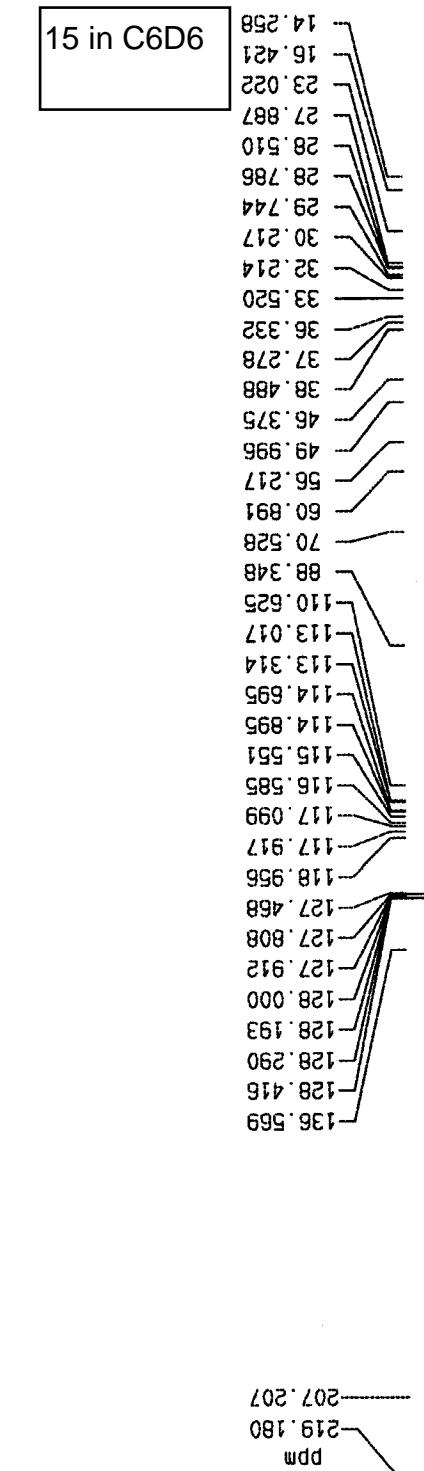
LB 0.10 Hz

GB 0

PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 10.00 cm
 F1P 5.886 ppm
 F1 1766.69 Hz
 F2P 0.511 ppm
 F2 153.46 Hz
 F2PPCM 0.28675 ppm/cm
 HZCM 80.66121 Hz/cm

13C with proton decoupling



Current Data Parameters
NAME pat_c1a
EXPT 5
PROCNO 1

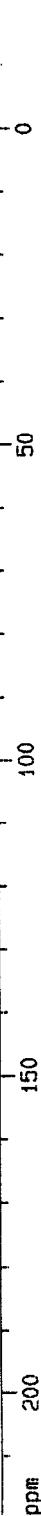
F2 - Acquisition Parameters
Date_ 20040202
Time 8.21
INSTRUM AV500WB
PROBHD 5 mm BBI 1H-88
PULPROG zg3030
TD 65536
CD 13
SOLVENT NS
NS 43804
DS 0
SWH 30030.029 Hz
FIDRES 0.459222 Hz
AQ 1.091224 sec
RG 3251
DW 16.650 usec
DE 6.00 usec
TE 300.0 K
D1 1.0000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 14.50 usec
PL1 -3.00 dB
SF01 125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL2 3.00 dB
PL12 20.54 dB
PL13 23.56 dB
SF02 500.1320005 MHz

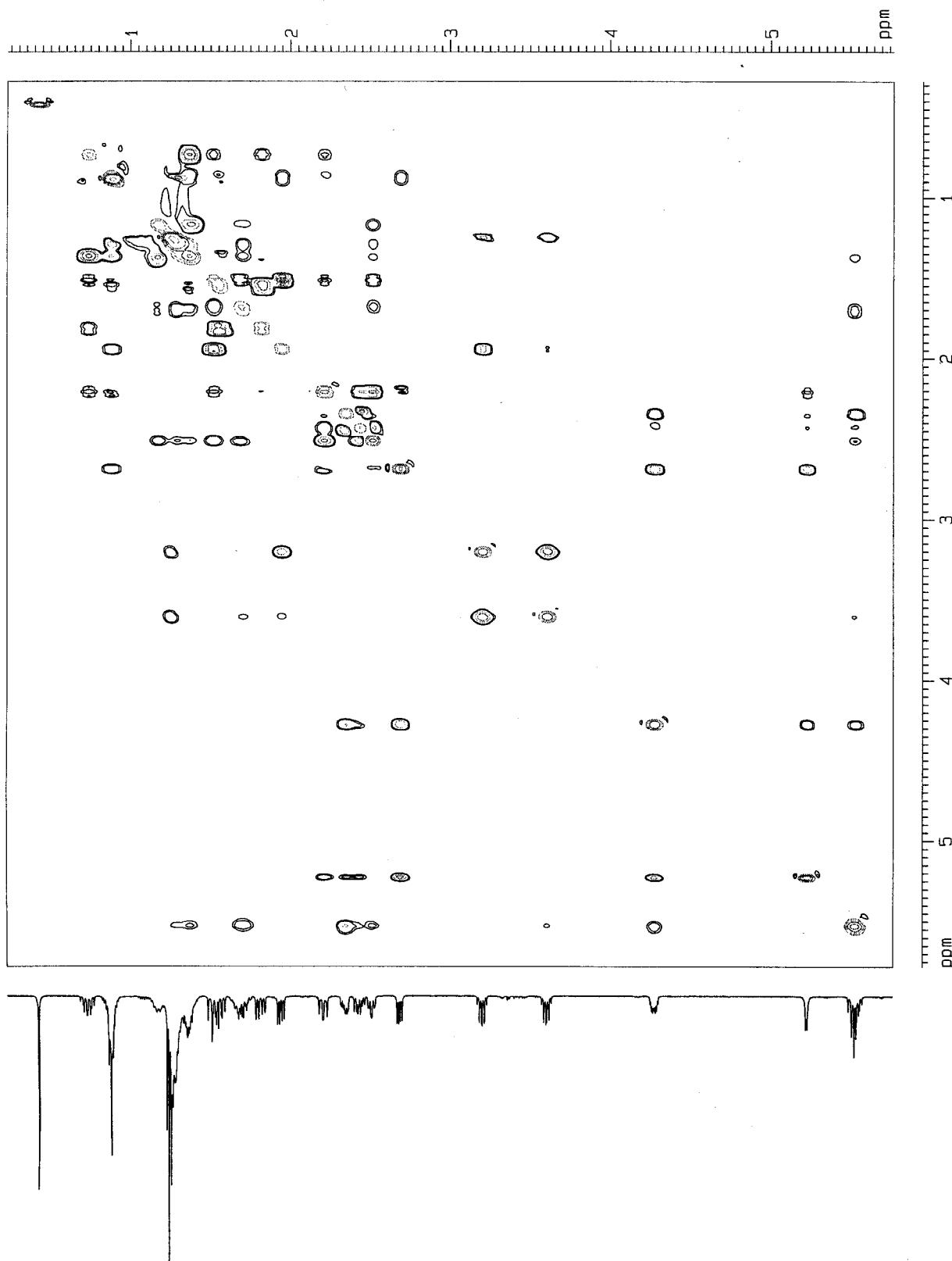
F2 - Processing parameters
SI 32768
SF 125.7577635 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 40.00 cm
F1P 219.264 ppm
F1 27574.16 Hz
F2P -19.528 ppm
F2 -245.86 Hz
PHON HZCM 1501.50098 Hz/cm

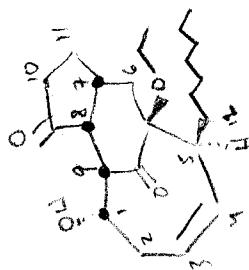


NOESY

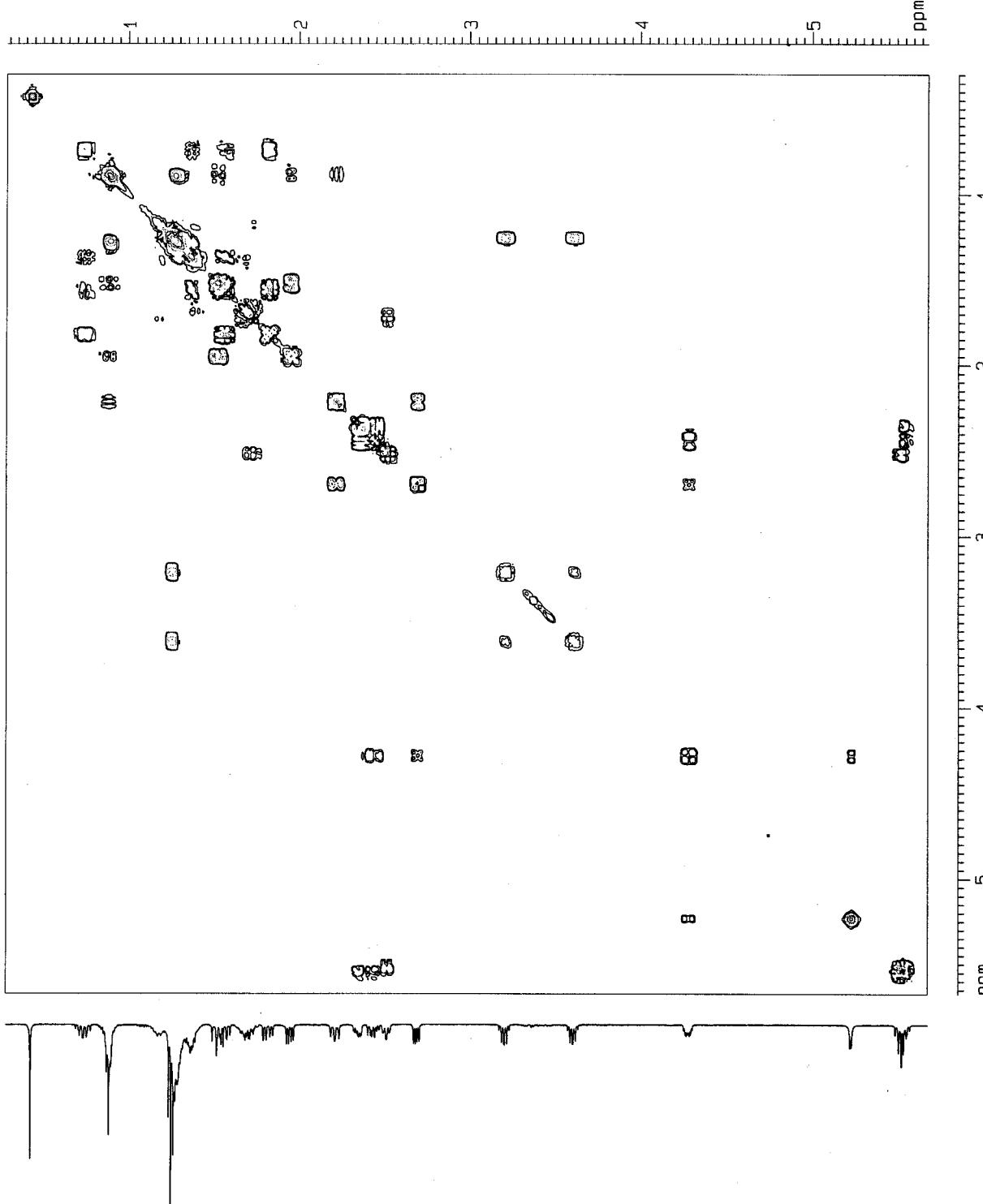
15



COSY 1

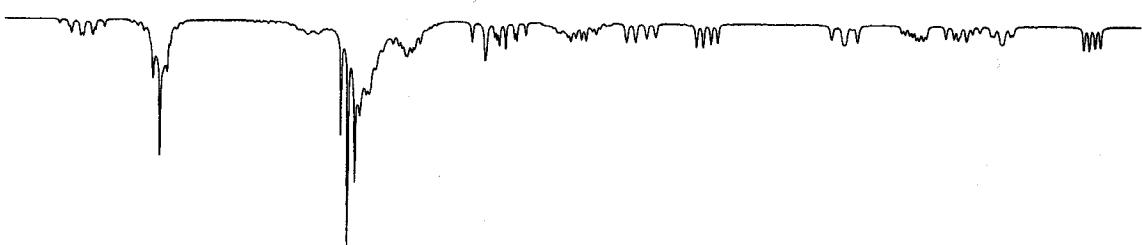
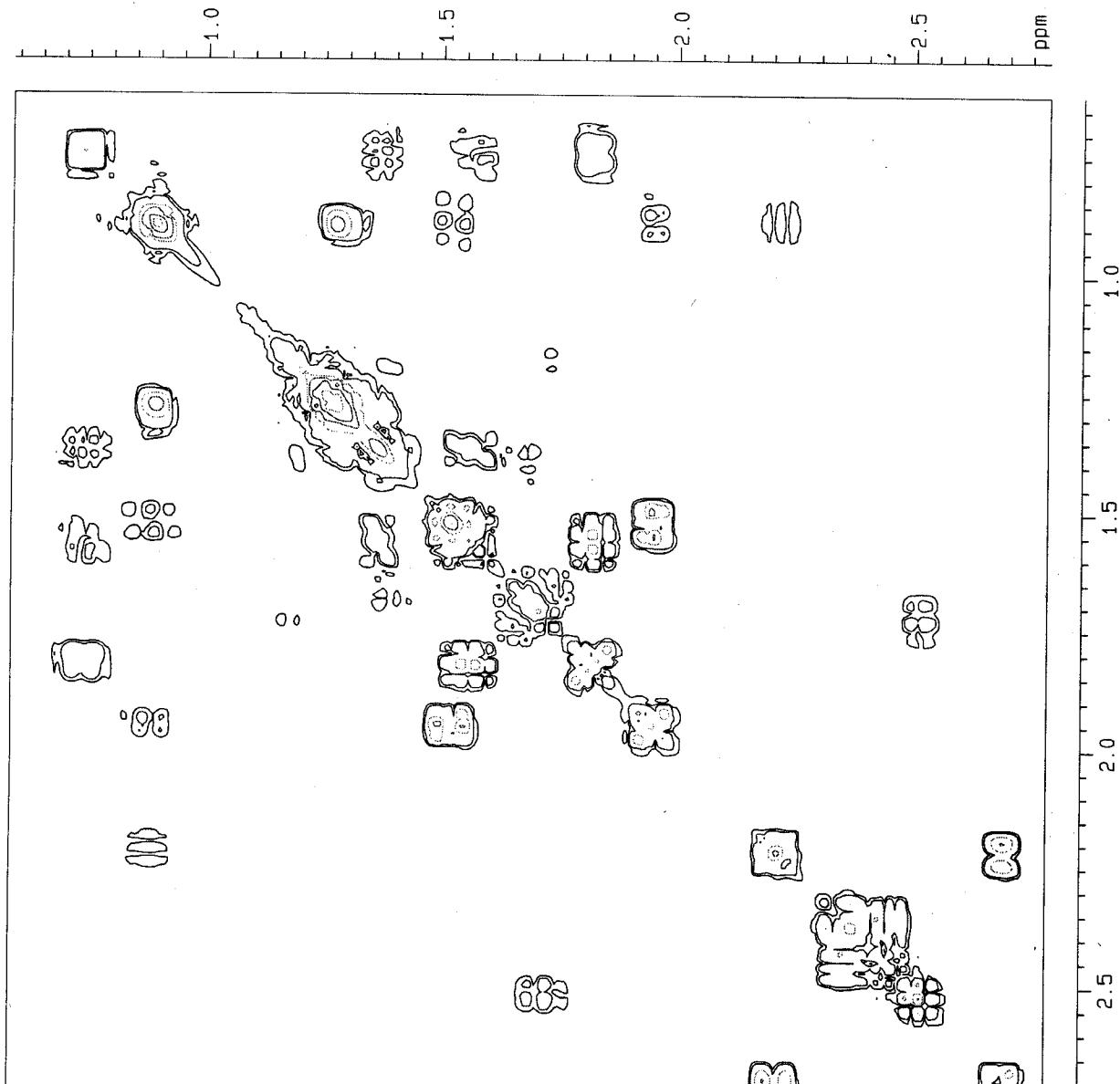


15



cosy 2

15



Current Data Parameters
NAME: ri-106
EXPT: 1
PRQCNO: 1

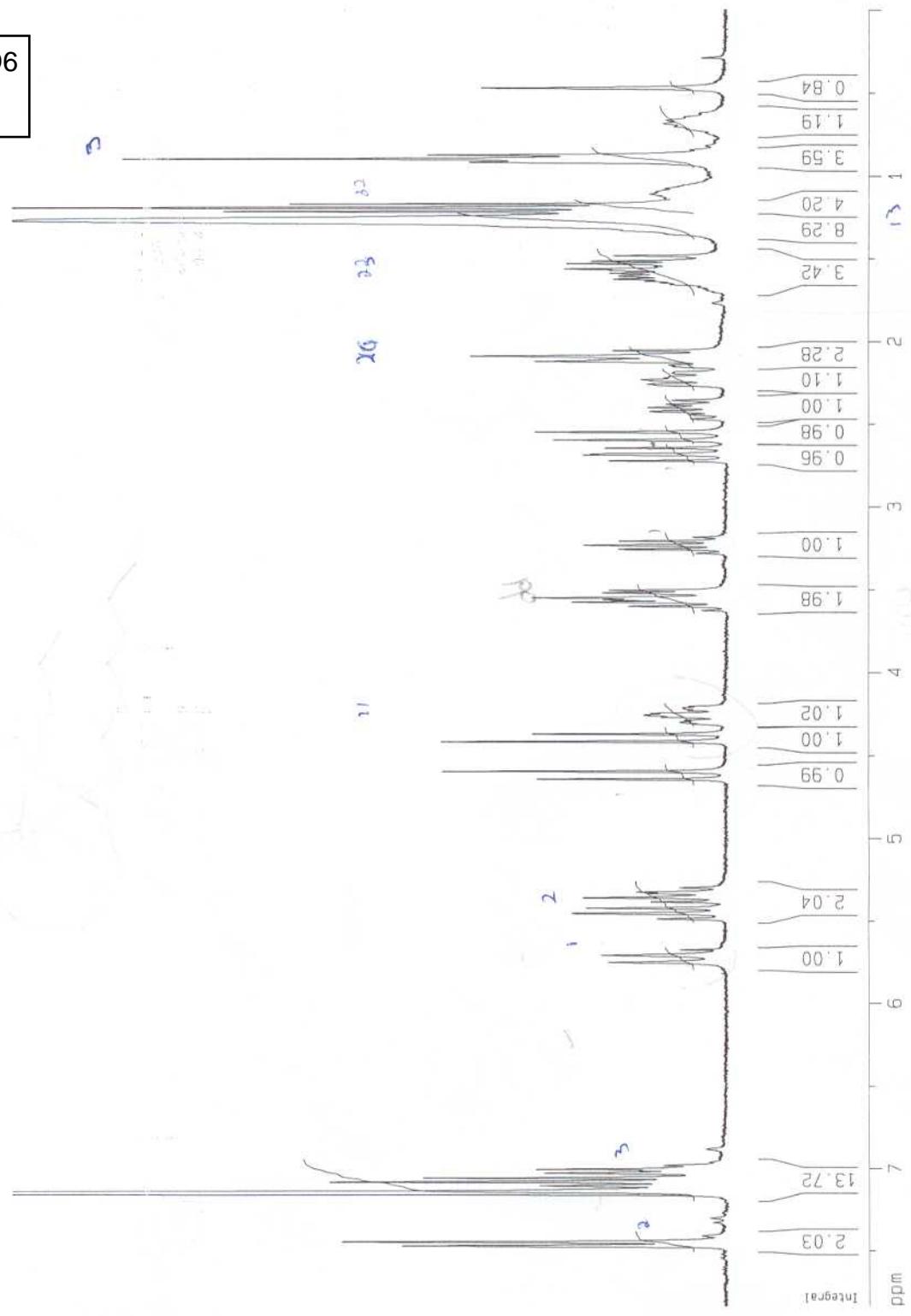
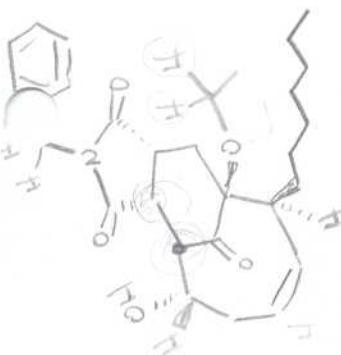
F2 - Acquisition Parameters
Date - 2003/10/17
Time - 13:15
INSTRUM: av300
PROBHD: 5 mm GNP
PULPROG: zg30
TD: 30720
SOLVENT: CDCl3
NS: 16
DS: 0
SWH: 5081.301 Hz
FIDRES: 0.165407 Hz
AQ: 3.0228980 SEC
RG: 406.4
DW: 98.400 usec
DE: 6.00 usec
TE: 300.0 K
D1: 1.0000000 SEC

==== CHANNEL f1 =====
NUC1: 1H
SI: 65536
SF: 300.13300000 MHz
WDW: EM
SSB: 0
LB: 0.10 Hz
GB: 0
PC: 1.00

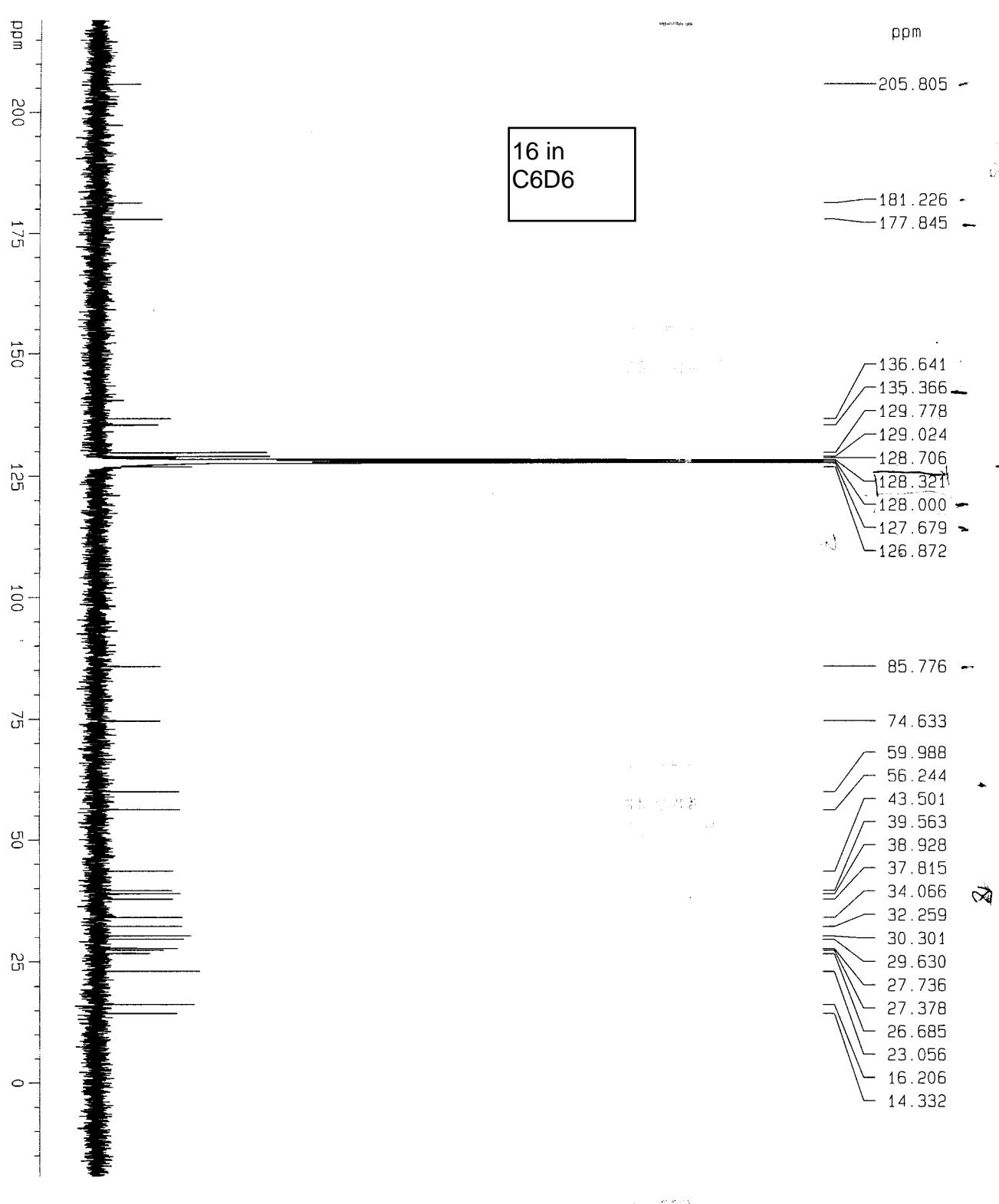
F2 - Processing parameters
SI: 65536
SF: 300.13300000 MHz
WDW: EM
SSB: 0
LB: 0.10 Hz
GB: 0
PC: 1.00

1D NMR plot parameters
CX: 20.00 ppm
CY: 50.00 ppm
F1P: 7.833 ppm
F1: 2350.81 Hz
F2P: -0.004 ppm
F2: -1.23 Hz
PPMCM: 0.39184 ppm/cm
HZCM: 117.60215 Hz/cm

16 in C6D6



RL-106



13C with proton decoupling

Current Data Parameters
 NAME r1-106
 EXPNO 3
 PROCN 1

F2 - Acquisition Parameters
 Date 2003/10/17
 Time 16:11
 INSTRUM av300
 PROBHD 5 mm QNP 1H/1
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 629
 DS 0
 SWH 17985.611 Hz
 FIDRES 0.558877 Hz
 AQ 0.911004 sec
 RG 3649.1
 DM 27.600 usec
 JE 6.00 usec
 TE 30.0 K
 D1 1.0000000 sec
 d11 0.0300000 sec
 d12 0.00002000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 5.00 usec
 PL1 -6.00 dB
 SF01 75.4752653 MHz

===== CHANNEL f2 =====
 CPDRG2 waltz16
 NUC2 1H
 PCP02 70.00 usec
 PL2 -3.00 dB
 PL12 13.48 dB
 PL13 15.63 dB
 SF02 300.1314660 MHz

F2 - Processing parameters
 SI 65336
 SF 75.467260 MHz
 MDW 0
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

10 NMR plot parameters
 CX 20.00 cm
 CY 100.00 cm
 F1P 219.062 ppm
 F1 16532.09 Hz
 -19.260 ppm
 F2P -1453.52 Hz
 F2 11.91609 ppm/cm
 PPMCH 899.28058 Hz/cm

201-78

