

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 methylacetoacetate **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 $\text{NaHCO}_{3(\text{aq})}$ 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 (C_4), 135.6 (CH), 127.0 (CH), 77.8 (CH), 63.9 (CH), 39.5 (CH_2), 38.4 (CH_2), 32.1 (CH_2), 31.8 (CH_2), 29.1 (CH_2), 29.1 (CH_2), 28.7 (CH_2), 22.6 (CH_2), 14.1 (CH_3); 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-triethylsilanyloxy-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 $\text{NH}_4\text{Cl}_{(\text{aq})}$. 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 D_6): 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 D_6): δ 168.7 (C_4), 134.0 (CH), 128.5 (CH), 76.6 (CH), 64.7 (CH), 40.6 (CH_2), 39.6 (CH_2), 32.4 (CH_2), 32.1 (CH_2), 29.5 (CH_2), 29.5 (CH_2), 29.2 (CH_2), 23.0 (CH_2), 14.3 (CH_3), 6.9 (CH_3), 5.0 (CH_2); 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 $\text{NH}_4\text{Cl}_{(\text{aq})}$ 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 (MgSO_4), 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 $\text{NH}_4\text{Cl}_{(\text{aq})}$ 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 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 D_6): 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); ^{13}C NMR (75 MHz, benzene D_6): δ 155.3 (C_4), 148.9 (C_4), 132.8 (CH), 129.6 (CH), 102.6 (CH), 83.5 (CH_2), 75.8 (CH), 63.5 (CH), 63.1 (CH_2), 38.3 (CH_2), 32.6 (CH_2), 32.2 (CH_2), 29.5 (CH_2), 29.5 (CH_2), 29.4 (CH_2), 23.0 (CH_2), 14.4 (CH_3), 14.3 (CH_3); 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^+ ($\text{C}_{18}\text{H}_{30}\text{O}_3$) calculated 294.21950, found 294.21827; mp = $60.4 - 61.9^{\circ}\text{C}$.

2-Non-1-enyl-6-vinyl-3,4-dihydro-2H-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 $\text{NH}_4\text{Cl}_{(\text{aq})}$ 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 (MgSO_4), 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 (C_4), 132.8 (CH), 132.7 (CH), 129.6 (CH), 114.5 (CH_2), 107.1 (CH), 73.4 (CH), 63.6 (CH), 38.4 (CH_2), 32.6 (CH_2), 32.2 (CH_2), 29.5 (CH_2), 29.5 (CH_2), 29.4 (CH_2), 23.0 (CH_2), 14.3 (CH_3); 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-dioxacyclopenta[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 (C_4), 132.6 (CH), 131.6 (C_4), 130.7 (CH), 117.7 (C_4), 74.9 (CH), 72.2 (CH), 65.5 (CH_2), 50.4 (CH), 39.4 (CH), 38.6 (CH_2), 36.5 (CH), 35.5 (CH_2), 32.6 (CH_2), 32.2 (CH_2), 32.0 (CH_2), 29.5 (CH_2), 29.5 (CH_2), 29.4 (CH_2), 23.0 (CH_2), 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-dioxo-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 μmol) 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-dioxo-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 (C_4), 170.8 (C_4), 146.2 (C_4), 138.1 (C_4), 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 (CH_2), 32.2 (CH_2), 31.8 (CH_2), 29.1 (CH_2), 28.8 (CH_2), 23.8 (CH_2), 22.6 (CH_2), 14.1 (CH_3); 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,7,8,9a,9b-hexahydro-4H-pyrano[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 D_6): 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 D_6): δ 180.8 (C_4), 178.5 (C_4), 136.2 (C_4), 137.7 (C_4), 132.4 (CH), 130.6 (C_4), 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 (CH_2), 65.8 (CH), 42.3 (CH_2), 41.7 (CH), 40.7 (CH), 38.6 (CH_2), 38.4 (CH), 32.2 (CH_2), 31.9 (CH_2), 29.2 (CH_2), 29.1 (CH_2), 27.7 (CH_2), 22.7 (CH_2), 15.6 (CH_3), 14.0 (CH_3); IR (CH_2Cl_2) 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-pyrano[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), $\text{MgBr}_2 \cdot \text{Et}_2\text{O}$ (58.7 mg, 0.227 mmol), Et_3N (0.063 mL, 0.445 mmol), NBM (85.1 mg, 0.445 mmol), CH_2Cl_2 (1 mL) gave cycloadduct **14** (43.6 mg, 79%).

8-Ethoxy-9-heptyl-13-hydroxy-tricyclo[6.5.1.0^{2,6}]tetradec-10-ene-3,14-dione (15)

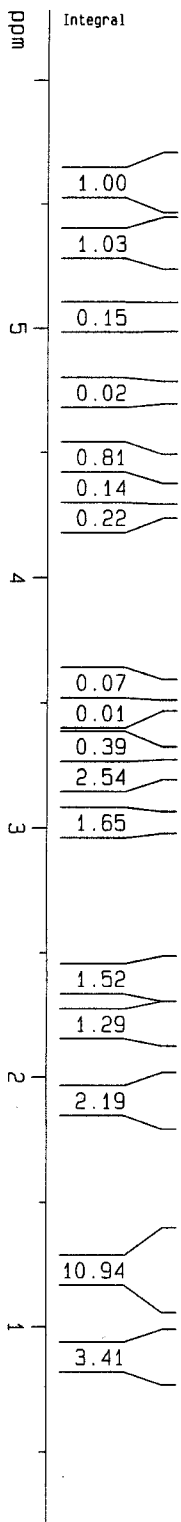
Triethylamine (1 drop) was added to a solution of the **11** (11.8 mg, 31.3 μmol) in toluene (3 mL) in a sealed tube. The solution was degassed with argon for 15 minutes. After this, the tube was sealed and placed in a wax bath at 160°C and the solution was stirred for 24 hours. The resulting solution 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 D_6): 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); ^{13}C NMR (75 MHz, CDCl_3): δ 219.2 (C_4), 209.3 (C_4), 136.4 (CH), 127.9 (CH), 88.5 (C_4), 77.4 (C_4), 70.1 (CH), 60.9 (CH_2), 55.5 (CH), 50.3 (CH), 46.0 (CH), 39.2 (CH), 37.5 (CH_2), 36.1 (CH_2), 32.7 (CH_2), 31.8 (CH_2), 29.6 (CH_2), 29.3 (CH_2), 28.9 (CH_2), 28.2 (CH_2), 27.2 (CH_2), 22.7 (CH_2), 16.1 (CH_3), 14.1 (CH_3); 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^+ ($\text{C}_{23}\text{H}_{36}\text{O}_4$) calculated 376.26136, found 376.26303.

4-Benzyl-8-ethoxy-9-heptyl-13-hydroxy-4-aza-tricyclo[6.5.1.0^{2,6}]tetradec-10-ene-3,5,14-trione (16)

Triethylamine (1 drop) was added to a solution of **14** (18 mg, 0.04 mmol) 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 D_6): 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 = 7.1 Hz, 3H); ^{13}C NMR (75 MHz, benzene D_6): δ 205.8 (C_4), 181.2 (C_4), 177.8 (C_4), 136.6 (C_4), 135.4 (C_4), 129.8 (CH), 129.0 (CH), 128.7 (CH), 128.3 (CH), 127.7 (CH), 126.9 (CH), 85.8 (C_4), 74.6 (CH), 60.0 (CH_2), 56.2 (CH), 43.5 (CH_2), 39.6 (CH), 38.9 (CH), 37.8 (CH), 34.1 (CH_2), 32.3 (CH_2), 30.3 (CH_2), 29.6 (CH_2), 27.7 (CH_2), 27.4 (CH_2), 26.7 (CH_2), 23.1 (CH_2), 16.2 (CH_3), 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.

8 in C6D6



Current Data Parameters
NAME pa2-140
EXPNO 1
PROCNO 1

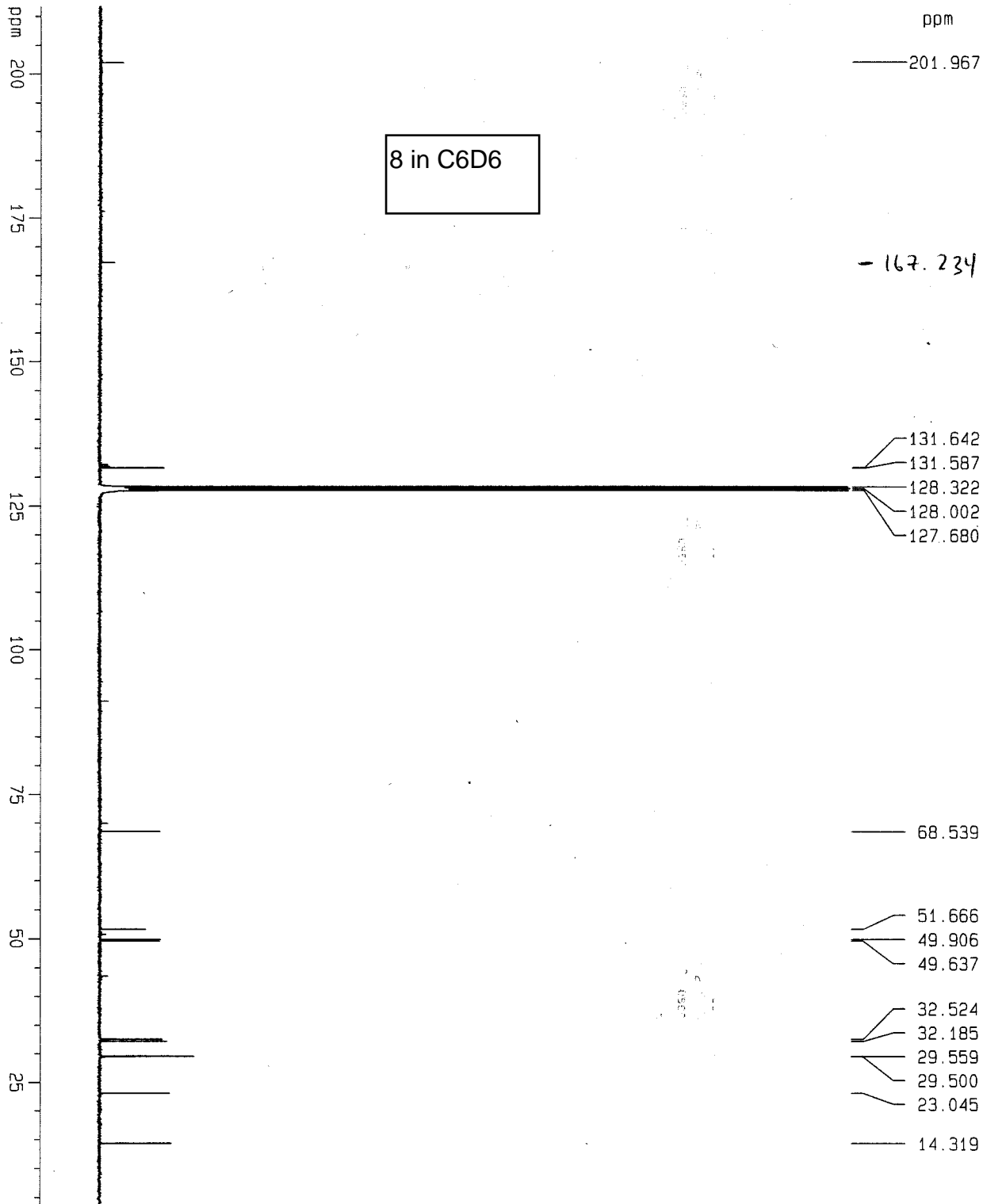
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Date_ 20030403
Time 18.31
INSTRUM av300
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 30720
SOLVENT CDCl3
NS 16
DS 0
SWH 5081.301 Hz
FIDRES 0.165407 Hz
AQ 3.028980 sec
RG 143.7
DE 98.400 usec
TE 300.0 K
D1 1.00000000 sec

===== 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
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 6.279 ppm
F1 1884.53 Hz
F2P 0.220 ppm
F2 66.04 Hz
PPMCM 0.30295 ppm/cm
HZCM 90.92460 Hz/cm

¹³C with proton decoupling



Current Data Parameters

NAME pa2-140
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters

Date_ 20030403
Time 18.54
INSTRUM av300
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 725
DS 0
SWH 17985.611 Hz
FIDRES 0.548877 Hz
AQ 0.9110004 sec
RG 362
DM 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 =====

CPDPRG2 waltz16
NUC2 ¹H
PCPD2 70.00 usec
PL2 -3.00 dB
PL12 13.48 dB
PL13 15.63 dB
SF02 300.1314860 MHz

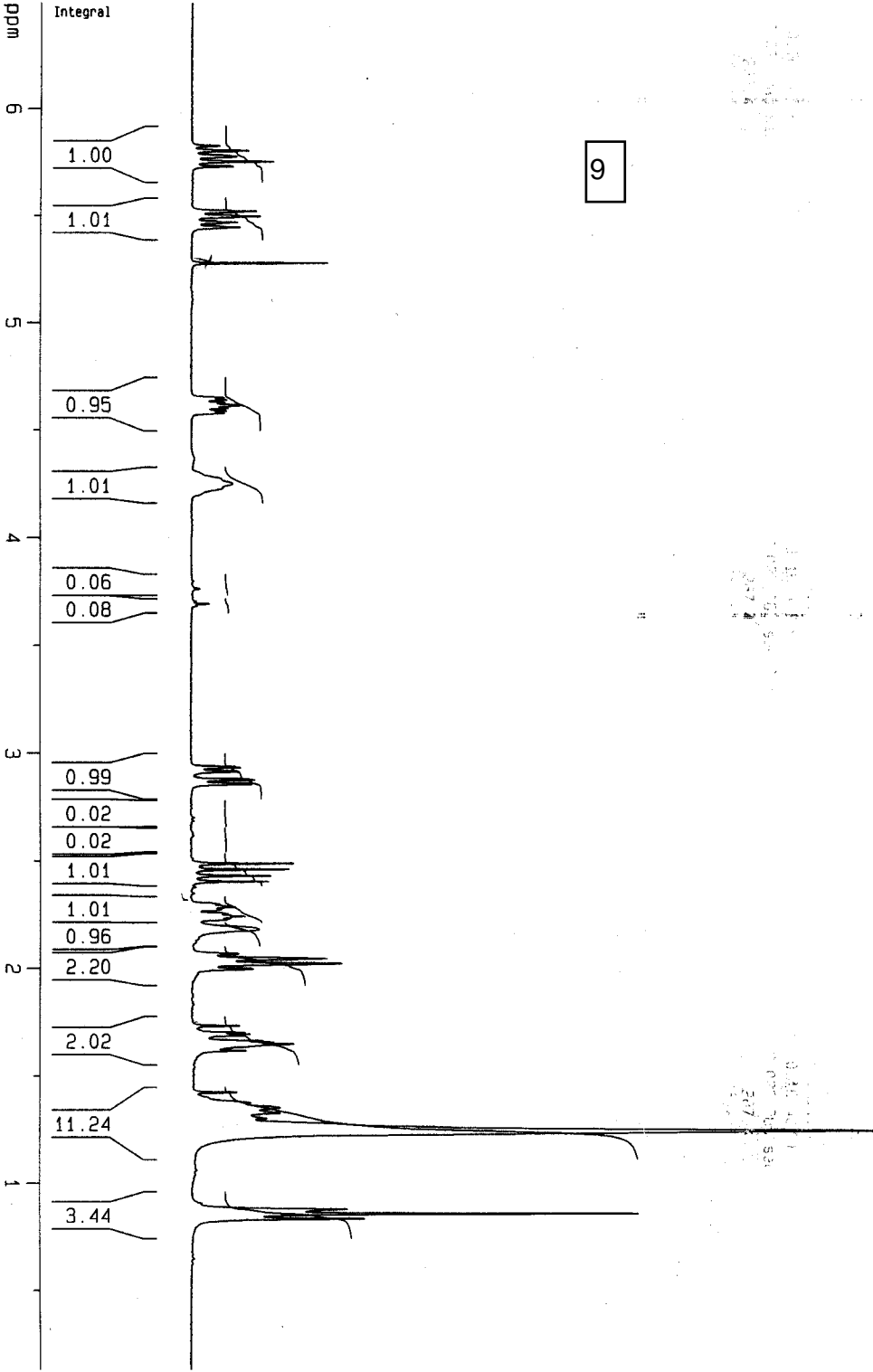
F2 - Processing parameters

SI 65536
SF 75.4677255 MHz
WDW 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 211.698 ppm
F1 15976.38 Hz
F2P 3.561 ppm
F2 268.77 Hz
PPMCM 10.40584 ppm/cm
HZCM 755.38062 Hz/cm

9



Current Data Parameters
NAME pa2-158
EXPNO 1
PROCNO 1

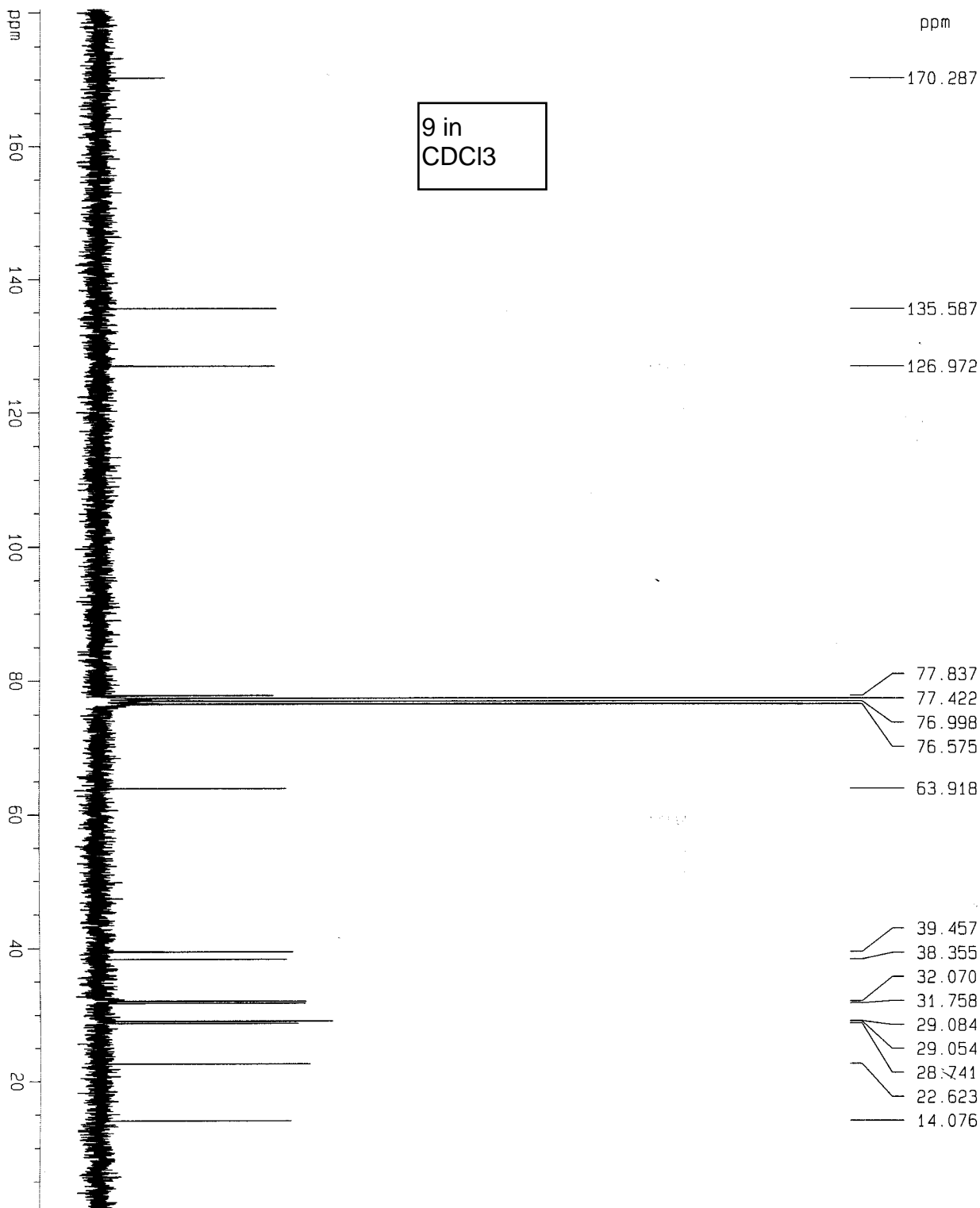
F2 - Acquisition Parameters
Date_ 20030422
Time 17.06
INSTRUM av300
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 30720
SOLVENT CDCl3
NS 16
DS 0
SWH 5081.301 Hz
FIDRES 0.165407 Hz
AQ 3.0228980 sec
RG 287.4
DM 98.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 300.1319477 MHz

F2 - Processing parameters
SI 65536
SF 300.1300002 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 6.489 ppm
F1 1947.56 Hz
F2P 0.134 ppm
F2 40.30 Hz
PPMCM 0.31774 ppm/cm
HZCM 95.36236 Hz/cm

¹³C with proton decoupling



Current Data Parameters

NAME	pac-158
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20030422
Time	17.16
INSTRUM	av300
PROBHD	5 mm GNP 1H/1
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl ₃
NS	354
DS	0
SWH	17985.611 Hz
FIDRES	0.54877 Hz
AQ	0.911004 sec
RG	574.7
DW	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	¹³ C
P1	5.00 usec
PL1	-6.00 dB
SFO1	75.4752653 MHz

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

CPDPRG2	waltz16
NUC2	¹ H
PCPD2	70.00 usec
PL2	-3.00 dB
PL12	13.48 dB
PL13	15.63 dB
SFO2	300.1314860 MHz

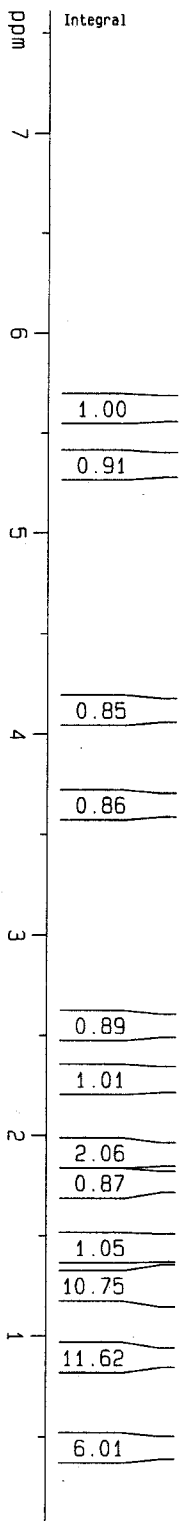
F2 - Processing parameters

SI	65536
SF	75.4677486 MHz
WDW	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	180.534 ppm
F1	13624.53 Hz
F2P	0.968 ppm
F2	73.04 Hz
PPMCM	8.97833 ppm/cm
HZCM	677.57446 Hz/cm

10 in C6D6



Current Data Parameters

NAME pa2-158

EXPNO 1

PROCNO 1

F2 - Acquisition Parameters

Date_ 20040115

Time 17.59

INSTRUM av300

PROBHD 5 mm QNP 1H/1

PULPROG zg30

TD 30720

SOLVENT CDCl3

NS 16

DS 0

SMH 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 1.00000000 sec

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

NUC1 1H

P1 10.50 usec

PL1 -3.00 dB

SFO1 300.1319477 MHz

F2 - Processing parameters

SI 65536

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 7.613 ppm

F1 2284.78 Hz

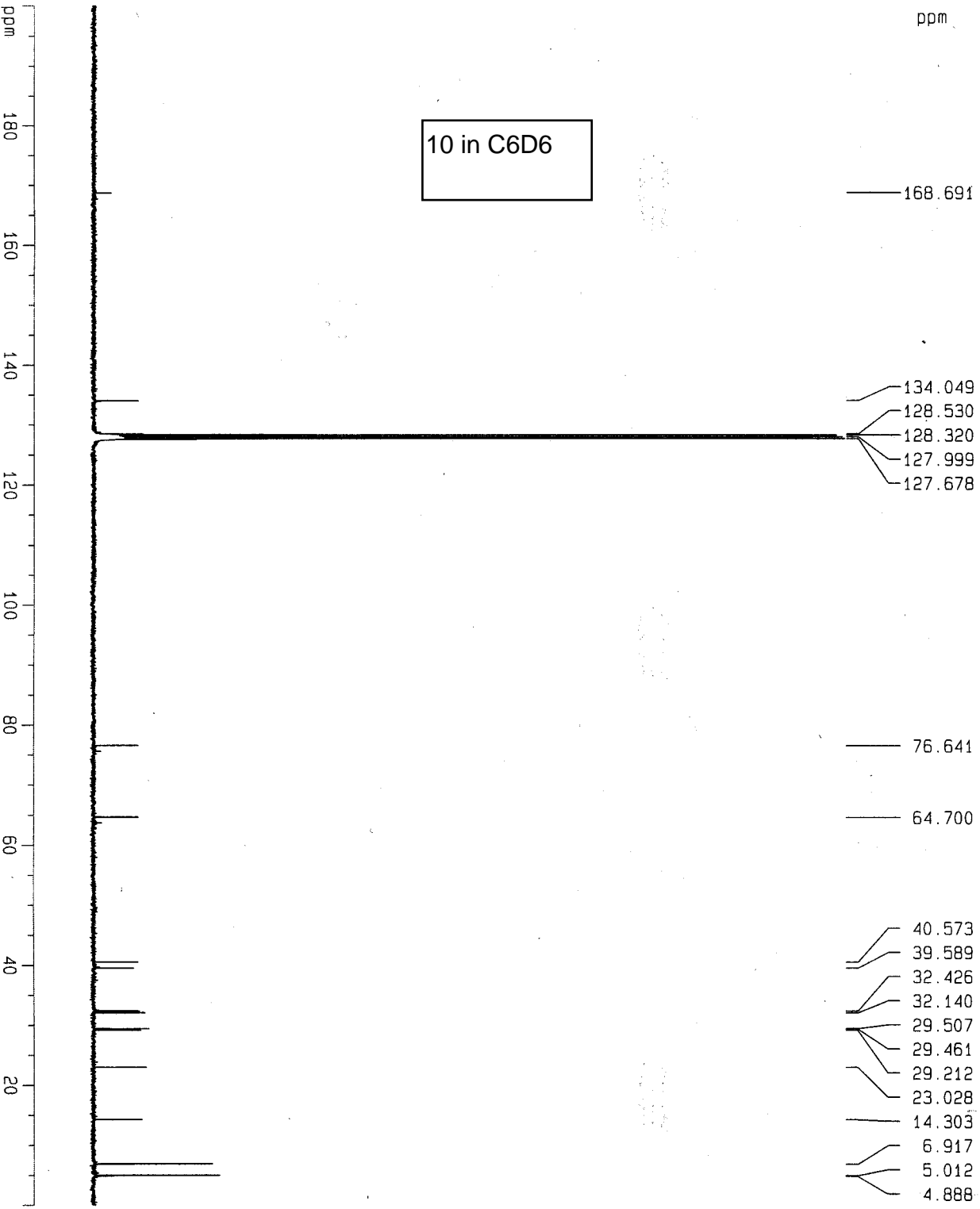
F2P 0.075 ppm

F2 22.36 Hz

PPMCM 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_ 20040115
Time 18.13

INSTRUM av300
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30

TD 32768
SOLVENT COC13
NS 446

DS 0
SWH 17985.611 Hz
FIDRES 0.548877 Hz

RG 0.911004 sec
RG 3649.1
DM 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

SFO1 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

SFO2 300.1314860 MHz

F2 - Processing parameters
SI 65536
SF 75.4677252 MHz

WDM EM
SSB 0
LB 1.00 Hz

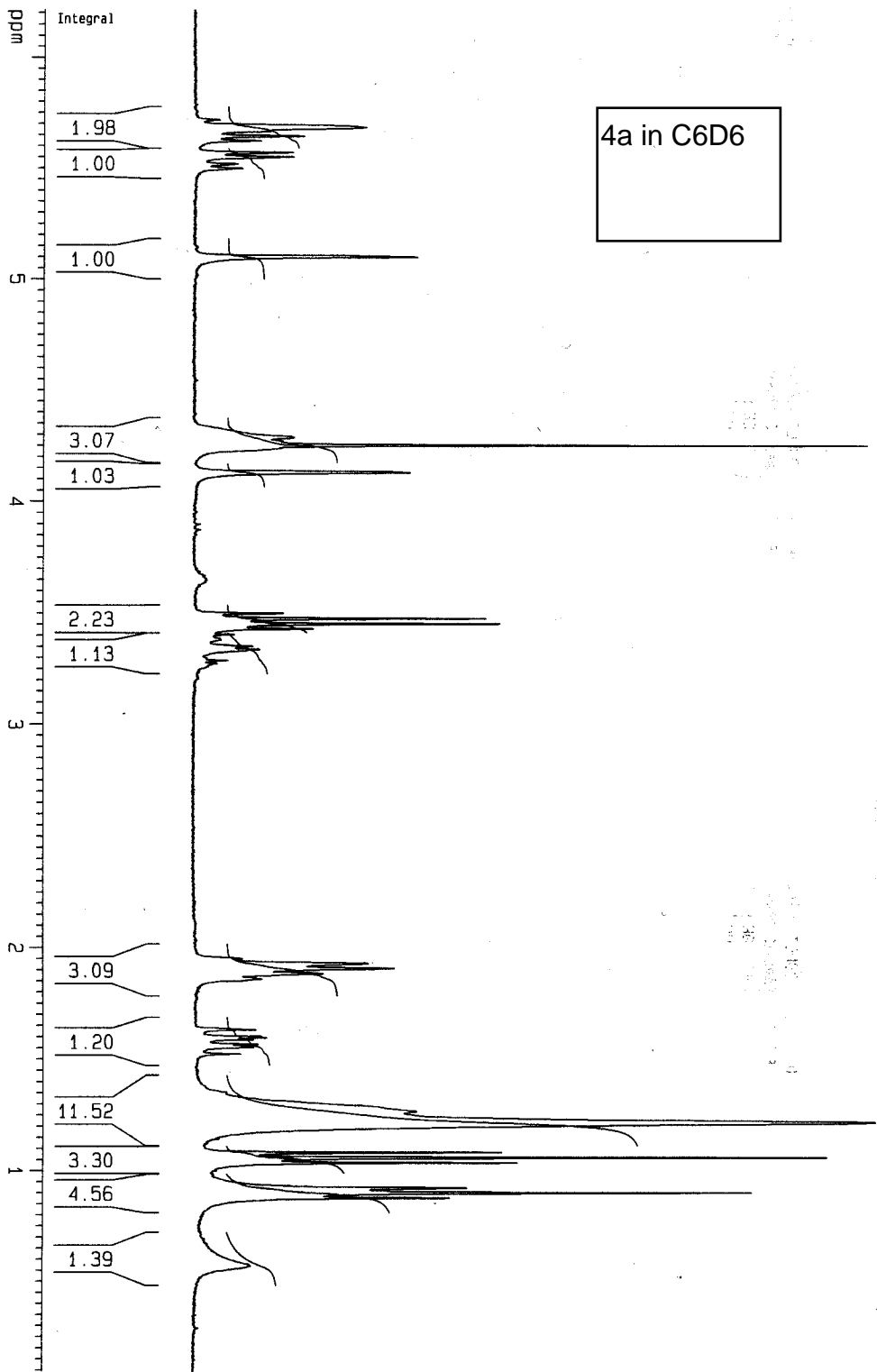
GB 0
PC 1.40

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
PPNCH 10.00000 ppm/cm
HZCM 754.67725 Hz/cm

4a in C6D6



Current Data Parameters
NAME pa3-10
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030527
Time 13.45

INSTRUM av300
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 30720
SOLVENT CDCl3
NS 16
DS 0

SMH 5081.301 HZ
FIDRES 0.165407 HZ
AQ 3.0228980 sec

RG 362
DW 98.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

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

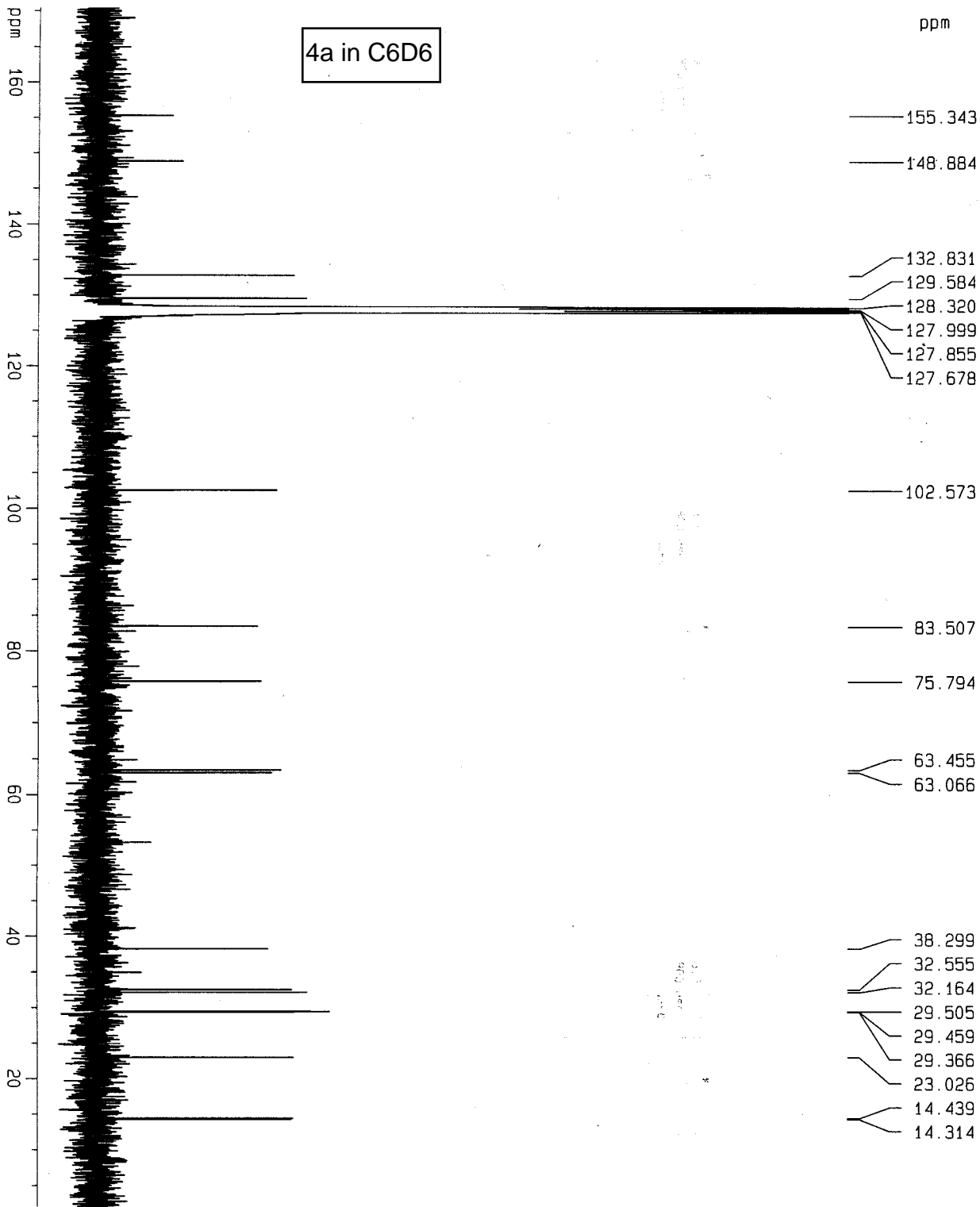
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SF01 300.1319477 MHz

F2 - Processing parameters

SI 65536
SF 300.1299997 MHz
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 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

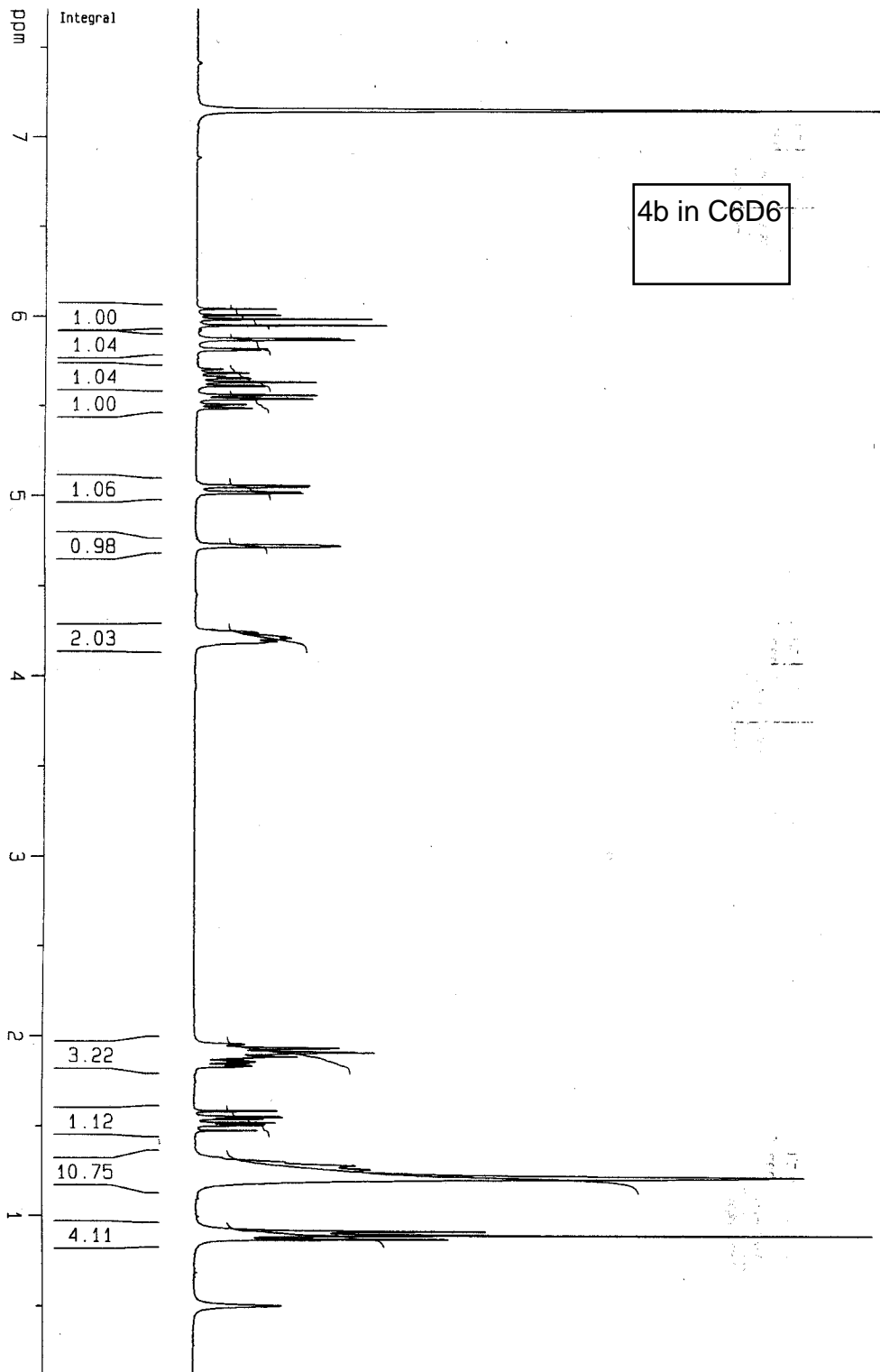
F2 - Acquisition Parameters
Date_ 20030527
Time 13.55
INSTRUM av300
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 802
DS 0
SWH 17985.611 Hz
FIDRES 0.548877 Hz
AQ 0.9110004 sec
RG 2298.8
DM 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

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

===== CHANNEL f2 =====
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
WOM 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
PPHMC 8.42071 ppm/cm
HZCM 635.49158 Hz/cm



Current Data Parameters
 NAME p33-3
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040115
 Time 18.42

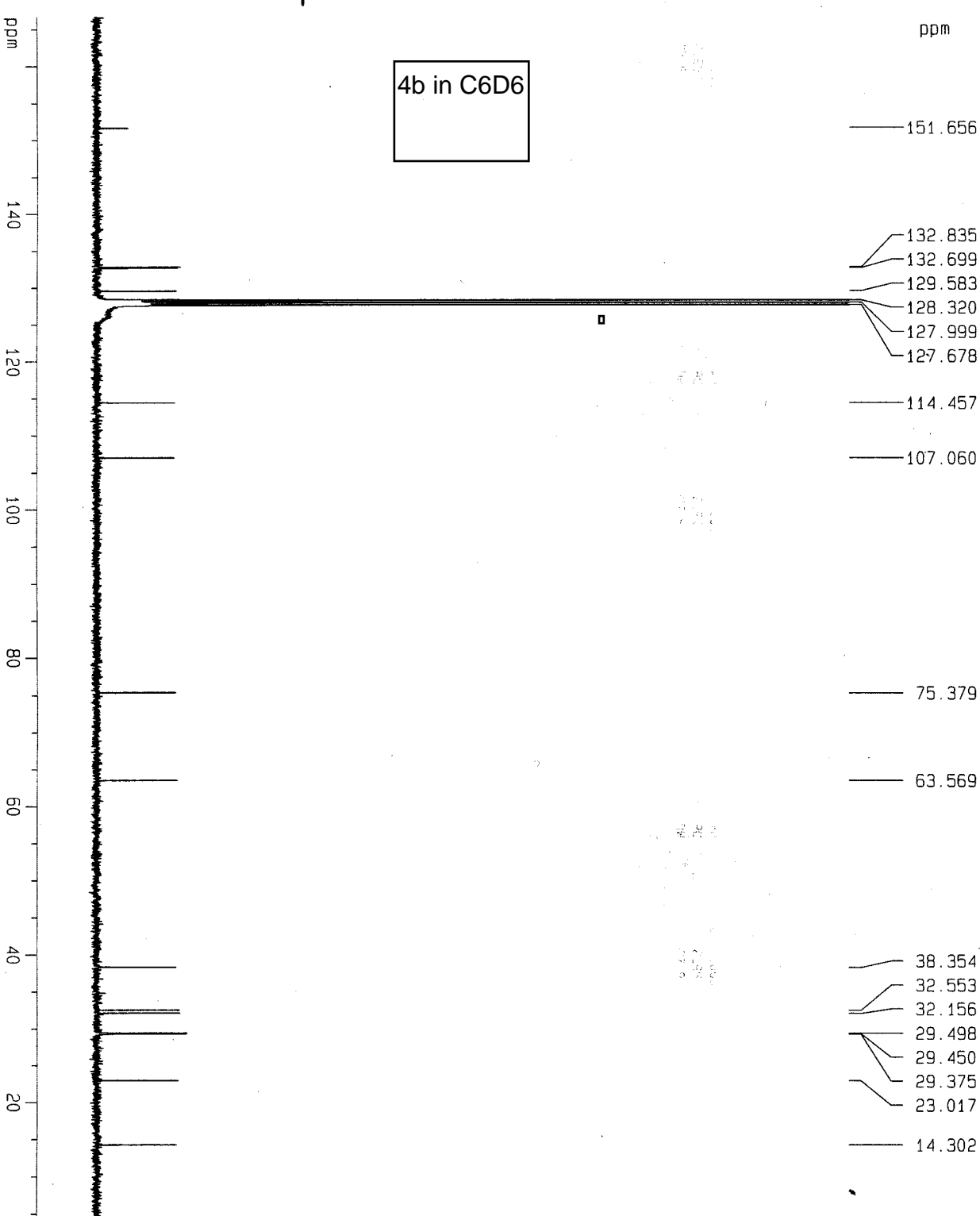
INSTRUM av300
 PROBHD 5 mm QNP 1H/1
 PULPROG zg30
 TD 30720
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 5081.301 Hz
 FIDRES 0.165407 Hz
 AQ 3.0226980 sec
 RG 287.4
 DW 98.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== 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
 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 113.85335 Hz/cm

¹³C with proton decoupling



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 zgpg30

TD 32768
SOLVENT CDCl3

NS 586
DS 0

SWH 17985.611 Hz
FIDRES 0.548877 Hz

AQ 0.9110004 sec
RG 2298.8

DM 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

SFO1 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
SFO2 300.1314860 MHz

F2 - Processing parameters
SI 65536

SF 75.4677255 MHz
WDW EM

SSB 0
LB 1.00 Hz

GB 0
PC 1.40

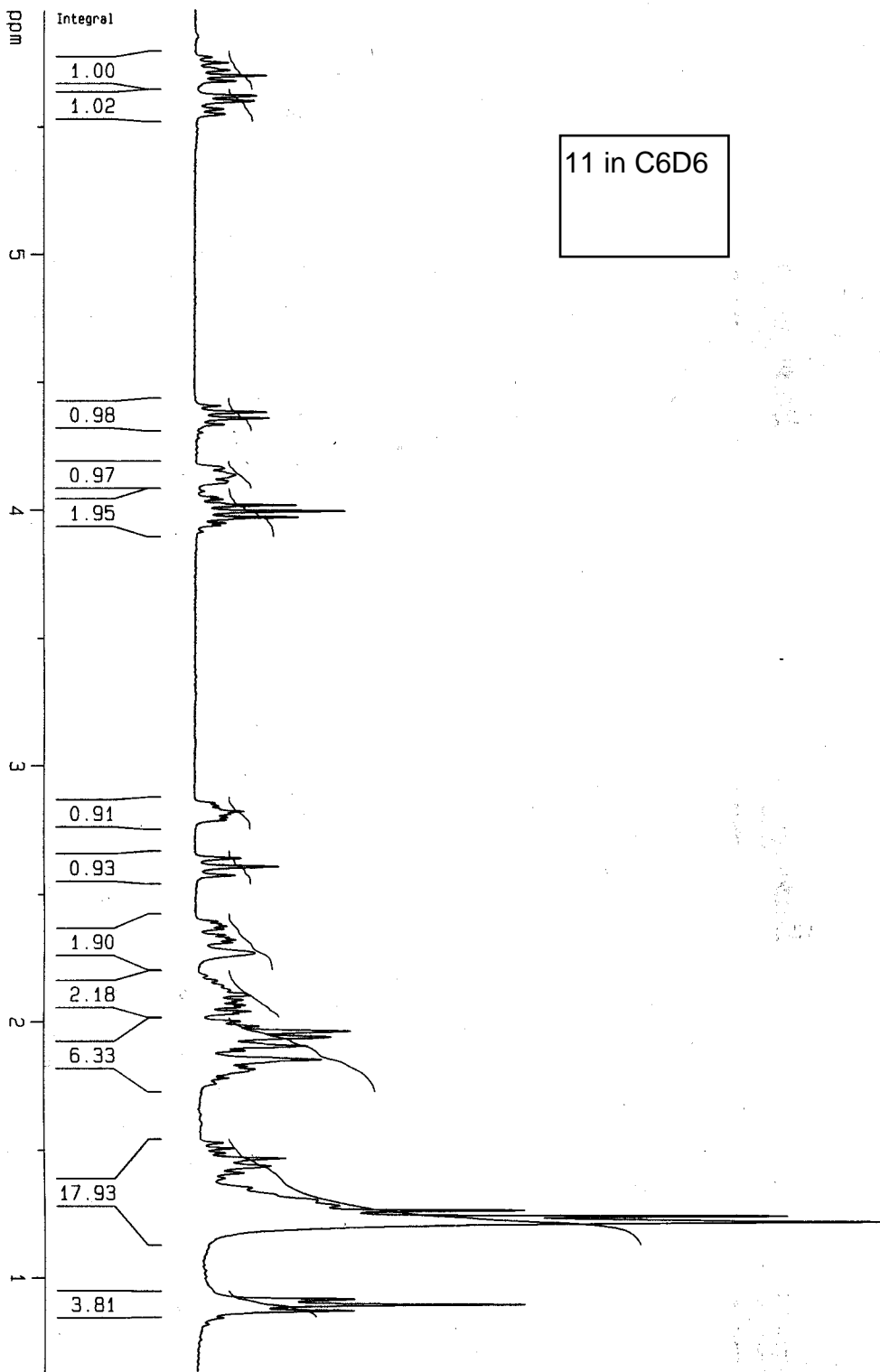
1D NMR plot parameters
CX 20.00 cm

CY 25.00 cm
F1P 166.614 ppm

F1 12573.96 Hz
F2P 4.527 ppm

F2 341.67 Hz
PPMCM 8.10432 ppm/cm

HZCM 611.51450 Hz/cm



Current Data Parameters
 NAME pa3-89
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20031022
 Time 22.54

INSTRUM av300
 PROBHD 5 mm QNP 1H/1
 PULPROG zg30

TD 30720
 SOLVENT ~~acetone~~ C₆D₆
 NS 16
 DS 0

SWH 5081.301 Hz
 FIDRES 0.166407 Hz
 AQ 3.0228980 sec

RG 287.4
 DW 98.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

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

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

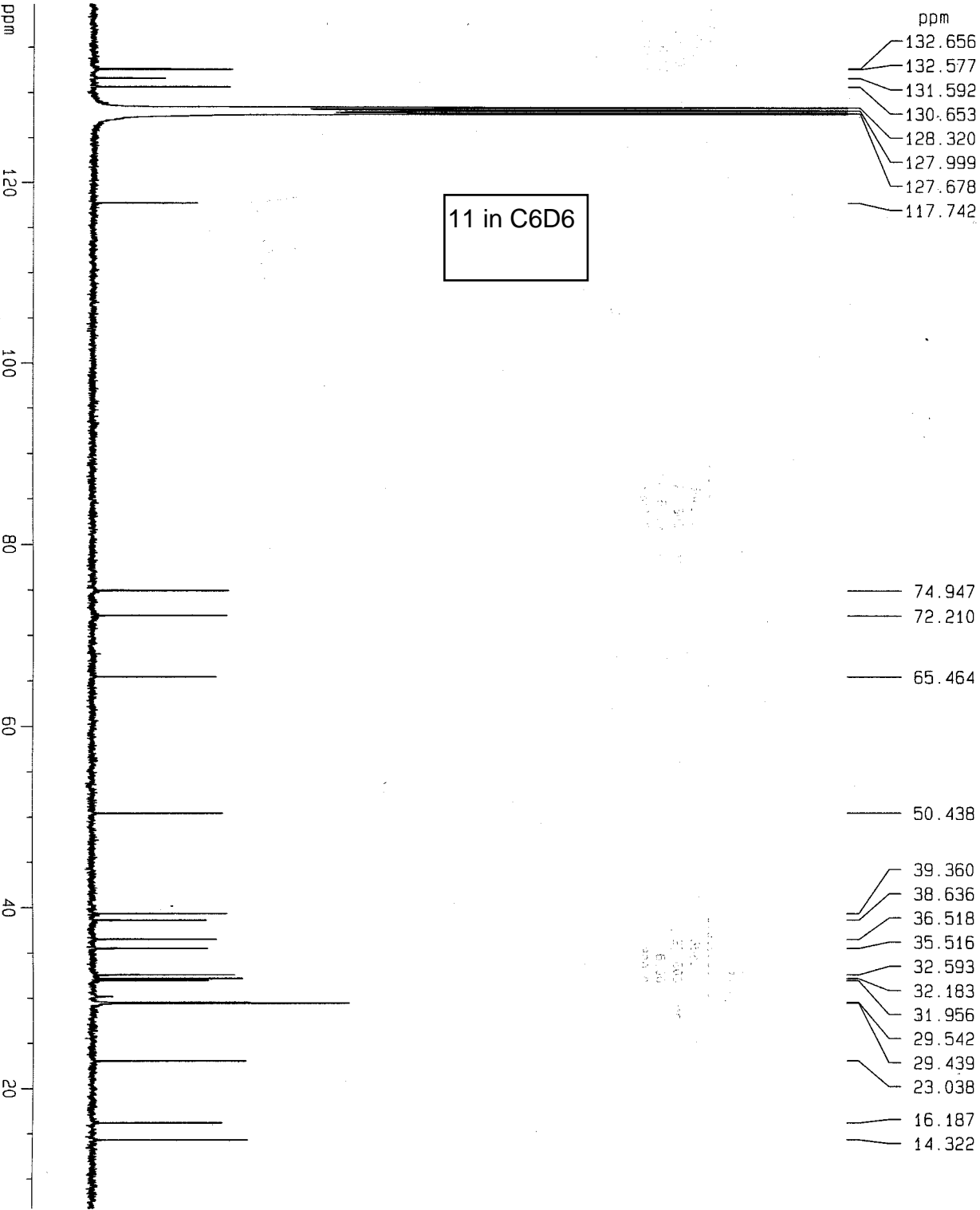
F2 - Processing parameters

SI 65536
 SF 300.1299996 MHz
 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 5.958 ppm
 F1 1788.24 Hz
 F2P 0.630 ppm
 F2 189.23 Hz
 PPMCM 0.26639 ppm/cm
 HZCM 79.95053 Hz/cm

¹³C with proton decoupling



Current Data Parameters
NAME pa3-89
EXPNO 1
PROCNO 2

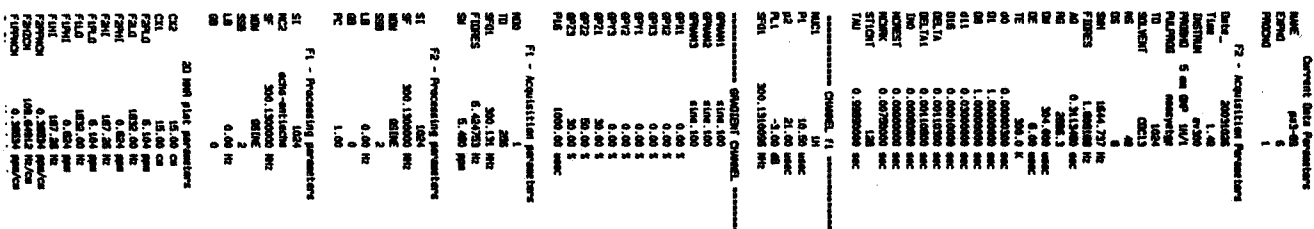
F2 - Acquisition Parameters
Date_ 20031023
Time 2.54
INSTRUM av300
PROBHD 5 mm GNP 1H/1
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 7168
DS 0
SWH 17985.611 Hz
FIDRES 0.548877 Hz
AQ 0.9110004 sec
RG 3649.1
DM 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

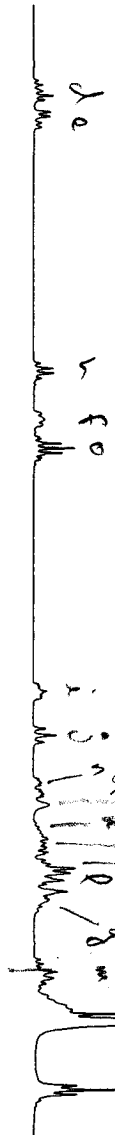
===== CHANNEL f1 =====
NUC1 ¹³C
P1 5.00 usec
PL1 -6.00 dB
SF01 75.475653 MHz

===== 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

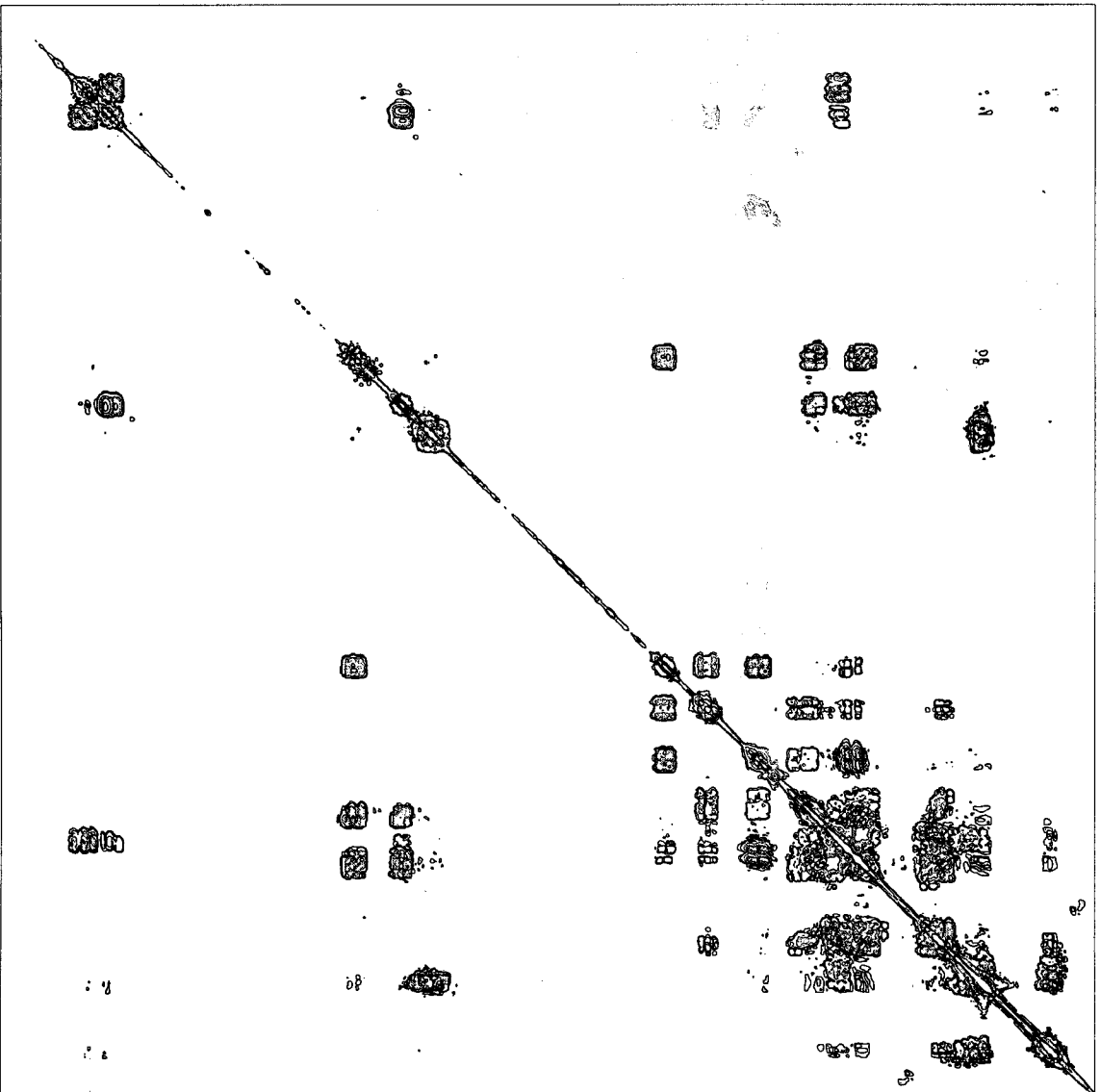
F2 - Processing parameters
SI 65536
SF 75.4677260 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 100.00 cm
F1P 139.732 ppm
F1 10545.27 Hz
F2P 6.739 ppm
F2 508.54 Hz
PPMCM 6.64968 ppm/cm
HZCM 501.83624 Hz/cm





1H COSY



Current Data Parameters
NAME: pas-88
EXPNO: 4
PROCNO: 1

F2 - Acquisition Parameters

Date_: 20031023
Time: 4.34
INSTRUM: spect
PROBHD: 5 mm QNP 1H/1
PULPROG: zgpg30
TD: 65536
SOLVENT: DMSO
DS: 4
DE: 0
TE: 300.2
FIDRES: 0.313460
AQ: 1824.6
RG: 304.000
DE: 6.00
TE: 300.2
D0: 0.0000300
D1: 1.0000000
D13: 0.0000000
D16: 0.0000000
AQ: 0.0000000
RG: 1.0000000
RG: 1.0000000

Channel f1

NUC1: 1H
P0: 5.25
P1: 10.50
P2: -3.00
SFO1: 300.1310203

GRAB1

GRAB1: 100
SFO1: 300.1310203
P0: 5.25
P1: 10.50
P2: -3.00
SFO1: 300.1310203

F1 - Acquisition Parameters

NO: 1
TD: 256
SFO1: 300.1310203
FIDRES: 6.424753
SN: 5.480
SI: 1024
SF: 300.1300000
WDW: SINE
SSB: 0
LB: 0.00
GB: 0
PC: 1.00

F1 - Processing Parameters

SI: 1024
SF: 300.1300000
WDW: SINE
SSB: 0
LB: 0.00
GB: 0

2D NMR Parameters

CX2: 15.00
CX1: 15.00
F2A0: 6.140
F2A1: 1842.66
F2A2: 0.559
F2A3: 187.52
F2A4: 1842.66
F2A5: 0.559
F2A6: 187.52
F2A7: 1842.66
F2A8: 0.559
F2A9: 187.52
F2A10: 1842.66
F2A11: 0.559
F2A12: 187.52
F2A13: 1842.66
F2A14: 0.559
F2A15: 187.52
F2A16: 1842.66
F2A17: 0.559
F2A18: 187.52
F2A19: 1842.66
F2A20: 0.559

EXPNO	4
PROCNO	1

DATE_	20031023
Time	4.34
INSTRUM	av300

TD	1024
SOLVENT	CCl3

SMH	1644.737 Hz
FIDRES	1.606188 Hz
AD	0.3143450 sec

```
DE      6.00 usec
TE      300.0 K
```

d13	0.00000400 sec
D16	0.00010000 sec

```
***** CHANNEL 1 *****
```

P1	10.50 usec
PL1	-3.00 dB

```
----- GHAJENI CHANNEL -----
GPMANI      sine.100
GPMAN2      sine.100
```

GPY1	0.00 %
GPY2	0.00 %

P16	1000.00 usec
-----	--------------

10	230
SFO1	300.131 MHz
FIDRES	6.424753 Hz

F2 - Processing parameters	SI
1024	

SSB	0
LB	0.00 Hz
0	0

SI	F1 - Processing parameters
1024	

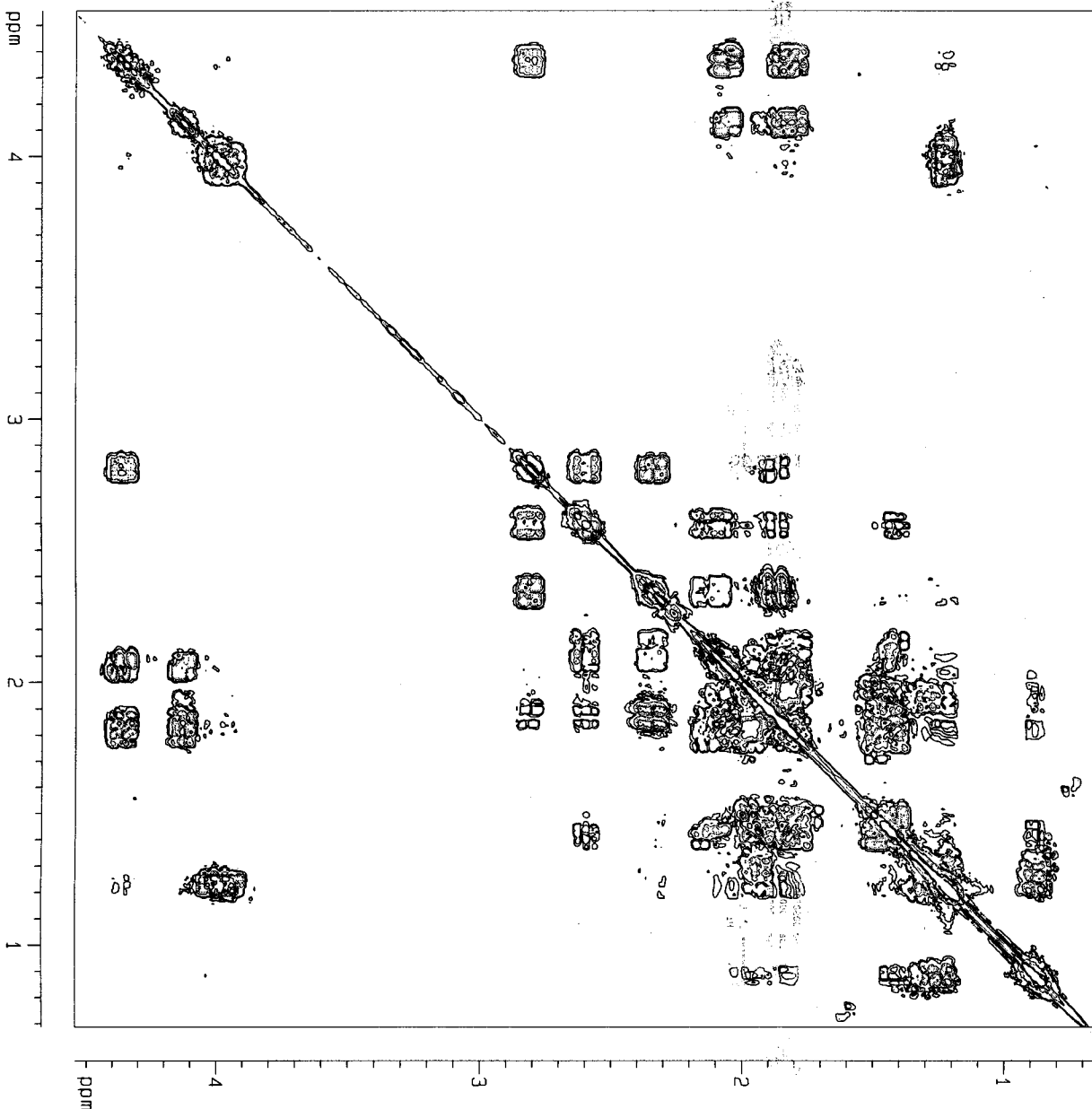
MON	SINE
SSB	0

20 NMR plot parameters
15.00 cm
CX2

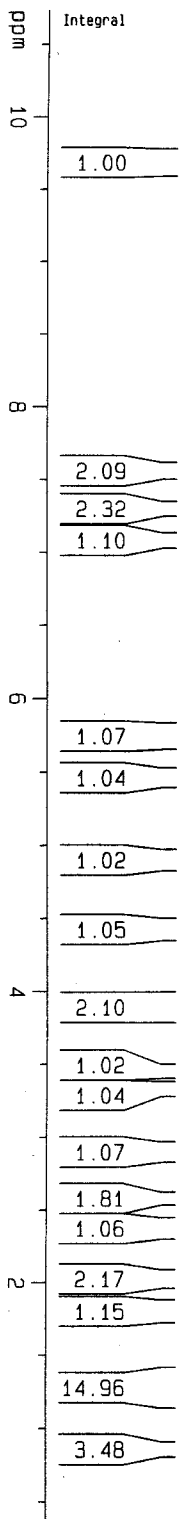
F2L0	1367.22 Hz
F2PHI	0.686 ppm

F1L0	1364.01 Hz
F1P0I	0.654 ppm
F1H1	196.31 Hz

F1PPMCH	0.25938 ppm/cm
F1HZCM	77.84660 Hz/cm



12



Current Data Parameters
 NAME pa3-74c
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20031006
 Time 17.00

INSTRUM av300

PROBHD 5 mm QNP 1H/1

PULPROG zg30

TD 30720

SOLVENT CDCl3

NS 16

DS 0

SMH 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 1.00000000 sec

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

NUC1 1H

P1 10.50 usec

PL1 -3.00 dB

SFO1 300.1319477 MHz

F2 - Processing parameters

SI 65536

SF 300.1300000 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 10.728 ppm

F1 3219.94 HZ

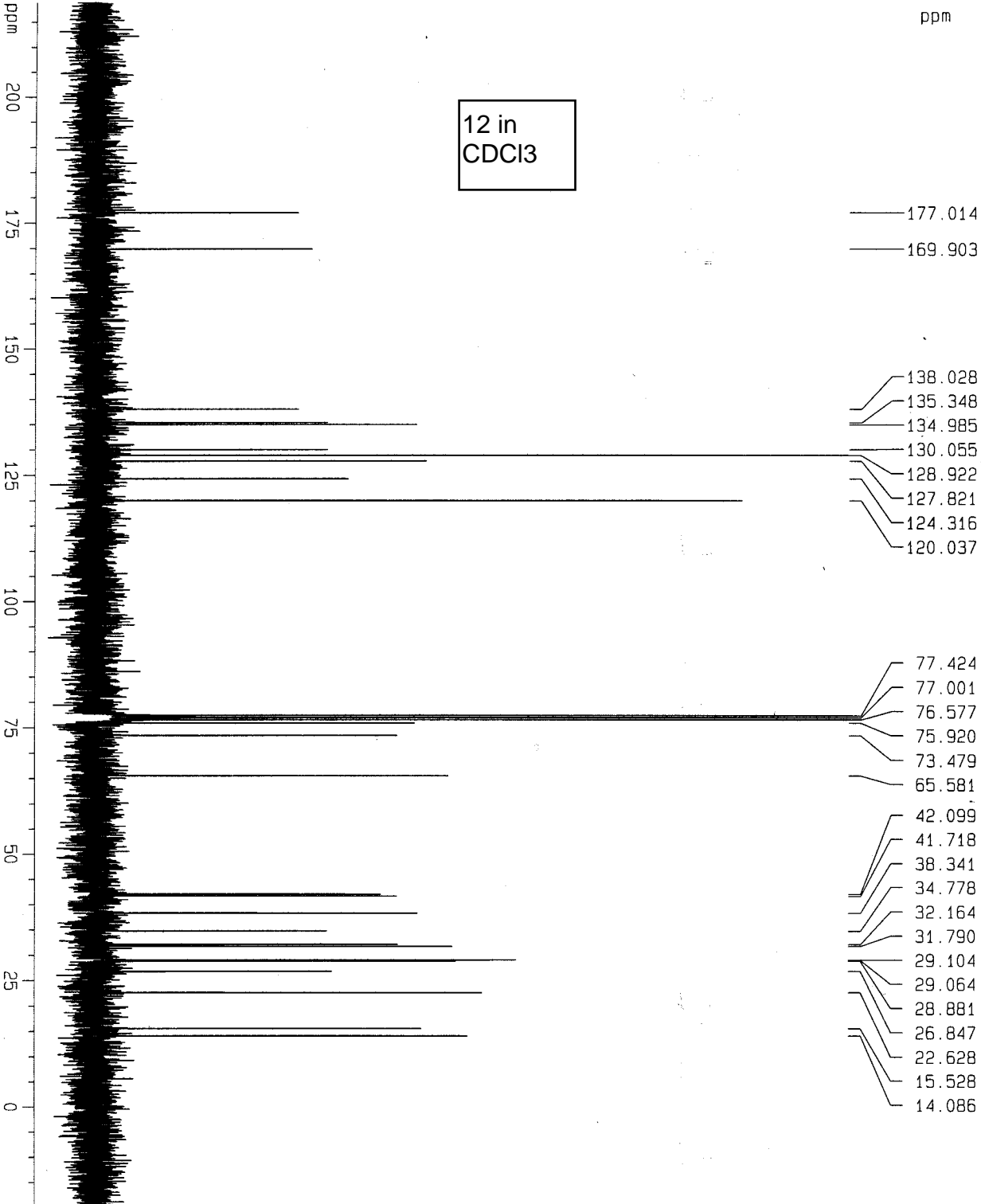
F2P 0.367 ppm

F2 110.03 HZ

PPMCM 0.51809 ppm/cm

HZCM 155.48545 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 av300
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 371
DS 0
SWH 17985.611 Hz
FIDRES 0.548877 Hz
AQ 0.9110004 sec
RG 8192
DM 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
SFO1 75.4752653 MHz

===== CHANNEL f2 =====
CPDPRG2 waitz16
NUC2 ¹H
PCPD2 70.00 usec
PL2 -3.00 dB
PL12 13.48 dB
PL13 15.63 dB
SFO2 300.1314860 MHz

F2 - Processing parameters
SI 65536
SF 75.4677492 MHz
WDW 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 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

Current Data Parameters
NAME EXPNO PROCNO
p03-74c 5 1

F2 - Acquisition Parameters

Date_ Time
20031006 18.45
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 16384
SOLVENT CDCl3
NS 2
DS 0
SWH 2063.726 Hz
FIDRES 2.301500 Hz
AQ 0.1871000 sec
RG 655.2
DE 5.00 usec
TE 300.0 K
D1 0.00000000 sec
d13 1.00000000 sec
D16 0.00000000 sec
D18 0.00100000 sec
D19 0.00100000 sec
D20 0.00100000 sec
D21 0.00100000 sec
D22 0.00100000 sec
D23 0.00100000 sec
D24 0.00100000 sec
D25 0.00100000 sec
D26 0.00100000 sec
D27 0.00100000 sec
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D84 0.00100000 sec
D85 0.00100000 sec
D86 0.00100000 sec
D87 0.00100000 sec
D88 0.00100000 sec
D89 0.00100000 sec
D90 0.00100000 sec
D91 0.00100000 sec
D92 0.00100000 sec
D93 0.00100000 sec
D94 0.00100000 sec
D95 0.00100000 sec
D96 0.00100000 sec
D97 0.00100000 sec
D98 0.00100000 sec
D99 0.00100000 sec
D100 0.00100000 sec

CHANNEL F1

NUC1 1H
P1 5.25 usec
PL1 0.00 dB
SFO1 300.1310000 MHz

GRADIENT CHANNEL

GP1M1 sine 100
GP1M2 sine 100
GP1M3 sine 100
GP1M4 sine 100
GP1M5 sine 100
GP1M6 sine 100
GP1M7 sine 100
GP1M8 sine 100
GP1M9 sine 100
GP1M10 sine 100
GP1M11 sine 100
GP1M12 sine 100
GP1M13 sine 100
GP1M14 sine 100
GP1M15 sine 100
GP1M16 sine 100
GP1M17 sine 100
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GP1M95 sine 100
GP1M96 sine 100
GP1M97 sine 100
GP1M98 sine 100
GP1M99 sine 100
GP1M100 sine 100

F1 - Acquisition Parameters

NUC1 1H
TD 256
SFO1 300.1316 MHz
FIDRES 11.907676 Hz
AQ 0.1871000 sec
RG 655.2
DE 5.00 usec
TE 300.0 K
D1 0.00000000 sec
d13 1.00000000 sec
D16 0.00000000 sec
D18 0.00100000 sec
D19 0.00100000 sec
D20 0.00100000 sec
D21 0.00100000 sec
D22 0.00100000 sec
D23 0.00100000 sec
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D25 0.00100000 sec
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D92 0.00100000 sec
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D94 0.00100000 sec
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D96 0.00100000 sec
D97 0.00100000 sec
D98 0.00100000 sec
D99 0.00100000 sec
D100 0.00100000 sec

F2 - Processing Parameters

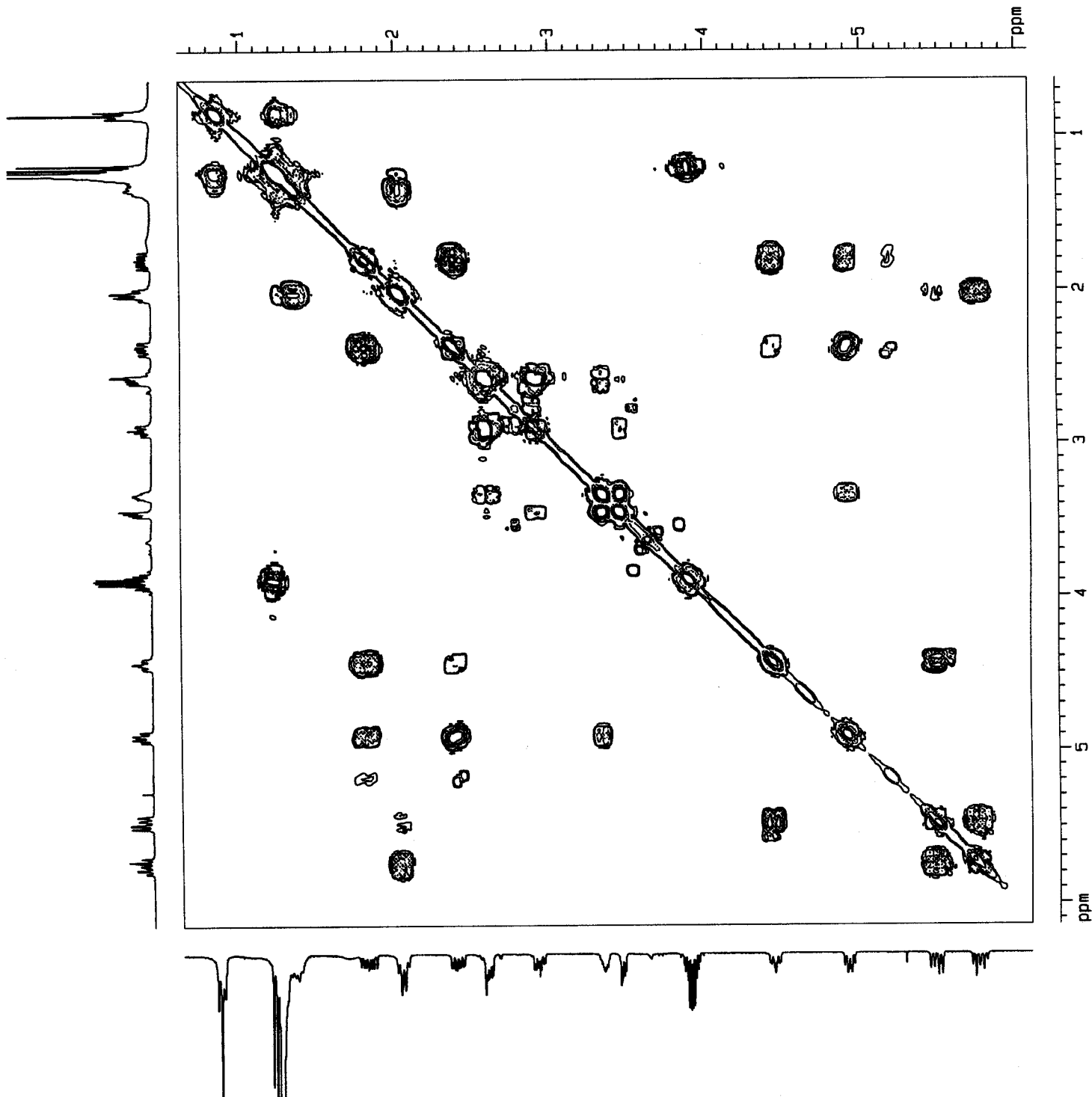
SI 1024
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

F1 - Processing Parameters

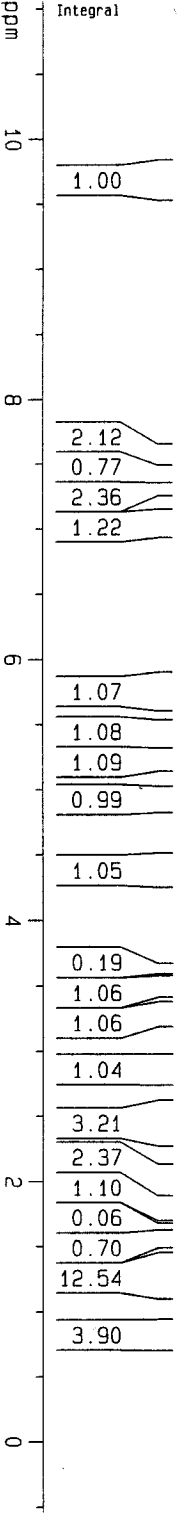
SI 1024
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

2D NMR plot parameters

C2 15.00 cm
C1 15.00 cm
F2R0 0.141 ppm
F2R1 1843.22 Hz
F2R2 0.000 ppm
F2R3 188.48 Hz
F2R4 0.000 ppm
F2R5 188.48 Hz
F2R6 0.000 ppm
F2R7 188.48 Hz
F2R8 0.000 ppm
F2R9 188.48 Hz
F2R10 0.000 ppm
F2R11 188.48 Hz
F2R12 0.000 ppm
F2R13 188.48 Hz
F2R14 0.000 ppm
F2R15 188.48 Hz
F2R16 0.000 ppm
F2R17 188.48 Hz
F2R18 0.000 ppm
F2R19 188.48 Hz
F2R20 0.000 ppm
F2R21 188.48 Hz
F2R22 0.000 ppm
F2R23 188.48 Hz
F2R24 0.000 ppm
F2R25 188.48 Hz
F2R26 0.000 ppm
F2R27 188.48 Hz
F2R28 0.000 ppm
F2R29 188.48 Hz
F2R30 0.000 ppm
F2R31 188.48 Hz
F2R32 0.000 ppm
F2R33 188.48 Hz
F2R34 0.000 ppm
F2R35 188.48 Hz
F2R36 0.000 ppm
F2R37 188.48 Hz
F2R38 0.000 ppm
F2R39 188.48 Hz
F2R40 0.000 ppm
F2R41 188.48 Hz
F2R42 0.000 ppm
F2R43 188.48 Hz
F2R44 0.000 ppm
F2R45 188.48 Hz
F2R46 0.000 ppm
F2R47 188.48 Hz
F2R48 0.000 ppm
F2R49 188.48 Hz
F2R50 0.000 ppm
F2R51 188.48 Hz
F2R52 0.000 ppm
F2R53 188.48 Hz
F2R54 0.000 ppm
F2R55 188.48 Hz
F2R56 0.000 ppm
F2R57 188.48 Hz
F2R58 0.000 ppm
F2R59 188.48 Hz
F2R60 0.000 ppm
F2R61 188.48 Hz
F2R62 0.000 ppm
F2R63 188.48 Hz
F2R64 0.000 ppm
F2R65 188.48 Hz
F2R66 0.000 ppm
F2R67 188.48 Hz
F2R68 0.000 ppm
F2R69 188.48 Hz
F2R70 0.000 ppm
F2R71 188.48 Hz
F2R72 0.000 ppm
F2R73 188.48 Hz
F2R74 0.000 ppm
F2R75 188.48 Hz
F2R76 0.000 ppm
F2R77 188.48 Hz
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F2R85 188.48 Hz
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F2R87 188.48 Hz
F2R88 0.000 ppm
F2R89 188.48 Hz
F2R90 0.000 ppm
F2R91 188.48 Hz
F2R92 0.000 ppm
F2R93 188.48 Hz
F2R94 0.000 ppm
F2R95 188.48 Hz
F2R96 0.000 ppm
F2R97 188.48 Hz
F2R98 0.000 ppm
F2R99 188.48 Hz
F2R100 0.000 ppm



13



Current Data Parameters
NAME pa2-164
EXPNO 1
PROCNO 1

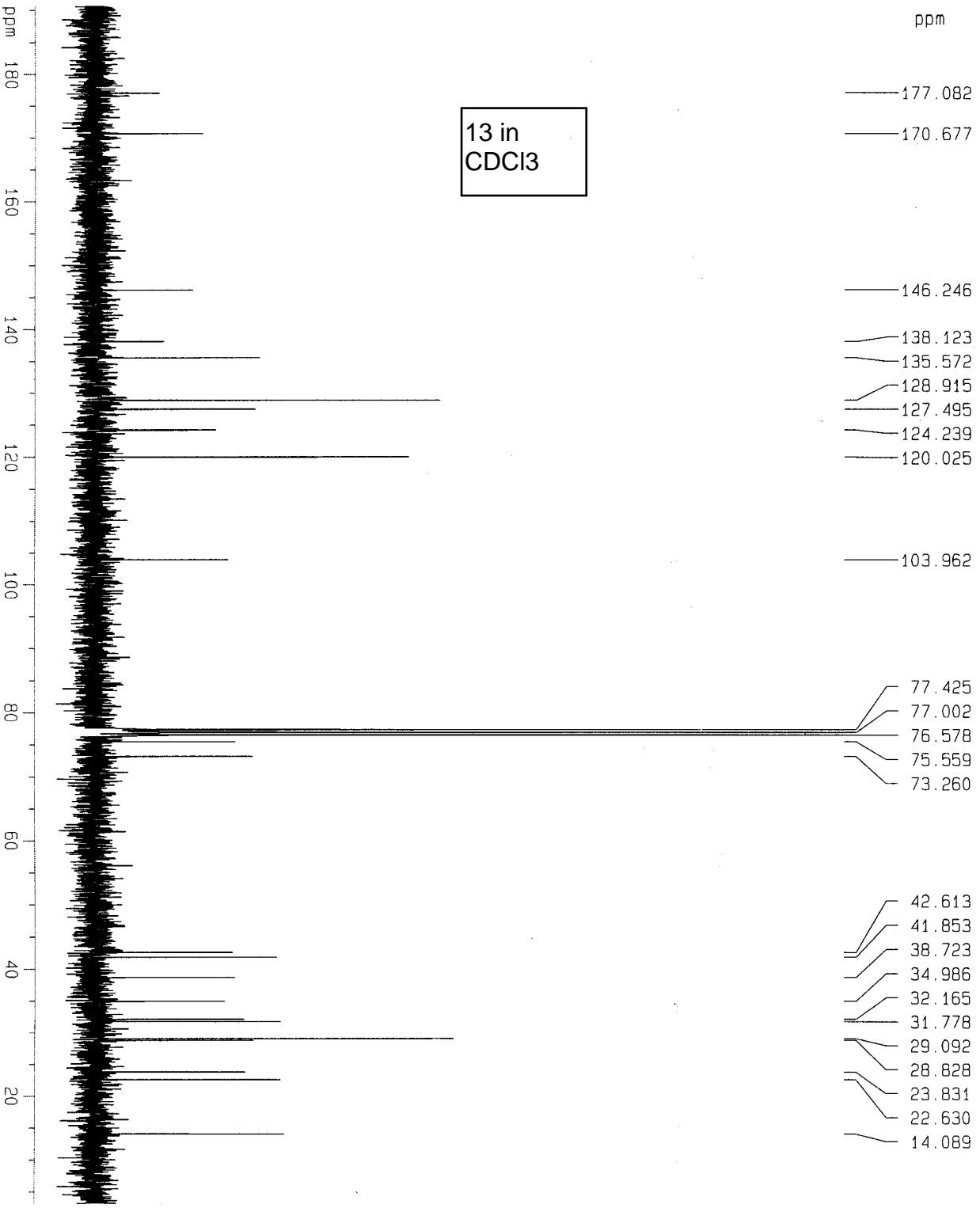
F2 - Acquisition Parameters
Date_ 20030429
Time 14.12
INSTRUM av300
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 30720
SOLVENT CDCl3
NS 16
DS 0
SWH 5081.301 Hz
FIDRES 0.165407 Hz
AQ 3.0228980 sec
RG 256
DM 98.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 300.1319477 MHz

F2 - Processing parameters
SI 65536
SF 300.1300001 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 11.062 ppm
F1 3320.00 Hz
F2P -0.545 ppm
F2 -163.72 Hz
PPMCM 0.58037 ppm/cm
HZCM 174.18587 Hz/cm

13C with proton decoupling



Current Data Parameters
NAME pa2-164
EXPNO 2
PROCNO 1

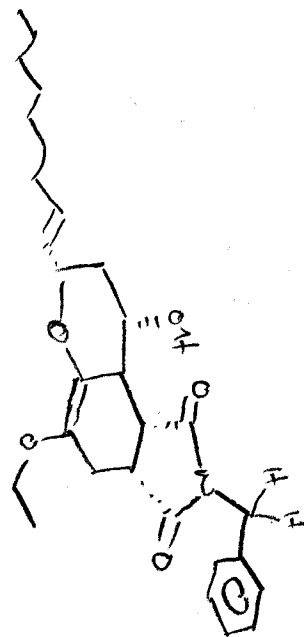
F2 - Acquisition Parameters
Date_ 20030429
Time 14.22
INSTRUM av300
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 388
DS 0
SWH 17985.611 Hz
FIDRES 0.54877 Hz
AQ 0.9110004 sec
RG 3251
DM 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

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

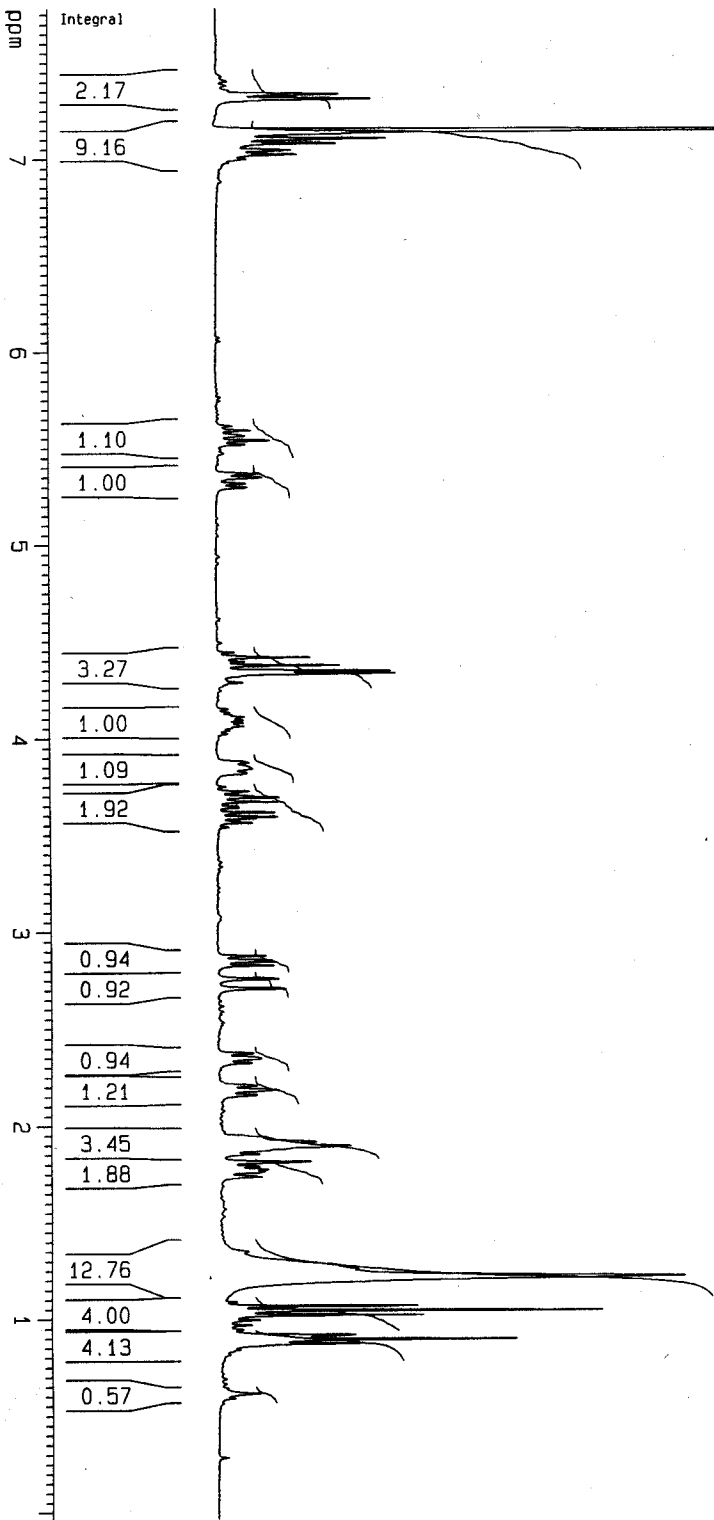
===== CHANNEL f2 =====
CPOPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -3.00 dB
PL12 13.48 dB
PL13 15.63 dB
SF02 300.1314860 MHz

F2 - Processing parameters
SI 65536
SF 75.4677486 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 6.00 cm
F1P 190.704 ppm
F1 14392.04 Hz
F2P 3.216 ppm
F2 242.69 Hz
PPMCM 9.37443 ppm/cm
HZCM 707.46722 Hz/cm



14 in C6D6



Current Data Parameters
NAME r1-110
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20031022
Time 12:56
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 228.1
DW 98.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

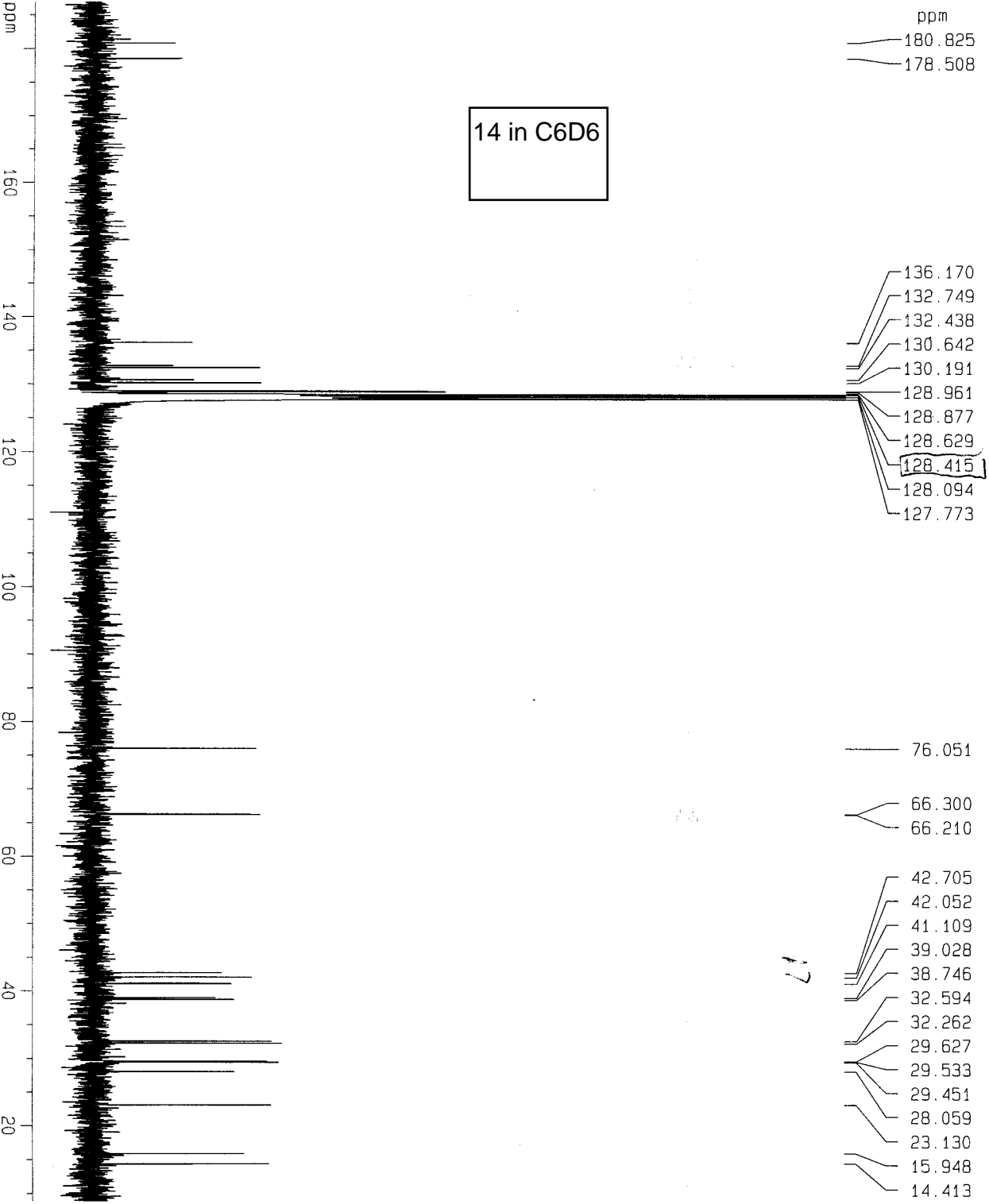
===== CHANNEL f1 =====
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 300.131947 MHz

F2 - Processing parameters
SI 65536
SF 300.130000 MHz
WDW 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 7.780 ppm
F1 2335.03 Hz
F2P -0.034 ppm
F2 -10.19 Hz
PPMCM 0.39070 ppm/cm
HZCM 117.26078 Hz/cm

13C with proton decoupling

14 in C6D6



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 CDCl3

DS 0
SWH 17985.611 Hz

FIDRES 0.54877 Hz
AQ 0.9110004 sec

RG 3649.1
DM 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

SFO1 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

SFO2 300.1314560 MHz

F2 - Processing parameters
SI 65536
SF 75.4677190 MHz

WDW EM
SSB 0
LB 1.00 Hz

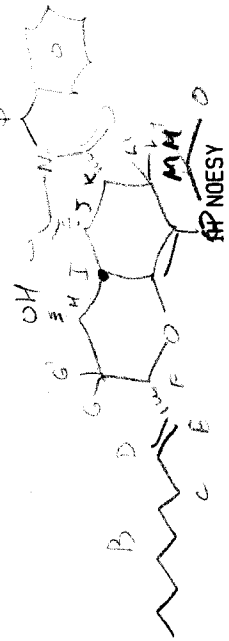
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 100.00 cm

F1P 186.823 ppm
F1 14059.08 Hz
F2P 8.831 ppm

F2 666.47 Hz
PPMCM 8.89957 ppm/cm

HZCM 671.63055 Hz/cm



Convert Raw Parameters
F1-1H
F2-1H

Acquisition Parameters
Date: 21.07
Time: 10.00
Pulse: 9.00
TD: 65536
SOLVENT: CDCl3
NS: 1024
DS: 4
SWH: 2048.20 Hz
FIDRES: 0.277767 Hz
AQ: 0.000000 sec
RG: 327.68
GB: 1.000000 sec
PC: 0.000000 sec
DLS: 0.000000 sec
DELTA: 0.0011200 sec
TE: 300.2 K
NUC1: 13C
NUC2: 1H
MAGNET: 0.0770000 sec
STENT: 1.28
T1: 0.0000000 sec

Channel: F1
NUC1: 13C
P1: 10.00 usec
PL1: 0.00 dB
SFO1: 300.131270 MHz

Channel: F2
SFO2: 100.625000 MHz
P2: 10.00 usec
PL2: 0.00 dB

Channel: F3
SFO3: 100.625000 MHz
P3: 10.00 usec
PL3: 0.00 dB

Channel: F4
SFO4: 100.625000 MHz
P4: 10.00 usec
PL4: 0.00 dB

Channel: F5
SFO5: 100.625000 MHz
P5: 10.00 usec
PL5: 0.00 dB

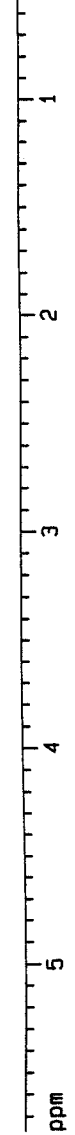
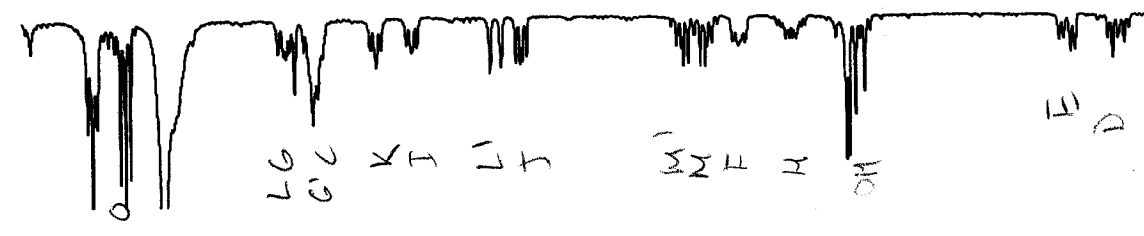
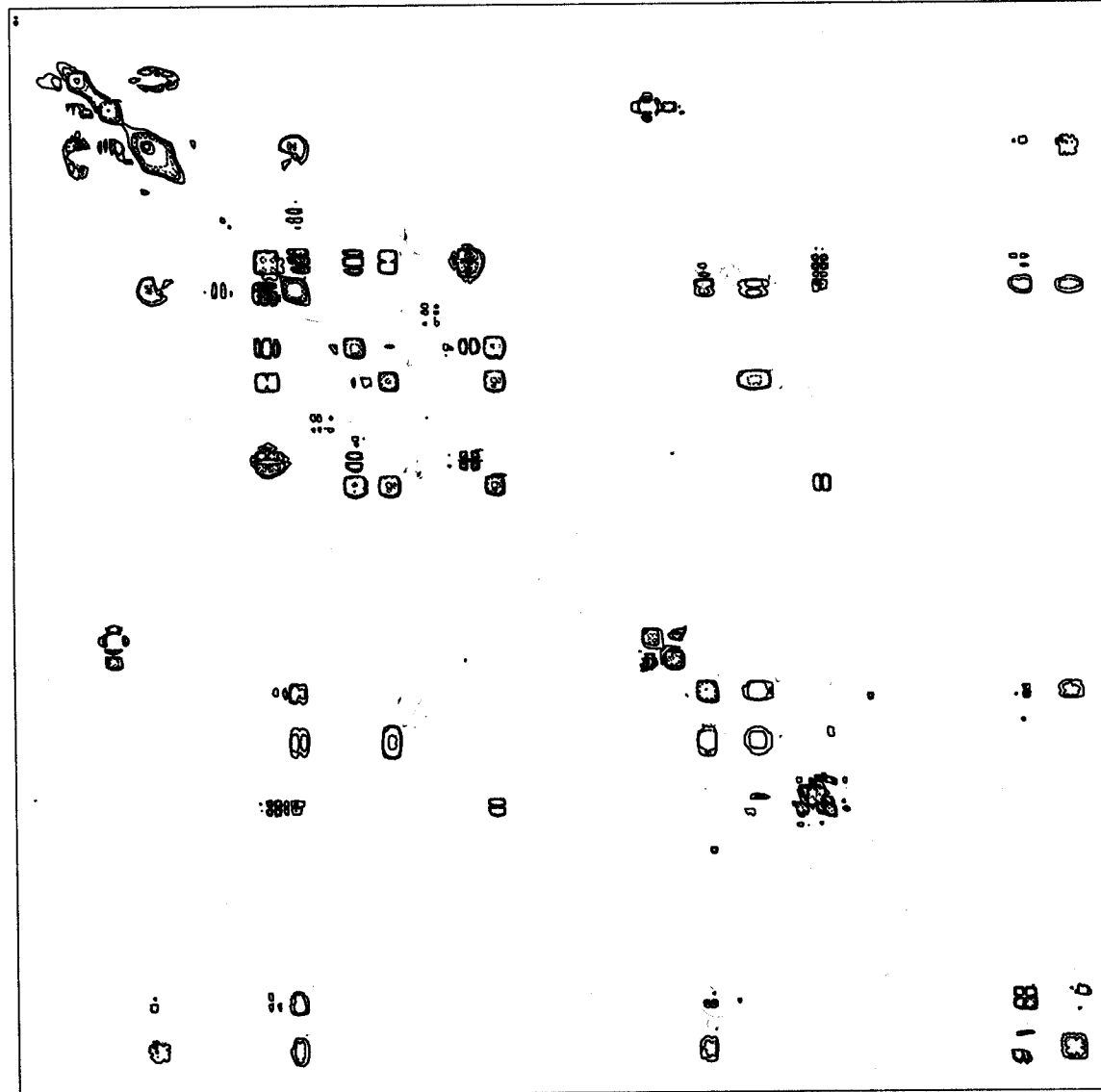
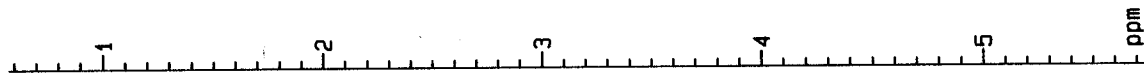
Channel: F6
SFO6: 100.625000 MHz
P6: 10.00 usec
PL6: 0.00 dB

Channel: F7
SFO7: 100.625000 MHz
P7: 10.00 usec
PL7: 0.00 dB

Channel: F8
SFO8: 100.625000 MHz
P8: 10.00 usec
PL8: 0.00 dB

Channel: F9
SFO9: 100.625000 MHz
P9: 10.00 usec
PL9: 0.00 dB

14



15 in C6D6



Current Data Parameters
NAME pac1a1sen
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040121
Time 11.44

INSTRUM av300
PROBHD 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
DM 98.400 usec

DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

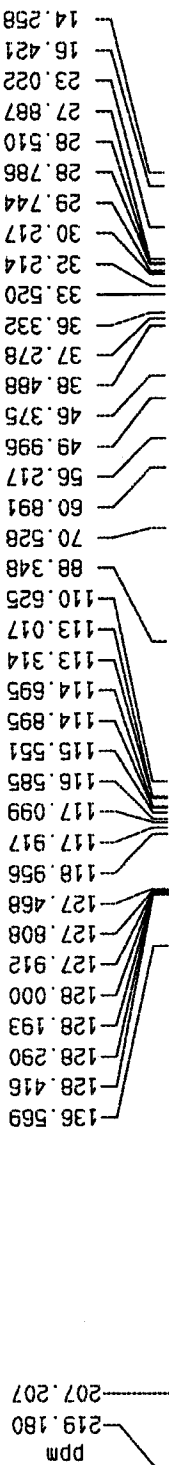
===== CHANNEL f1 =====
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 300.1319477 MHz

F2 - Processing parameters
SI 65536
SF 300.1299998 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 5.886 ppm
F1 1766.69 Hz
F2P 0.511 ppm
F2 153.46 Hz
PPMCM 0.26875 ppm/cm
HZCM 80.66121 Hz/cm

¹³C with proton decoupling

15 in C6D6



```

Current Data Parameters
NAME      pat_fla
EXPNO     5
PROCNO    1

F2 - Acquisition Parameters
Date_     20040202
Time      8.21
INSTRUM   AV500WB
PROBHD    5 mm BBI 1H-BB
PULPROG   zgpg30
TO        65536
SOLVENT   CDCl3
NS         43804
DS         0
SMH        30030.029 Hz
FIDRES     0.458222 Hz
AQ         1.0912244 sec
RG         3251
DM         16.650 usec
DE         6.00 usec
TE         300.0 K
D1         1.00000000 sec
d11        0.03000000 sec
d12        0.0002000 sec

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

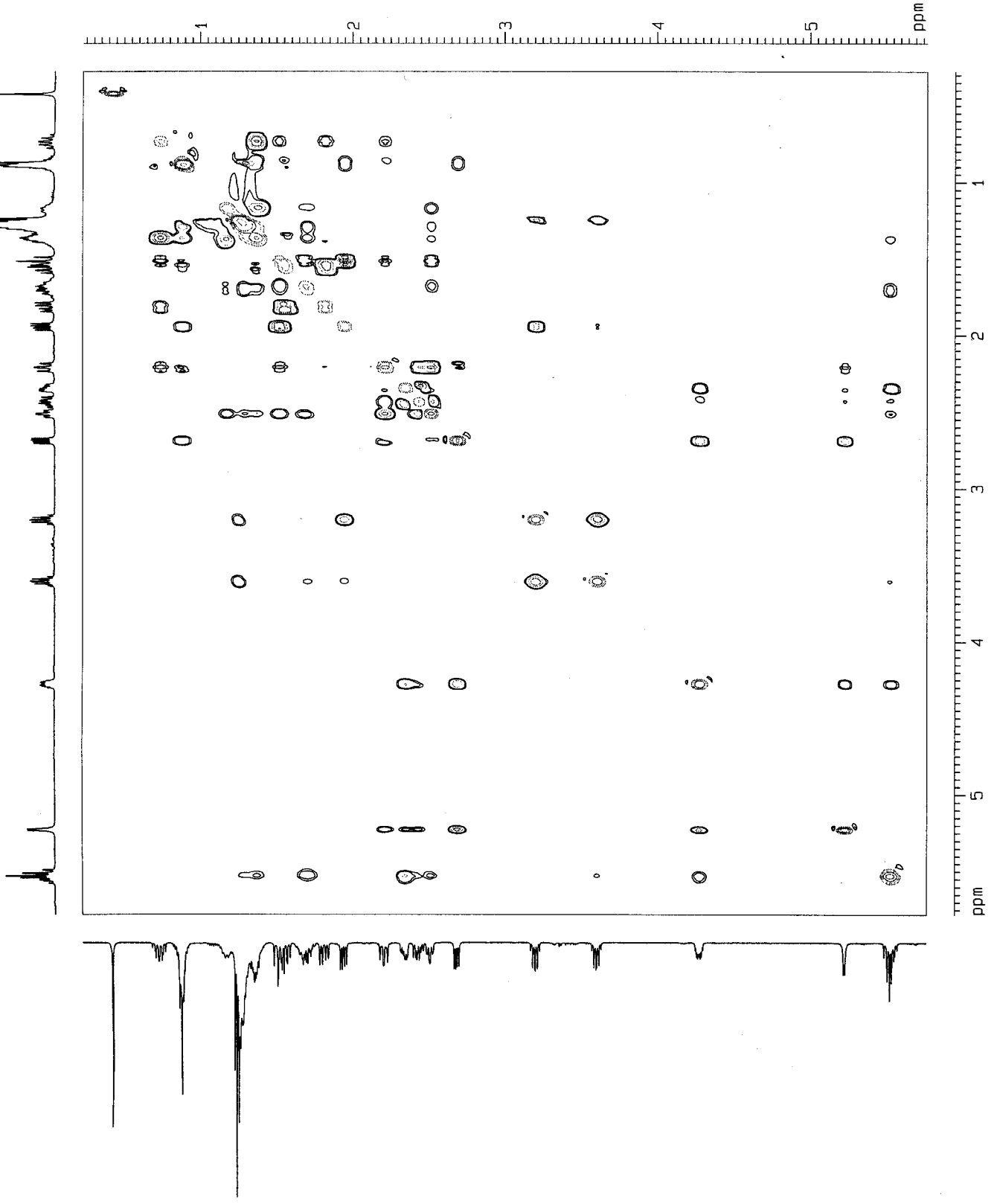
===== CHANNEL f2 =====
CPOPRG2    waltz16
NUC2        1H
PCPD2       80.00 usec
PL2         3.00 dB
PL12        20.84 dB
PL13        23.56 dB
SF02        500.1320005 MHz

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

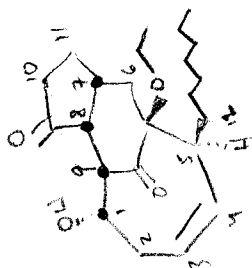
1D NMR plot parameters
CX          20.00 cm
CY          400.00 cm
FIP         219.264 ppm
F1          27574.16 Hz
F2          -19.528 ppm
PRNCH       11.93963 ppm/cm
HZCN        1501.50098 Hz/cm
    
```

NOESY

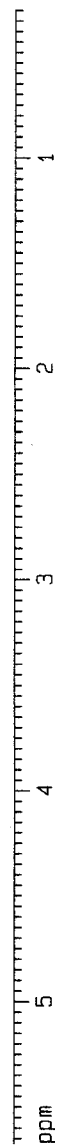
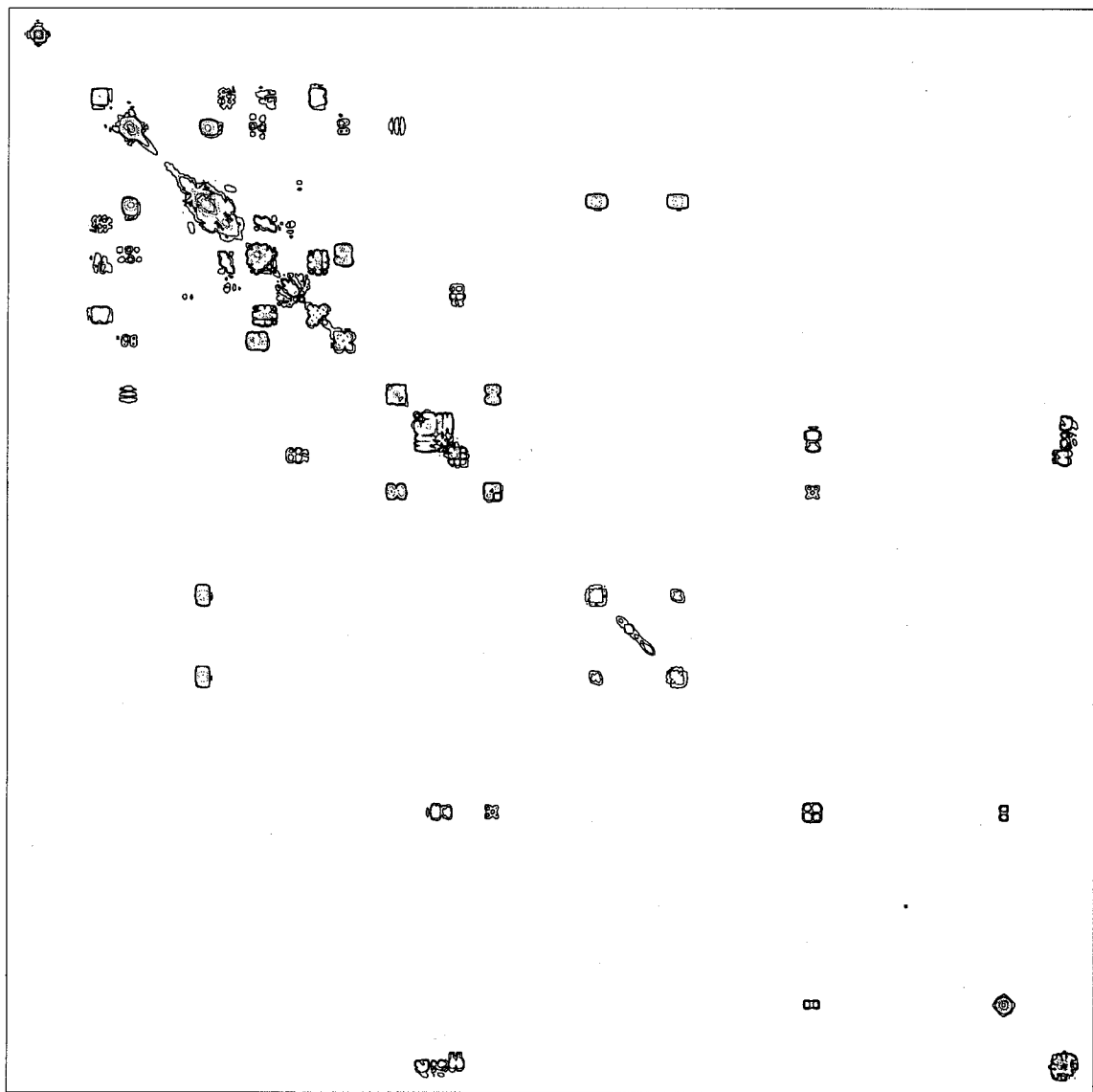
15



cosy 1

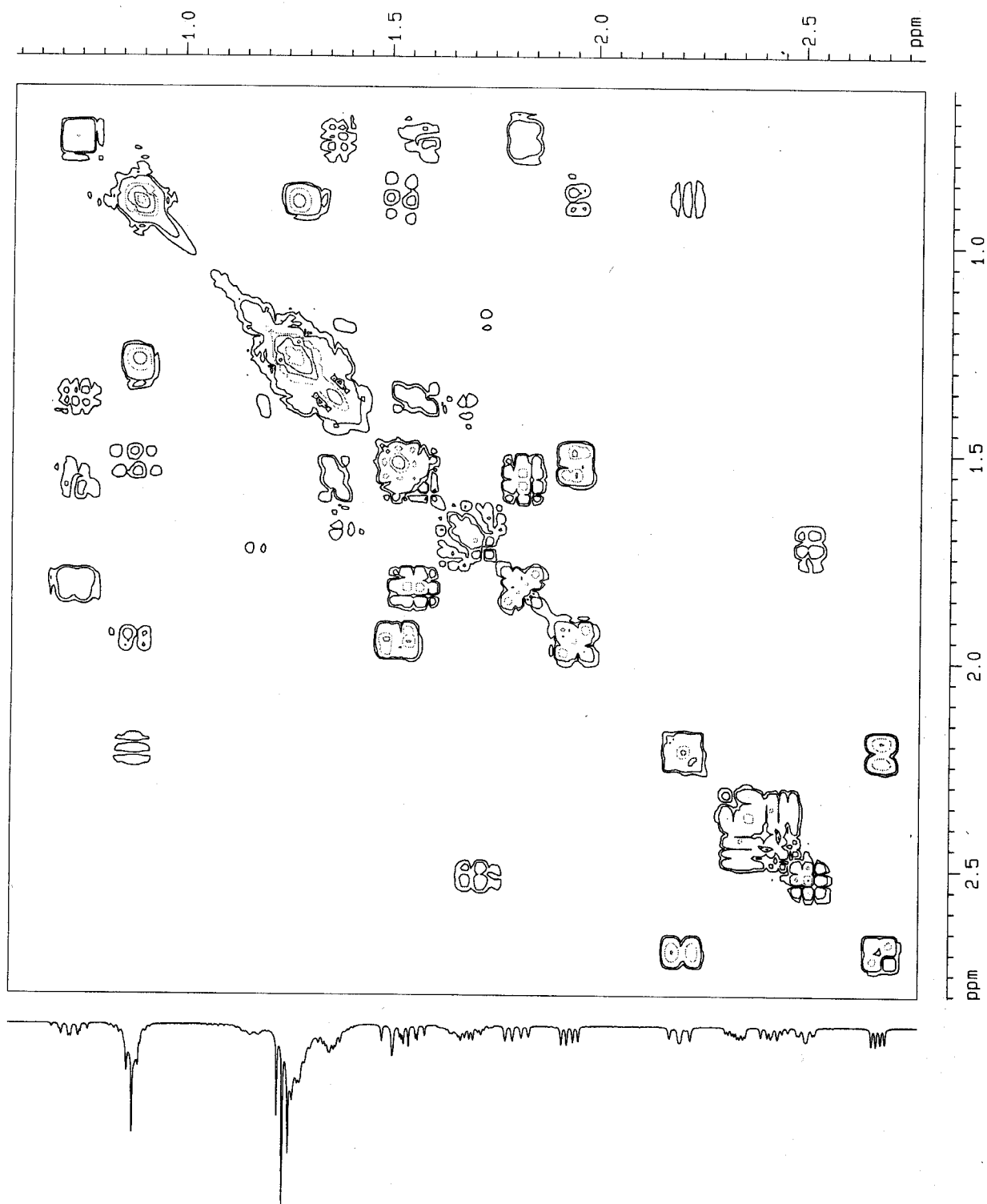


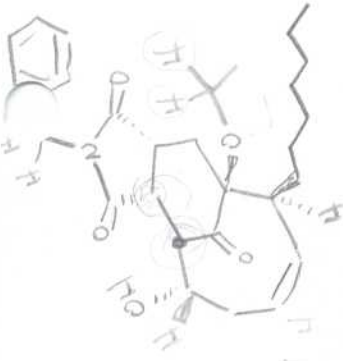
15



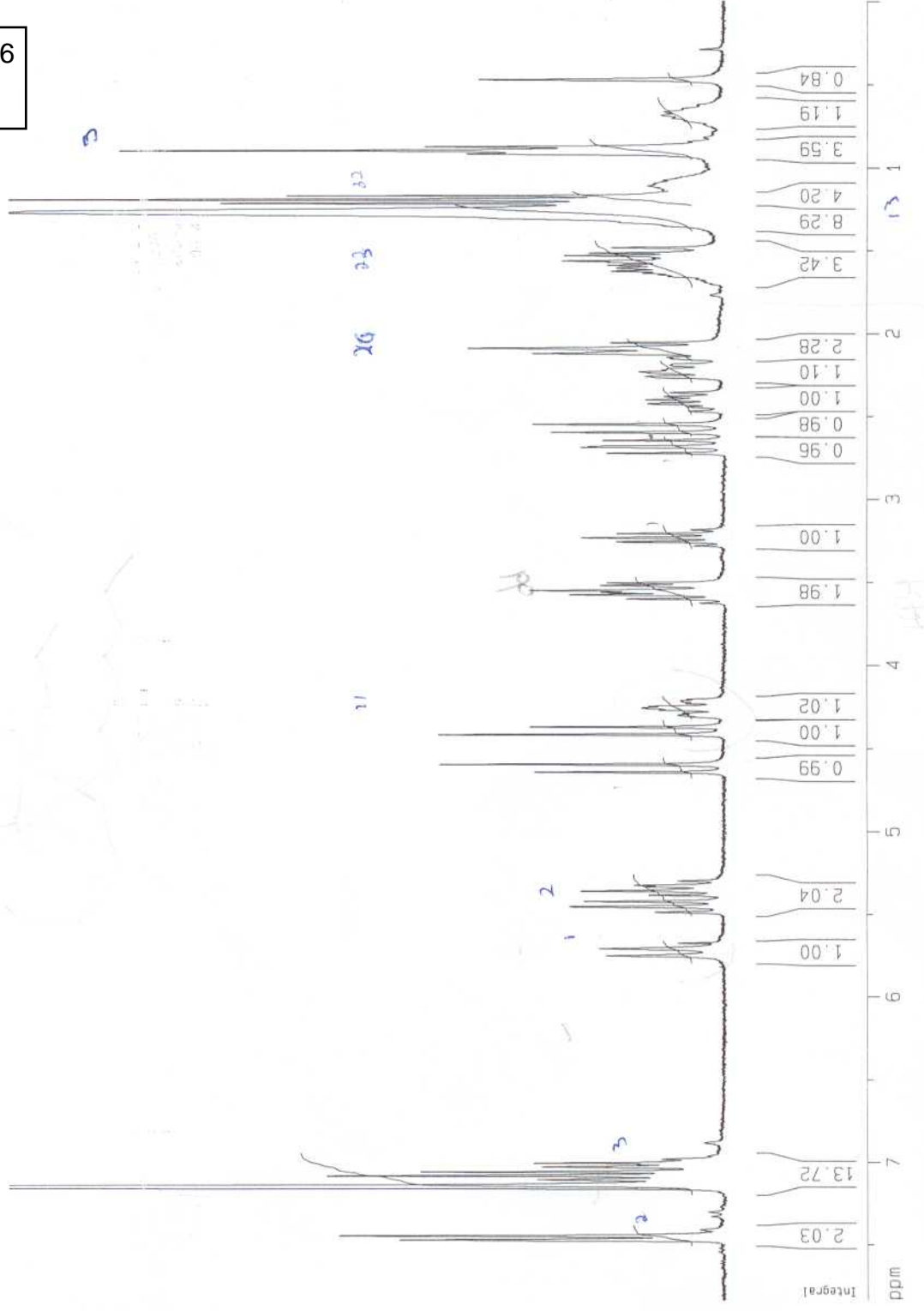
cosy 2

15



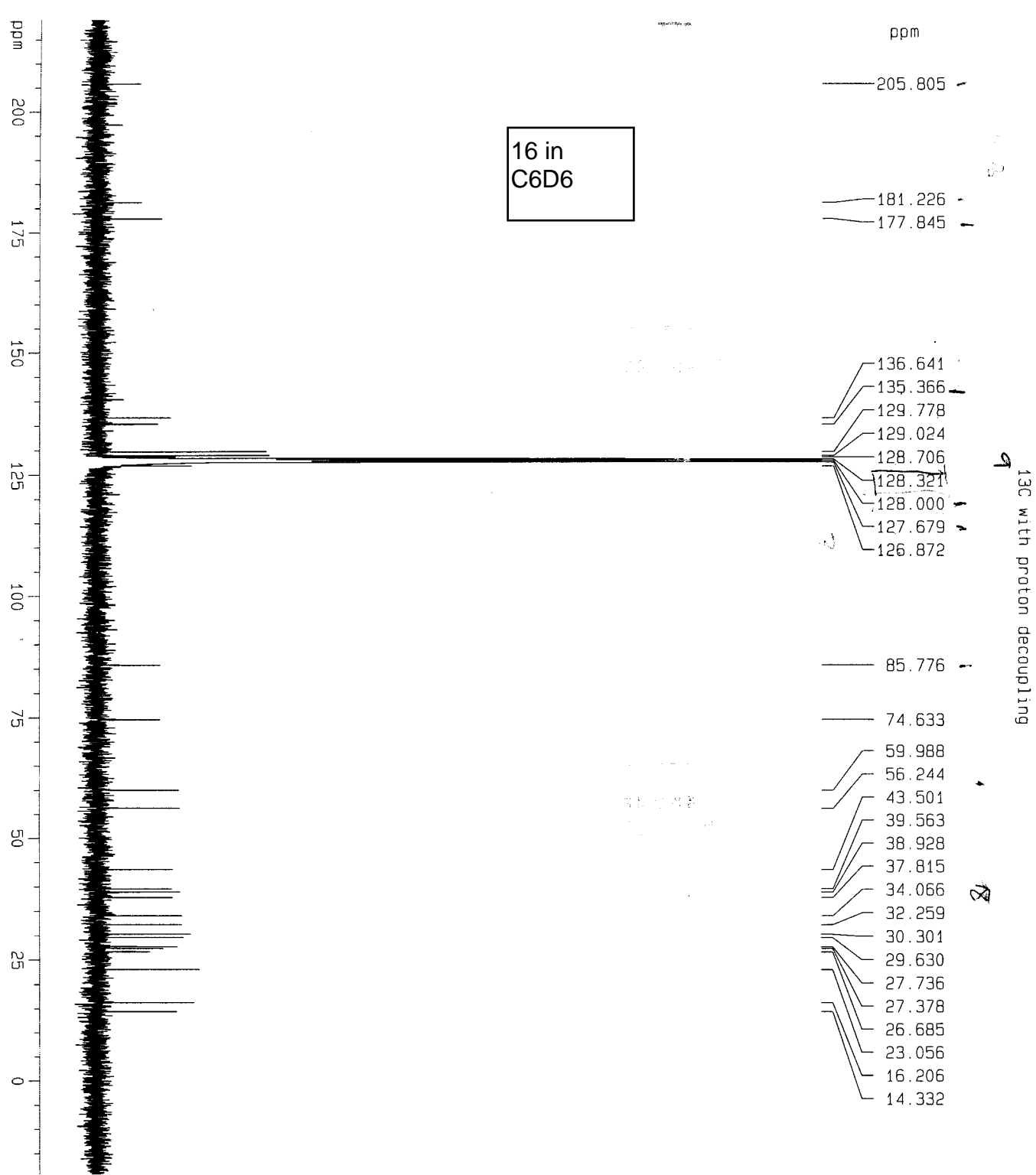


16 in C6D6



Current Data Parameters
NAME r1-106
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20031017
Time 13.15
INSTRUM av300
PROBHD 5 mm GNP 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 406.4
DW 98.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 300.1319477 MHz
F2 - Processing parameters
SI 65536
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 50.00 cm
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



Current Data Parameters

NAME	r1-106
EXPNO	3
PROCNO	1

F2 - Acquisition Parameters

Date_	20031017
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	0.54877 Hz
AQ	0.9110004 sec
RG	3649.1
DW	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
SFO1	75.4752653 MHz

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

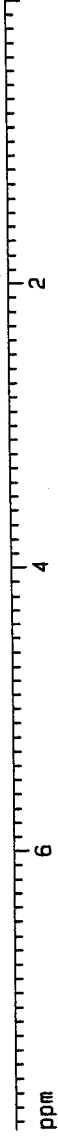
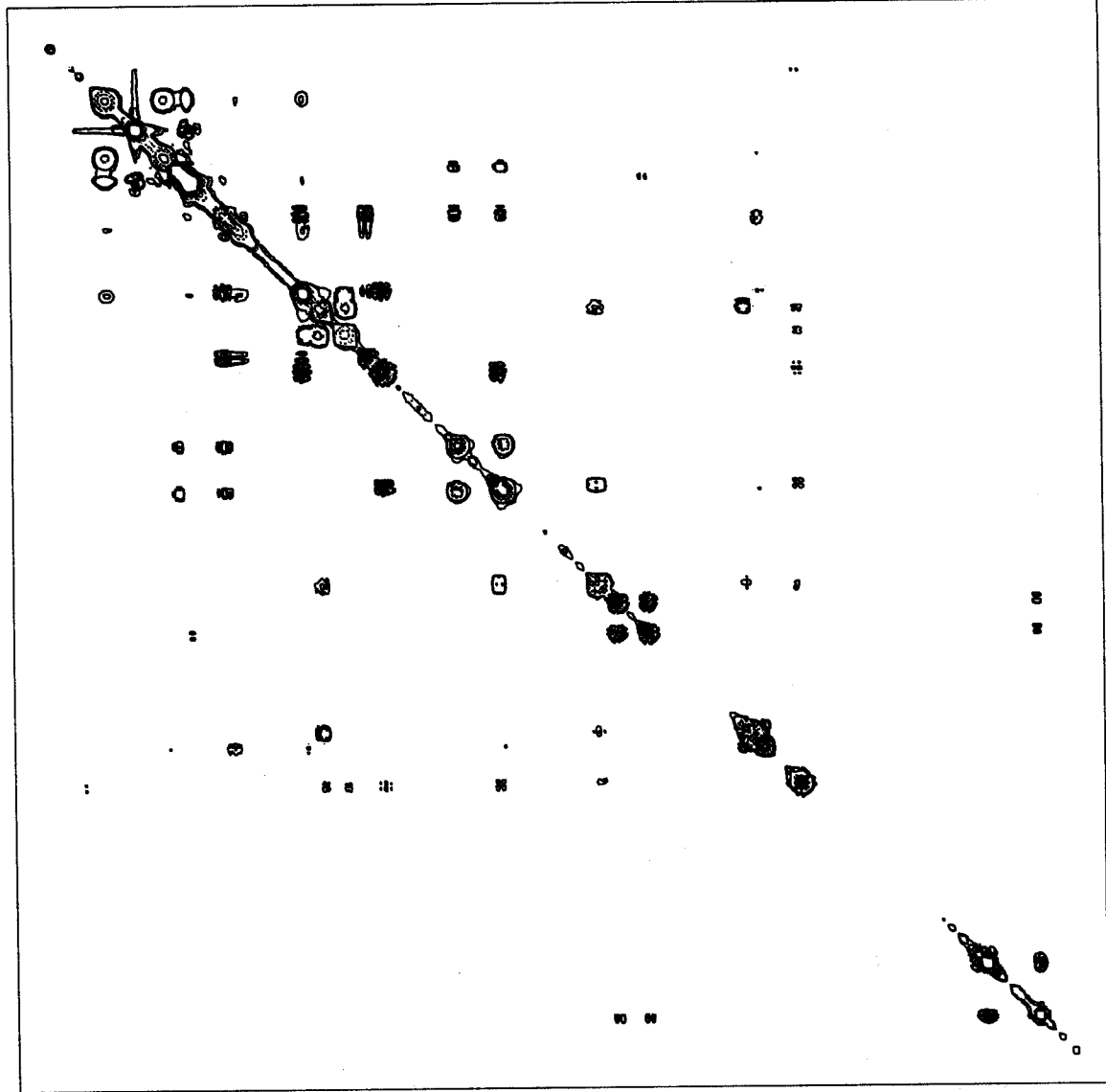
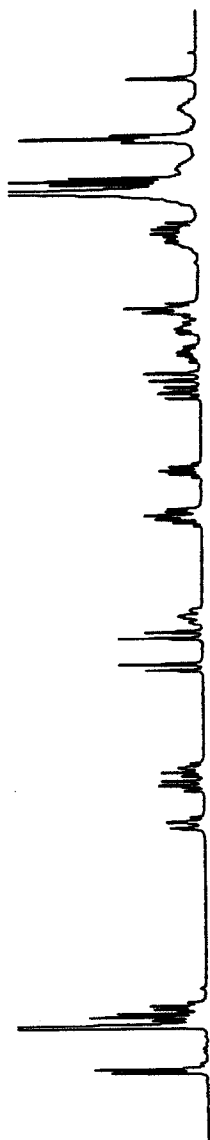
CPDPRG2	waltz16
NUC2	1H
PCPD2	70.00 usec
PL2	-3.00 dB
PL12	13.48 dB
PL13	15.63 dB
SFO2	300.1314860 MHz

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	219.062 ppm
F1	16532.09 Hz
F2P	-19.260 ppm
F2	-1453.52 Hz
PPMCH	11.91609 ppm/cm
HZCM	899.28058 Hz/cm



Current Data Parameters
Date_ 20031021
Time 1.40
Pulse Program 1
F1-100
F2-100
F3-100
F4-100
F5-100
F6-100
F7-100
F8-100
F9-100
F10-100
F11-100
F12-100
F13-100
F14-100
F15-100
F16-100
F17-100
F18-100
F19-100
F20-100
F21-100
F22-100
F23-100
F24-100
F25-100
F26-100
F27-100
F28-100
F29-100
F30-100
F31-100
F32-100
F33-100
F34-100
F35-100
F36-100
F37-100
F38-100
F39-100
F40-100
F41-100
F42-100
F43-100
F44-100
F45-100
F46-100
F47-100
F48-100
F49-100
F50-100
F51-100
F52-100
F53-100
F54-100
F55-100
F56-100
F57-100
F58-100
F59-100
F60-100
F61-100
F62-100
F63-100
F64-100
F65-100
F66-100
F67-100
F68-100
F69-100
F70-100
F71-100
F72-100
F73-100
F74-100
F75-100
F76-100
F77-100
F78-100
F79-100
F80-100
F81-100
F82-100
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F89-100
F90-100
F91-100
F92-100
F93-100
F94-100
F95-100
F96-100
F97-100
F98-100
F99-100
F100-100

