Concise synthesis of a pentasaccharide of the anti-leishmanial triterpenoid saponin isolated from *Maesa balansae*

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SUPPORTING INFORMATION

Contents

Experimental

General. All reagents and solvents were dried prior to use according to standard methods.[i] Commercial reagents were used without further purification unless otherwise stated. Analytical TLC was performed on Silica Gel 60-F_{254} with detection by fluorescence and/or by charring following immersion in a 10% ethanolic solution of sulfuric acid. An orcinol dip, prepared by the careful addition of concentrated sulfuric acid (20 mL) to an ice-cold solution of 3,5-dihydroxytoluene (360 mg) in EtOH (150 mL) and H_2O (10 mL), was used to detect deprotected compounds by charring. Flash chromatography was performed with Silica Gel 60. Optical rotations were measured at the sodium D-line at ambient temperature. ^{1}H NMR and ^{13}C NMR spectra were recorded on a spectrometer at 300 and 75 MHz.

***p*-Methoxyphenyl 6-*O*-(*tert*-butyldiphenylsilyl)-β-D-galactopyranoside (3).** To a solution of compound **2** (2.0 g, 7.0 mmol) in dry pyridine (30 mL) was added TBDPS-Cl (2.3 mL, 9.0 mmol) and the solution was stirred for 12 hours at room temperature. Solvents were evaporated *in vacuo* and the residual syrup was purified by flash chromatography using *n*-hexane-EtOAc (2:1) as eluent to afford pure compound **3** (3.4 g, 93%) as colourless thick syrup. $[\alpha]_D^{25}$ +102 (*c* 1.0, $CHCl_3$). ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.56-7.16 (m, 10H, ArH), 6.90, 6.49 (2d, 4H, *J* 9.0 Hz, $C_6H_4OCH_3$), 4.56 (d, 1H, *J* 7.5 Hz, H-1), 3.93 (m, 1H, H-2), 3.81 (m, 2H, H-6a, H-6b), 3.70 (dd, 1H, *J* 3.9 Hz, 9.3 Hz, H-3), 3.56 (s, 3H, $C_6H_4OCH_3$), 3.54 (m, 1H, H-4), 3.41 (m, 1H, H-5), 0.94 (s, 9H, $SiC(CH_3)_3$). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 155.27, 151.46, 135.6(2), 133.2, 129.7, 127.8(2), 118.9, 114.4 (ArC), 102.8 (C-1), 75.6, 73.7, 71.1, 69.3, 63.6 (C-6), 55.3 ($C_6H_4OCH_3$), 26.9 (3C, $SiC(CH_3)_3$), 19.3 ($SiC(CH_3)_3$). HRMS Calcd. for $C_{29}H_{36}O_7SiNa$ $(M+Na)^+$: 547.2128; found 547.2126.

***p*-Methoxyphenyl 3,4-*O*-isopropylidene-6-*O*-(*tert*-butyl diphenylsilyl)-β-D-galactopyranoside (4).** To a slurry of compound **3** (3.0 g, 5.7 mmol) in dry acetone (30 mL) and 2,2-DMP (1.0 mL, 8.6 mmol) was added (S)-10-camphorsulfonic acid (50 mg) and the mixture was stirred at room temperature for 2 hours when TLC (*n*-hexane-EtOAc; 4:1) showed complete conversion of the starting material. The solution was neutralized with Et_3N and the solvents were evaporated *in vacuo*. The residue was purified by flash chromatography using *n*-hexane-EtOAc (5:1) to afford pure compound **4** (3.1 g, 97%) as white foam. $[\alpha]_D^{25}$ +122 (*c* 1.1, $CHCl_3$). ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.59-7.19 (m, 10H, ArH), 6.91, 6.67 (2d, 4H, *J* 9.0 Hz, $C_6H_4OCH_3$), 4.53 (d, 1H, *J* 8.1 Hz, H-1), 4.15 (bd, 1H, *J* 5.4 Hz, H-4), 4.04 (m, 1H, H-3), 3.87 (m, 3H, H-5, H-6^a, H-6b), 3.71 (dd, 1H, *J* 8.1 Hz, 9.3 Hz, H-2), 3.67 (s, 3H, $C_6H_4OCH_3$), 2.62 (bs, 1H, O*H*), 1.43, 1.28 (2s, 6H, 2×isopropylidene CH_3), 1.01 (s, 9H, $SiC(CH_3)_3$). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 155.5, 151.3, 135.7(5), 133.3, 129.8(2), 127.7(4), 118.7(2), 114.5(2) (ArC), 110.2 ($C(CH_3)_2$), 102.0 (C-1), 79.0, 74.2, 73.5, 73.4, 63.0 (C-6), 55.4 ($C_6H_4OCH_3$), 28.3, 26.5 (2×isopropylidene CH_3), 27.0(3) ($SiC(CH_3)_3$), 19.4 ($SiC(CH_3)_3$). HRMS Calcd. for $C_{32}H_{40}O_7SiNa$ $(M+Na)^+$: 587.2441; found 587.2439.

***p*-Tolyl 2,3-*O*-isopropylidene-1-thio-α-L-rhamnopyranoside (5).** To a solution of known *p*-tolyl 1-thio-α-L-rhamnopyranoside (4.0 g, 14.8 mmol) in dry acetone (20 mL) was added 2,2-DMP (2 mL, 19.2 mmol) followed by 10-camphorsulfonic acid (50 mg) and the solution was stirred at room temperature for 1 hour when the TLC showed complete conversion of the starting material to a faster moving component. The solution was neutralized with Et_3N and the solvents were evaporated *in vacuo*. The residue was purified by flash chromatography using *n*-hexane-EtOAc (3:1) to afford pure compound **5** (4.2 g, 92%) as colourless thick syrup. $[\alpha]_D^{25}$ +81 (*c* 1.1, $CHCl_3$). ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.28, 7.02 (2d, 4H, *J* 8.1 Hz, $SC_6H_4CH_3$), 5.59 (s, 1H, H-1), 4.24 (d, 1H, *J* 5.7 Hz, H-2),

4.06-3.95 (m, 2H, H-3, H-5), 3.58 (bs, 1H, O*H*), 3.34 (t, 1H, *J* 9.1 Hz), 2.27 (s, 3H, $SC_6H_4CH_3$), 1.47, 1.30 (2s, 6H, 2×isopropylidene CH_3), 1.18 (d, 3H, *J* 6.0 Hz, C-CH_3). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 137.1, 132.3(2), 129.6, 129.5(2) (ArC), 109.2 ($C(CH_3)_2$), 83.7 (C-1), 78.4, 76.4, 74.7, 66.6, 28.0, 26.2 (2×isopropylidene CH_3), 21.0 ($SC_6H_4CH_3$), 17.0 (C-CH_3).

***p*-Tolyl 4-*O*-benzyl-2,3-*O*-isopropylidene-1-thio-α-L-rhamnopyranoside (6).** To a solution of compound **5** (3.0 g, 9.7 mmol) in dry DMF (25 mL) was added NaH (930 mg, 19.4 mmol, 50% dispersion in mineral oil) followed by BnBr (1.7 mL, 14.6 mmol) at 0 °C and the mixture was stirred at room temperature for 2 hours. The excess NaH was carefully neutralized by drop wise addition of MeOH. The resulting mixture was concentrated *in vacuo*. The residue was dissolved in diethyl ether (40 mL) and washed successively with H_2O (2×50 mL). Organic layer was collected, dried (Na_2SO_4) and evaporated to syrup. The crude compound was purified by flash chromatography using *n*-hexane-EtOAc (6:1) to afford pure compound **6** (3.4 g, 89%) as colourless oil. $[\alpha]_D^{25}$ +93 (*c* 1.1, $CHCl_3$). ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.10-6.83 (m, 9H, ArH), 5.36 (s, 1H, H-1), 4.65, 4.36 (2d, AB system, 2H, *J* 11.7 Hz, CH_2Ph), 4.04 (m, 2H, H-2, H-4), 3.86 (m, 1H, H-5), 3.00 (dd, 1H, *J* 6.6 Hz, 9.6 Hz, H-3), 2.10 (s, 3H, $SC_6H_4CH_3$), 1.25, 1.12 (2s, 6H, 2×isopropylidene CH_3), 0.98 (d, 3H, *J* 6.3 Hz, C-CH_3). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 138.5, 137.4, 132.5(2), 130.2, 129.8(2), 128.2(2), 128.0(2), 127.6 (ArC), 109.3 ($C(CH_3)_2$), 84.2 (C-1), 81.5, 78.6, 76.8, 72.8, 66.1, 28.2, 26.7 (2×isopropylidene CH_3), 21.3 ($SC_6H_4CH_3$), 17.9 (C-CH_3). HRMS Calcd. for $C_{23}H_{28}O_4SNa$ $(M+Na)^+$: 423.1606; found 423.1603.

***p*-Tolyl 4-*O*-benzyl-1-thio-α-L-rhamnopyranoside (7).** A solution of compound **6** (3.0 g, 7.5 mmol) in AcOH-H_2O (9:1, 30 mL) was stirred at 85 °C for 2 hours when TLC showed complete

conversion of the starting material to a slower moving spot. The solvents were evaporated *in vacuo* and the residue was purified by flash chromatography using *n*-hexane-EtOAc (3:1) to afford pure compound **7** (2.5 g, 92%) as white foam. $[\alpha]_D^{25}$ +101 (*c* 1.1, $CHCl_3$). ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.35-7.06 (m, 9H, ArH), 5.42 (s, 1H, H-1), 4.83, 4.67 (2d, AB system, 2H, *J* 11.1 Hz, C*H*$_2$Ph), 4.23 (m, 2H, H-2, H-5), 3.94 (dd, 1H, *J* 2.4 Hz, 8.4 Hz, H-3), 3.45 (t, 1H, *J* 8.4 Hz, H-4), 2.34 (s, 3H, SC_6H_4C*H*$_3$), 1.29 (d, 3H, *J* 6.3 Hz, C-C*H*$_3$). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 138.4, 137.0, 131.8(2), 130.7, 129.7(2), 128.4(2), 127.8(2), 127.7 (ArC), 87.9 (C-1), 81.8, 74.9, 72.7, 72.0, 68.5, 21.2 (SC_6H_4*C*H_3), 18.0 (C-*C*H_3). HRMS Calcd. for $C_{20}H_{24}O_4SNa$ $(M+Na)^+$: 383.1293; found 383.1291.

***p*-Tolyl 3,4-di-*O*-benzyl-1-thio-α-L-rhamnopyranoside (8).** A mixture of compound **7** (2.3 g, 6.4 mmol) and Bu_2SnO (1.7 g, 7.0 mmol) in dry toluene (50 mL) was refluxed for 12 hours with a Dean-Stark apparatus for azeotropic removal of water. The volume of the mixture was reduced *in vacuo* to 15 mL and Bu_4NI (2.4 g, 6.4 mmol) was added followed by BnBr (990 μL, 8.3 mmol). The resulting mixture was stirred at room temperature for 12 hours. After concentrating the mixture, the crude product was dissolved in CH_2Cl_2 (40 mL) and washed successively with H_2O (2×50 mL). The organic layer was separated, dried (Na_aSO_4) and evaporated. The crude product thus obtained was purified by flash chromatography using *n*-hexane-EtOAc (5:1) to give pure compound **8** (2.6 g, 89%). $[\alpha]_D^{25}$ +95 (*c* 1.1, $CHCl_3$). ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.29-7.02 (m, 14H, ArH), 5.38 (s, 1H, H-1), 4.83, 4.59 (2d, AB system, 2H, *J* 11.1 Hz, C*H*$_2$Ph), 4.65 (s, 2H, C*H*$_2$Ph), 4.14 (m, 2H, H-2, H-5), 3.77 (dd, 1H, *J* 3.3 Hz, 9.0 Hz, H-3), 3.47 (t, 1H, *J* 9.0 Hz, H-4), 2.72 (bs, 1H, O*H*), 2.29 (s, 3H, SC_6H_4C*H*$_3$), 1.26 (d, 3H, *J* 6.3 Hz, C-C*H*$_3$). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 138.5, 137.7, 137.0, 131.9(2), 130.6, 129.5(2), 128.5(2), 128.2(2), 127.9(3), 127.8(2), 127.5 (ArC), 87.4 (C-1), 80.2(2),

75.2, 72.1, 70.0, 68.7, 21.2 ($SC_6H_4CH_3$), 17.9 (C-CH_3). HRMS Calcd. for $C_{27}H_{30}O_4SNa$ $(M+Na)^+$: 473.1762; found 473.1760.

***p*-Tolyl 2-*O*-acetyl-3,4-di-*O*-benzyl-1-thio-α-L-rhamnopyranoside (9).** To a solution of compound **8** (2.5 g, 5.5 mmol) in dry pyridine (15 mL) was added Ac_2O (10 mL) and the solution was stirred at room temperature for 2 hours. The solvents were evaporated *in vacuo* and co-evaporated with toluene to remove traces of pyridine. The residue was purified by flash chromatography using *n*-hexane-EtOAc (6:1) to afford pure compound **9** (2.7 g, 97%) as colourless thick syrup. $[\alpha]_D^{25}$ +103 (*c* 1.1, $CHCl_3$). ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.31-7.04 (m, 14H, ArH), 5.55 (bs, 1H, H-2), 5.28 (s, 1H, H-1), 4.90, 4.67, 4.59, 4.50 (4d, AB system, 4H, *J* 11.1 Hz, 2×CH_2Ph), 4.18 (m, 1H, H-5), 3.85 (dd, 1H, *J* 3.3 Hz, 9.3 Hz, H-3), 3.43 (t, 1H, *J* 9.3 Hz, H-4), 2.30 (s, 3H, $SC_6H_4CH_3$), 2.11 (s, 3H, $COCH_3$), 1.30 (d, 3H, *J* 6.0 Hz, C-CH_3). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 169.5 ($COCH_3$), 138.6, 137.7, 137.4, 132.3(2), 130.4, 129.8(2), 128.4(2), 128.3, 128.2(3), 127.8(3), 127.5 (ArC), 86.4 (C-1), 80.1, 78.4, 75.3, 71.8, 70.5, 69.0, 21.2 ($COCH_3$), 20.9 ($SC_6H_4CH_3$), 17.9 (C-CH_3). HRMS Calcd. for $C_{29}H_{32}O_5SNa$ $(M+Na)^+$: 515.1868; found 515.1865.

***p*-Methoxyphenyl 2-*O*-acetyl-3,4-di-*O*-benzyl-α-L-rhamnopyranosyl-(1→2)-3,4-*O*-isopropylidene-6-*O*-(*tert*-butyl diphenylsilyl)-β-D-galactopyranoside (10).** A mixture of compound **4** (1.5 g, 2.7 mmol), compound **9** (1.7 g, 3.5 mmol) and MS 4Å (2 g) in dry CH_2Cl_2 (30 mL) was stirred under nitrogen for 1 hour. NIS (945 mg, 4.2 mmol) was added and the mixture was cooled to 10 °C when H_2SO_4-silica (50 mg) was added. The mixture was stirred for 45 minutes when TLC showed complete consumption of the acceptor **4**. The mixture was filtered through a pad of Celite and the filtrate was washed successively with $Na_2S_2O_3$ (2×30 mL), $NaHCO_3$ (2×30 mL) and

brine (30 mL). The organic layer was separated, dried (Na_2SO_4) and evaporated *in vacuo*. The residue was purified by flash chromatography using *n*-hexane-EtOAc (3:1) to afford pure disaccharide **10** (2.2 g, 87%) as white foam. $[\alpha]_D^{25}$ +78 (*c* 1.0, $CHCl_3$). ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.72-7.22 (m, 20H, ArH), 7.00, 6.69 (2d, 4H, *J* 9.0 Hz, $C_6H_4OCH_3$), 4.46 (dd, 1H, *J* 1.8 Hz, 3.3 Hz, H-2′), 5.28 (d, 1H, *J* 1.8 Hz, H-1′), 4.92, 4.68, 4.63, 4.46 (4d, AB system, 4H, 2×CH_2Ph), 4.68 (d, 1H, *J* 6.0 Hz, H-1), 4.27 (dd, 1H, *J* 6.0 Hz, 9.3 Hz, H-2), 4.23 (m, 2H, H-6a, H-6b), 4.01-3.92 (m, 5H, H-3, H-3′, H-4, H-5, H-5′), 3.72 (s, 3H, $C_6H_4OCH_3$), 3.46 (t, 1H, *J* 9.6 Hz, H-4′), 2.15 (s, 3H, $COCH_3$), 1.54, 1.32 (2s, 6H, 2×isopropylidene CH_3), 1.34 (d, 3H, *J* 6.3 Hz, C-CH_3), 1.08 (s, 9H, $SiC(CH_3)_3$). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 170.3 ($COCH_3$), 155.2, 151.8, 138.9, 138.1, 135.6(2), 133.2, 129.7(2), 128.3(3), 128.2(4), 128.0(2), 127.9, 127.7(3), 127.6, 127.5(2), 127.3, 118.3(2), 114.5(2) (ArC), 110.5 ($C(CH_3)_2$), 100.7 (C-1), 96.1 (C-1′), 80.0, 79.6, 77.9, 74.8, 73.8, 73.4, 71.6, 68.7, 67.8, 62.9 (C-6), 55.6 ($C_6H_4OCH_3$), 27.9, 26.3 (2×isopropylidene CH_3), 26.8(3) ($SiC(CH_3)_3$), 21.1 ($COCH_3$), 19.2 ($SiC(CH_3)_3$), 18.0 (C-CH_3). HRMS Calcd. for $C_{54}H_{64}O_{12}SiNa$ $(M+Na)^+$: 955.4065; found 955.4062.

***p*-Methoxyphenyl 3,4-di-*O*-benzyl-α-L-rhamnopyranosyl-(1→2)-3,4-*O*-isopropylidene-6-*O*-(*tert*-butyl diphenylsilyl)-β-D-galactopyranoside (11).** To a solution of compound **10** (2 g, 2.1 mmol) in dry MeOH (25 mL) was added NaOMe in MeOH (0.5 M, 2 mL) and the solution was stirred at room temperature for 2 hours. The solution was neutralized with DOWEX 50W H^+ resin and filtered through a cotton plug. The filtrate was evaporated and purified by flash chromatography to afford the disaccharide acceptor **11** (1.9 g, 98%) as white foam. $[\alpha]_D^{25}$ +83 (*c* 1.2, $CHCl_3$). ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.72-7.21 (m, 20H, ArH), 7.01, 6.71 (2d, 4H, *J* 9.0 Hz, $C_6H_4OCH_3$), 5.37 (d, 1H, *J* 1.2 Hz, H-1′), 4.88, 4.66 (2d, AB system, *J* 11.4 Hz, CH_2Ph), 4.69 (d, 1H, *J* 8.1 Hz, H-

1), 4.60 (s, 2H, C*H*$_2$Ph), 4.26-4.18 (m, 3H, H-2, H-3, H-6a), 4.08 (m, 1H, H-5′), 4.01 (m, 4H, H-2′, H-4, H-5, H-6b), 3.83 (dd, 1H, *J* 3.3 Hz, 9.0 Hz, H-3′), 3.72 (s, 3H, C$_6$H$_4$OC*H*$_3$), 3.48 (t, 1H, *J* 9.0 Hz, H-4′), 2.51 (bs, 1H,O*H*), 1.54, 1.31 (2s, 6H, 2×isopropylidene-C*H*$_3$), 1.32 (d, 3H, *J* 6.3 Hz, C-C*H*$_3$), 1.07 (s, 9H, (SiC(C*H*$_3$)$_3$). ^{13}C NMR (CDCl$_3$, 75 MHz) δ: 155.1, 151.7, 138.8, 138.0, 135.6(5), 133.2, 129.7, 128.4(2), 128.2(2), 127.7(3), 127.7(5), 127.4(2), 127.4, 118.1(2), 114.5(2), 110.4 (*C*(CH$_3$)$_2$), 110.4 (C-1), 97.7 (C-1′), 80.1, 79.9(2), 75.0, 74.8, 73.8, 73.4, 71.9, 68.6, 67.4, 62.9 (C-6), 55.6 (C$_6$H$_4$OCH$_3$), 27.9, 26.4 (2×isopropylidene-*C*H$_3$), 26.7(3) (SiC(*C*H$_3$)$_3$), 19.2 (Si*C*(CH$_3$)$_3$), 17.9 (C-*C*H$_3$). HRMS Calcd. for C$_{52}$H$_{62}$O$_{11}$SiNa (M+Na)$^+$: 913.3959; found 913.3956.

***p*-Methoxyphenyl 2,3,4-tri-*O*-acetyl-α-L-rhamnopyranosyl-(1→2)-3,4-di-*O*-benzyl-α-L-rhamnopyranosyl-(1→2)-3,4-*O*-isopropylidene-6-*O*-(*tert*-butyl diphenylsilyl)-β-D-galactopyranoside (13).** A mixture of disaccharide acceptor **11** (1.7 g, 1.9 mmol), compound **12** (990 mg, 2.5 mmol) and MS 4Å (2 g) in dry CH$_2$Cl$_2$ (25 mL) was stirred under nitrogen for 1 hour. NIS (730 mg, 3.25 mmol) was added and the mixture was cooled to 10 °C when H$_2$SO$_4$-silica (50 mg) was added. The mixture was stirred for 45 minutes when TLC showed complete consumption of the acceptor **11**. The mixture was filtered through a pad of Celite and the filtrate was washed successively with Na$_2$S$_2$O$_3$ (2×30 mL), NaHCO$_3$ (2×30 mL) and brine (30 mL). The organic layer was separated, dried (Na$_2$SO$_4$) and evaporated *in vacuo*. The residue was purified by flash chromatography using *n*-hexane-EtOAc (2:1) to afford pure trisaccharide **13** (1.9 g, 86%) as white foam. [α]$_D^{25}$ +59 (*c* 1.1, CHCl$_3$).^{1}H NMR (CDCl$_3$, 300 MHz) δ: 7.72-7.22 (m, 20H, ArH), 7.01, 6.72 (2d, 4H, *J* 9.0 Hz, C$_6$*H*$_4$OCH$_3$), 5.46 (dd, 1H, *J* 1.5 Hz, 3.3 Hz, H-2″), 5.38 (dd, 1H, *J* 3.3 Hz, 10.2 Hz, H-3″), 5.29 (d, 1H, *J* 1.5 Hz, H-1″), 5.07 (t, 1H, *J* 10.2 Hz, H-4″), 4.92 (d, 1H, *J* 1.5 Hz, H-1′), 4.90, 4.69, 4.67, 4.57 (4d, AB system, 4H, 2×C*H*$_2$Ph), 4.70 (d, 1H, *J* 6.3 Hz, H-1), 4.17 (m, 3H, H-2,

H-3, H-6a), 4.09-3.90 (m, 5H, H-2′, H-4, H-5, H-5′, H-6b), 3.86 (dd, 1H, *J* 2.7 Hz, 9.3 Hz, H-3′), 3.71 (s, 3H, $C_6H_4OC\mathit{H_3}$), 3.57 (t, 1H, *J* 9.3 Hz, H-4′), 2.09, 2.05, 1.99 (3s, 9H, 3×$COC\mathit{H_3}$), 1.54, 1.31 (2s, 6H, 2×isopropylidene-$C\mathit{H_3}$), 1.34, 1.23 (2d, 6H, *J* 6.3 Hz, 2×C-$C\mathit{H_3}$). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 169.9, 169.7, 169.6 (3×$\mathit{C}OCH_3$), 155.1, 151.6, 138.9, 138.4, 135.5(4), 133.1, 129.6, 128.2(2), 128.1(2), 127.9(3), 127.6(6), 127.3(3), 118.1(2), 114.4(2) (ArC), 110.3 ($\mathit{C}(CH_3)_2$), 100.4 (C-1), 99.4 (C-1″), 97.5 (C-1′), 80.2, 79.8, 79.3, 76.2, 75.5, 74.9, 73.7, 73.3, 72.2, 71.1, 69.7, 69.1, 68.2, 66.7, 62.9 (C-6), 55.5 ($C_6H_4O\mathit{C}H_3$), 27.9, 26.3 (2×isopropylidene-$\mathit{C}H_3$), 26.7(3) (Si$\mathit{C}(CH_3)_3$), 20.7, 20.6(2) (3×CO$\mathit{C}H_3$), 20.6 (C-$\mathit{C}H_3$), 19.1 (Si$\mathit{C}(CH_3)_3$), 17.9 (C-$\mathit{C}H_3$). HRMS Calcd. for $C_{64}H_{78}O_{18}SiNa$ $(M+Na)^+$: 1185.4855; found 1185.4852.

***p*-Methoxyphenyl 2,3,4-tri-*O*-acetyl-α-L-rhamnopyranosyl-(1→2)-3,4-di-*O*-benzyl-α-L-rhamnopyranosyl-(1→2)-3,4-*O*-isopropylidene-β-D-galactopyranoside (14).** To a solution of trisaccharide **13** (1.8 g, 1.5 mmol) in dry THF (25 mL) was added Bu_4NF (1 M in THF, 5.9 mL, 2.0 mmol) followed by AcOH (1 mL, 17.4 mmol) and the solution was stirred at room temperature for 12 hours. The solvents were evaporated and the residue was purified by flash chromatography using *n*-hexane-EtOAc (2:1) to give pure compound **14** (1.2 g, 81%) as colourless thick syrup. $[\alpha]_D^{25}$ +81 (*c* 1.0, $CHCl_3$). ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.55-7.47 (m, 10H, ArH), 7.19, 6.99 (2d, 4H, *J* 9.0 Hz, $C_6\mathit{H_4}OCH_3$), 5.65 (dd, 1H, *J* 1.8 Hz, 3.3 Hz, H-2″), 5.56 (dd, 1H, *J* 3.3 Hz, 9.9 Hz, H-3″), 5.49 (d, 1H, *J* 1.8 Hz, H-1″), 5.26 (t, 1H, *J* 9.9 Hz, H-4″), 5.13, 4.94, 4.85, 4.79 (4d, 4H, *J* 11.7 Hz, 2×C$\mathit{H_2}$Ph), 5.10 (d, 1H, *J* 6.3 Hz, H-1), 4.95 (d, 1H, *J* 1.5 Hz, H-1′), 4.46-4.35 (m, 4H, H-2′, H-3, H-5″, H-6a), 4.24 (dd, 1H, *J* 6.3 Hz, 9.9 Hz, H-2), 4.19-4.12 (m, 4H, H-4, H-5, H-5′, H-6b), 4.04 (dd, 1H, *J* 2.7 Hz, 9.3 Hz, H-3′), 3.99 (s, 3H, $C_6H_5OC\mathit{H_3}$), 3.79 (t, 1H, *J* 9.6 Hz, H-4′), 2.36, 2.31, 2.26

(3s, 9H, 3×COC*H*$_3$), 1.82, 1.57 (2s, 6H, 2 × isopropylidene-C*H*$_3$), 1.57 (d, 3H, *J* 6.0 Hz, C-C*H*$_3$), 1.45 (d, 3H, *J* 6.3 Hz, C-C*H*$_3$). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 169.6, 169.5, 169.4 (3×*C*OCH$_3$), 155.4, 151.5, 139.0, 138.5, 128.3(2), 128.2(2), 127.8(2), 127.4(2), 127.3(2), 117.9(2), 114.7(2) (ArC), 110.6 (*C*(CH$_3$)$_2$), 100.3 (C-1), 99.5 (C-1″), 97.5 (C-1′), 80.1(2), 79.4, 75.1, 75.0, 73.8, 73.7, 72.3, 71.2, 69.9, 69.2, 68.4, 66.8, 62.2 (C-6), 55.5 (C$_6$H$_4$O*C*H$_3$), 28.1, 26.6 (2×isopropylidene-*C*H$_3$), 20.9, 20.8, 20.7 (3×CO*C*H$_3$), 18.1 (C-*C*H$_3$), 17.2 (C-*C*H$_3$). HRMS Calcd. for $C_{48}H_{60}O_{18}Na$ $(M+Na)^+$: 947.3677; found 947.3675.

***p*-Methoxyphenyl 2,3,4-tri-*O*-acetyl-α-L-rhamnopyranosyl-(1→2)-3,4-di-*O*-acetyl-α-L-rhamnopyranosyl-(1→2)-3,4,6-tri-*O*-acetyl-β-D-galactopyranoside (15).** A solution of compound **14** (1.2 g, 1.3 mmol) in AcOH-H_2O (9:1, 20 mL) was stirred at 85 °C for 3 hours. After the removal of solvents *in vacuo*, the residue was dissolved MeOH (1:2, 15 mL), $Pd(OH)_2$ (50 mg) was added and the mixture was stirred under H_2 atmosphere for 6 hours. The mixture was filtered through a pad of Celite and the filtrate was evaporated *in vacuo*. The residue was dissolved in dry pyridine (15 mL), Ac_2O (10 mL) was added and the solution was stirred at room temperature for 3 hours. The solvents were evaporated and co-evaporated with toluene to remove traces of pyridine. The residue was purified by flash chromatography using *n*-hexane-EtOAc (3:1) to afford pure compound **15** (1.1 g, 83%) as white foam. $[\alpha]_D^{25}$ +67 (*c* 1.0, $CHCl_3$). ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 6.95, 6.76 (2d, 4H, *J* 9.0 Hz, C$_6$*H*$_4$OCH$_3$), 5.32 (bs, 1H, H-4), 5.24 (dd, 1H, *J* 2.1 Hz, 8.7 Hz, H-3″), 5.22 (bs, 1H, H-2"), 5.20-4.94 (m, 5H, H-1", H-3, H-3', H-4', H-4"), 4.87 (d, 1H, *J* 7.8 Hz, H-1), 4.75 (bs, 1H, H-1'), 4.17-4.02 (m, 4H, H-2', H-5', H-5", H-6a), 3.95 (m, 3H, H-2, H-5, H-6b), 3.74 (s, 3H, C$_6$H$_4$OC*H*$_3$), 2.12, 2.10, 2.01(3), 1.99, 1.96(2) (8s, 24H, 8×COC*H*$_3$), 1.21 (d, 3H, *J* 6.0 Hz, C-C*H*$_3$), 1.18 (d, 3H, *J* 6.3 Hz, C-C*H*$_3$). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 169.7, 169.4(2), 169.2(3), 169.1(2)

(8×*C*OCH$_3$), 155.7, 150.7, 118.5 (2), 114.5(2) (ArC), 100.8 (C-1), 99.4 (C-1″), 99.1 (C-1′), 77.4, 73.4, 73.1, 71.1, 70.8, 70.6, 70.2, 69.8, 68.5, 67.1, 67.0, 66.8, 61.1 (C-6), 55.3 ($C_6H_4OCH_3$), 20.7(3), 20.6(3), 20.5(2) (8×CO*C*H$_3$), 17.4, 17.3 (2×C-*C*H$_3$). HRMS Calcd. for $C_{41}H_{54}O_{23}Na$ $(M+Na)^+$: 937.2954; found937.2952.

2,3,4-tri-*O*-acetyl-α-L-rhamnopyranosyl-(1→2)-3,4-di-*O*-acetyl-α-L-rhamnopyranosyl-(1→2)-3,4,6-tri-*O*-acetyl-β-D-galactopyranosyl trichloroacetimidate (16). To a stirred solution of compound **15** (1.0 g, 1.1 mmol) in CH_3CN-H_2O (9:1, 20 mL) was added CAN (1.2 g, 2.2 mmol) and the mixture was stirred at room temperature for 1 hour when TLC (*n*-hexane-EtOAc, 2:1) showed complete conversion of the starting material to a slower moving component. The mixture was concentrated *in vacuo*, the residue was dissolved in CH_2Cl_2 (30 mL) and washed with H_2O (2×30 mL). The organic layer was collected, dried (Na_2SO_4) and concentrated to syrup. The crude product was purified by flash chromatography using *n*-hexane-EtOAc (2:1) as eluent to afford 2,3,4-tri-*O*-acetyl-α-L-rhamnopyranosyl-(1→2)-3,4-di-*O*-acetyl-α-L-rhamnopyranosyl-(1→2)-3,4,6-tri-*O*-acetyl-β-D-galactopyranose (800 mg, 91%) as light yellow foam. It was then dissolved in dry CH_2Cl_2 (20 mL), CCl_3CN (270 μL, 2.7 mmol) was added followed by DBU (150 μL, 1.0 mmol) and the solution was stirred at room temperature for 2 hours. The solution was concentrated *in vacuo* and the crude product was purified by flash chromatography using *n*-hexane-EtOAc (3:1) as eluent to afford pure compound **16** (860 mg, 93%) as white foam. Since the glycosyl trichloroacetimidates are not enough stable, it was used for further reaction without detailed characterization.

Propargyl 4,6-*O*-benzylidene-3-*O*-(4-methoxybenzyl)-β-D-glucopyranoside (18). A mixture of known propargyl 4,6-*O*-benzylidene β-D-glucopyranoside **17** (3.0 g, 3.8 mmol) and Bu_2SnO (1.0 g,

4.2 mmol) in dry MeOH (30 mL) was refluxed for 3 hours. The resulting solution was concentrated *in vacuo*, co-evaporated with toluene and the residue was dried under vacuum for 30 minutes. It was dissolved in dry DMF (20 mL) and MBnCl (665 μL, 4.9 mmol) was added followed by CsF (750 mg, 4.2 mmol) and the solution was stirred at room temperature for 6 hours. After evaporating the solvents *in vacuo* the residue was dissolved in CH_2Cl_2 (40 mL) and washed with H_2O (50 mL) and brine (50 mL). The organic layer was collected, dried (Na_2SO_4) and evaporated. The crude product thus obtained was purified by flash chromatography using *n*-hexane-EtOAc (3:1) as eluent to afford pure compound **18** (3.5 g, 84%) as white foam. $[\alpha]_D^{25}$ +107 (*c* 1.2, $CHCl_3$). ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.50-7.38 (m, 5H, ArH), 7.29, 6.82 (2d, 4H, *J* 9.0 Hz, $C_6H_4OCH_3$), 5.56 (s, 1H, C*H*Ph), 4.87, 4.75 (2d, 2H, AB system, *J* 11.4 Hz, CH_2Ph), 4.60 (d, 1H, *J* 7.5 Hz, H-1), 4.40 (m, 2H, OCH_2–C≡CH), 4.35 (dd, 1H, *J* 4.8 Hz, 10.2 Hz, H-6a), 3.80 (s, 3H, $C_6H_4OCH_3$), 3.77 (t, 1H, *J* 10.5 Hz, H-3), 3.67-3.56 (m, 3H, H-2, H-4, H-6b), 3.46 (m, 1H, H-5), 2.63 (bs, 1H, O*H*), 2.48 (t, 1H, *J* 2.4 Hz, OCH_2–C≡C*H*). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 159.3, 137.4, 130.5(2), 129.7, 128.9(2), 126.1(2), 113.8(2) (ArC), 101.3 (*C*HPh), 101.0 (C-1), 81.3, 79.7, 78.5 (OCH_2–*C*≡CH), 75.6, 74.2, 74.1, 68.6, 66.6 (C-6), 56.0, 55.0 ($C_6H_4OCH_3$). HRMS Calcd. for $C_{24}H_{26}O_7Na$ $(M+Na)^+$: 449.1576; found 449.1573.

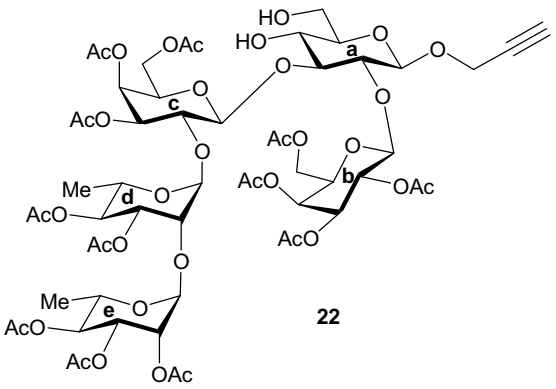
Propargyl 2,3,4,6-tetra-*O*-acetyl-β-D-galactopyranosyl-(1→2)-4,6-*O*-benzylidene-β-D-glucopyranoside (20). A mixture of compound **18** (2 g, 4.7 mmol), compound **19** (2.6 g, 5.6 mmol) and MS 4Å (2 g) in dry CH_2Cl_2 (25 mL) was stirred under nitrogen for one hour. NIS (1.5 g, 6.7 mmol) was added and the mixture was cooled to -40 °C followed by addition of H_2SO_4-silica (40 mg). The mixture was allowed to stir at -40 °C for 6 hours when TLC showed complete consumption of the acceptor **18**. At this point H_2SO_4-silica (30 mg) was added and the reaction

temperature was raised to room temperature and the mixture was stirred for an additional 2 hours. The mixture was filtered through a pad of Celite. The filtrate was diluted with CH_2Cl_2 (20 mL) and washed successively with $Na_2S_2O_3$ (2×50 mL), $NaHCO_3$ (2×50 mL) and brine (50 mL). The organic layer was collected, dried (Na_2SO_4) and evaporated *in vacuo*. The residue was purified by flash chromatography using *n*-hexane-EtOAc (3:1) to afford pure compound **20** (2.4 g, 82%). $[\alpha]_D^{25}$ +47 (*c* 1.0, $CHCl_3$). ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.49-7.34 (m, 5H, ArH), 5.53 (s, 1H, C*H*Ph), 5.39 (bd, 1H, *J* 2.7 Hz, H-4′), 5.23 (dd, 1H, *J* 8.1 Hz, 10.5 Hz, H-2′), 5.06 (dd, 1H, *J* 2.7 Hz, 10.5 Hz, H-3′), 4.81 (d, 1H, *J* 8.1 Hz, H-1′), 4.67 (d, 1H, *J* 7.8 Hz, H-1), 4.47 (dd, 1H, *J* 2.4 Hz, 12.6 Hz, H-6a′), 4.45-4.30 (m, 3H, H-2, H-5′, H-6b′), 4.21 (m, 2H, OC*H*$_2$–C≡CH), 4.10 (m, 2H, H-6a, H-6b), 3.86 (t, 1H, *J* 10.2 Hz, H-3), 3.79 (t, 1H, *J* 10.2 Hz, H-4), 3.48 (m, 1H, H-5), 2.95 (bs, 1H, O*H*), 2.53 (t, 1H, *J* 2.4 Hz, OCH$_2$–C≡C*H*), 2.15, 2.06, 2.04, 1.98 (4s, 12H, 4×COC*H*$_3$). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 170.7, 170.2, 170.1, 170.0 (4×*C*OCH$_3$), 136.8, 129.2, 128.2(2), 126.2(2) (ArC), 101.9 (*C*HPh), 101.7 (C-1′), 100.9 (C-1), 83.5, 79.7, 78.2 (OCH$_2$–*C*≡CH), 75.6, 72.1, 70.9, 70.7, 69.7, 68.4, 66.9, 66.1, 61.2, 56.9, 20.9, 20.6(2), 20.5 (4×CO*C*H$_3$). HRMS Calcd. for $C_{30}H_{36}O_{15}Na$ $(M+Na)^+$: 659.1952; found 659.1954.

Propargyl 2,3,4-tri-*O*-acetyl-α-L-rhamnopyranosyl-(1→2)-3,4-di-*O*-acetyl-α-L-rhamnopyranosyl-(1→2)-3,4,6-tri-*O*-acetyl-β-D-galactopyranosyl-(1→3)-2-*O*-(2,3,4,6-tetra-*O*-acetyl-β-D-galactopyranosyl)-4,6-*O*-benzylidene-β-D-glucopyranoside (21). A mixture of compound **16** (800 mg, 0.85 mmol), compound **20** (450 mg, 0.7 mmol) and MS 4Å (500 mg) in dry CH_2Cl_2 (10 mL) was stirred under nitrogen at room temperature for 30 minutes. TMSOTf (20 μL) was added and the stirring was continued at room temperature till TLC showed complete consumption of the acceptor disaccharide **20** (6 hours). Then the mixture was neutralized with Et_3N

and filtered through a pad of Celite, washed twice with CH_2Cl_2 (5 mL) and the entire CH_2Cl_2 filtrate was evaporated *in vacuo*. The crude residue thus obtained was purified by flash chromatography using 2:1 *n*-hexane-EtOAc as eluent to afford the pentasaccharide **21** (810 mg) along with the trehalose type product resulted from the self condensation of the donor trisaccharide as evident from mass spectroscopy. We failed to separate this impurity after repeated chromatography and proceed for the next step with the mixture.

Propargyl 2,3,4-tri-*O*-acetyl-α-L-rhamnopyranosyl-(1→2)-3,4-di-*O*-acetyl-α-L-rhamnopyranosyl-(1→2)-3,4,6-tri-*O*-acetyl-β-D-galactopyranosyl-(1→3)-2-*O*-(2,3,4,6-tetra-*O*-acetyl-β-D-galactopyranosyl)-β-D-glucopyranoside (22). Compound **21** (as obtained from the previous step) (800 mg, 0.6 mmol) was dissolved in 80% aqueous AcOH (10 mL) and the solution was stirred at 80 °C for 2 hours when TLC showed complete conversion of the starting material to a slower running spot and to our satisfaction, a light spot was observed at the site of the starting material, assumed to be the trehalose-type impurity carried over from the last step. Flash chromatography using *n*-hexane-EtOAc (2:1) afforded the pure compound **22** (630 mg). $[\alpha]_D^{25}$ +73 (*c* 1.1, $CHCl_3$). ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 5.71 (d, 1H, *J* 3.9 Hz, H-4^b), 5.42-5.40 (m, 2H, H-2^e, H-4^c), 5.37(dd, 1H, *J* 3.3 Hz, 9.9 Hz, H-3^e), 5.30-5.27 (m, 2H, H-2^b, H-4^e), 5.17 (m, 2H, H-1^e, 3^b), 5.07-5.00 (m, 3H, H-3^c, H-3^d, H-1^d), 4.95 (d, 1H, *J* 8.4 Hz, H-1^a), 4.84 (d, 1H, *J* 8.0 Hz, H-1^b), 4.68 (m, 2H, C*H*$_2$-C≡CH), 4.66 (d, 1H, *J* 7.6 Hz, H-1^c), 4.45 (m, 2H, H-2^a, H-6^b), 4.21-4.15 (m, 2H, H-5^d, H-2^d), 4.13 (m, 3H, H-6'^{b}, H-6^c, H-6'^{c}), 4.01-3.91 (m, 4H, H-2^c, H-3^a, H-4^a, H-6^a), 3.78 (m, 1H, H-6'^{a}), 3.58 (m, 2H, H-5^b, H-5^c), 3.17 (m,



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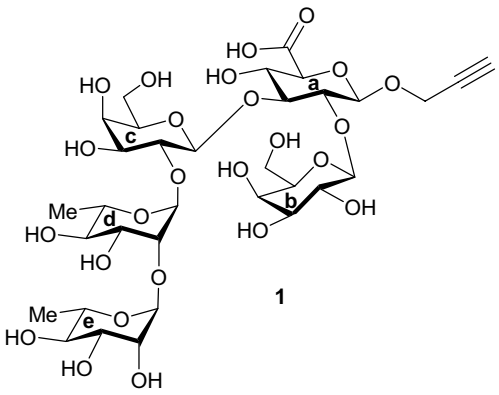
1H, H-5[a]), 2.53 (s, 1H, CH_2-C≡C*H*), 2.17, 2.14, 2.13, 2.11, 2.09, 2.08, 2.04, 2.03, 1.99, 1.98, 1.97, 1.95 (12s, 36H, 12 × COC*H*$_3$), 1.34 (d, 3H, *J* 6.4 Hz, C-C*H*$_3$), 1.17 (d, 3H, *J* 6.0 Hz, C-C*H*$_3$). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 170.6, 170.2(3), 170.1(3), 169.9(2), 169.73(2), 169.49 (12×*C*OCH$_3$), 100.9(C-1[a]), 99.0(C-1[b]), 98.9(C-1[c]), 98.7(C-1[d]), 97.9(C-1[e]), 81.2, 79.8, 79.7, 78.4(OCH_2–*C*≡CH), 75.9, 75.5, 75.1, 74.5, 72.3, 71.7, 70.8, 70.6, 70.5, 70.0, 69.9, 69.5, 68.9, 68.2, 67.1, 66.5, 61.8 (C-6[b]), 61.3(C-6[a]), 60.9 (C-6[c]), 56.5, 29.6(4), 20.8(3), 20.6(3), 20.4(2) (12×CO*C*H$_3$), 18.2, 17.3 (2×C-*C*H$_3$). HRMS Calcd. for $C_{57}H_{78}O_{36}Na$ $(M+Na)^+$: 1361.4171; found 1361.4173.

Propargyl 2,3,4-tri-*O*-acetyl-α-L-rhamnopyranosyl-(1→2)-3,4-di-*O*-acetyl-α-L-rhamnopyranosyl-(1→2)-3,4,6-tri-*O*-acetyl-β-D-galactopyranosyl-(1→3)-2-*O*-(2,3,4,6-tetra-*O*-acetyl-β-D-galactopyranosyl)-β-D-glucopyranosiduronic acid (23). To a solution of the compound **22** (600 mg, 0.45 mmol) in 10 mL CH_2Cl_2 and 2 mL H_2O were added aq. NaBr (1M 0.2 mL), aq. tetrabutylammonium bromide (1M, 0.4 mL), TEMPO (19 mg) and saturated aq. $NaHCO_3$ (1.1 mL) at 0 ^oC. To the resulting mixture, aq. NaOCl (1.3 mL) was added and the mixture was allowed to stir for 1.5 hours when the temperature was raised to room temperature. At this point TLC showed complete conversion of the starting material to a faster moving spot, presumably the corresponding aldehyde derivative. The mixture was neutralized with 1N HCl (as required) to keep the pH of the mixture at 6-7. Then *tert*-butanol (6 mL), $NaOCl_2$ (0.4g in 1.5 mL H_2O) and NaH_2PO_4 (0.5 g in 4 mL H_2O) were added and the mixture was allowed to stir at room temperature for another 4 hours when TLC showed complete conversion. The mixture was diluted with saturated NaH_2PO_4 and the product was extracted with EtOAc. The organic layer was dried (Na_2SO_4) and evaporated. The crude product thus obtained was purified by flash chromatoghraphy using *n*-hexane-EtOAc (1:1) to *n*-hexane-EtOAc (1:3) to afford compound **23** (470 mg, 78 %). ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 170.4

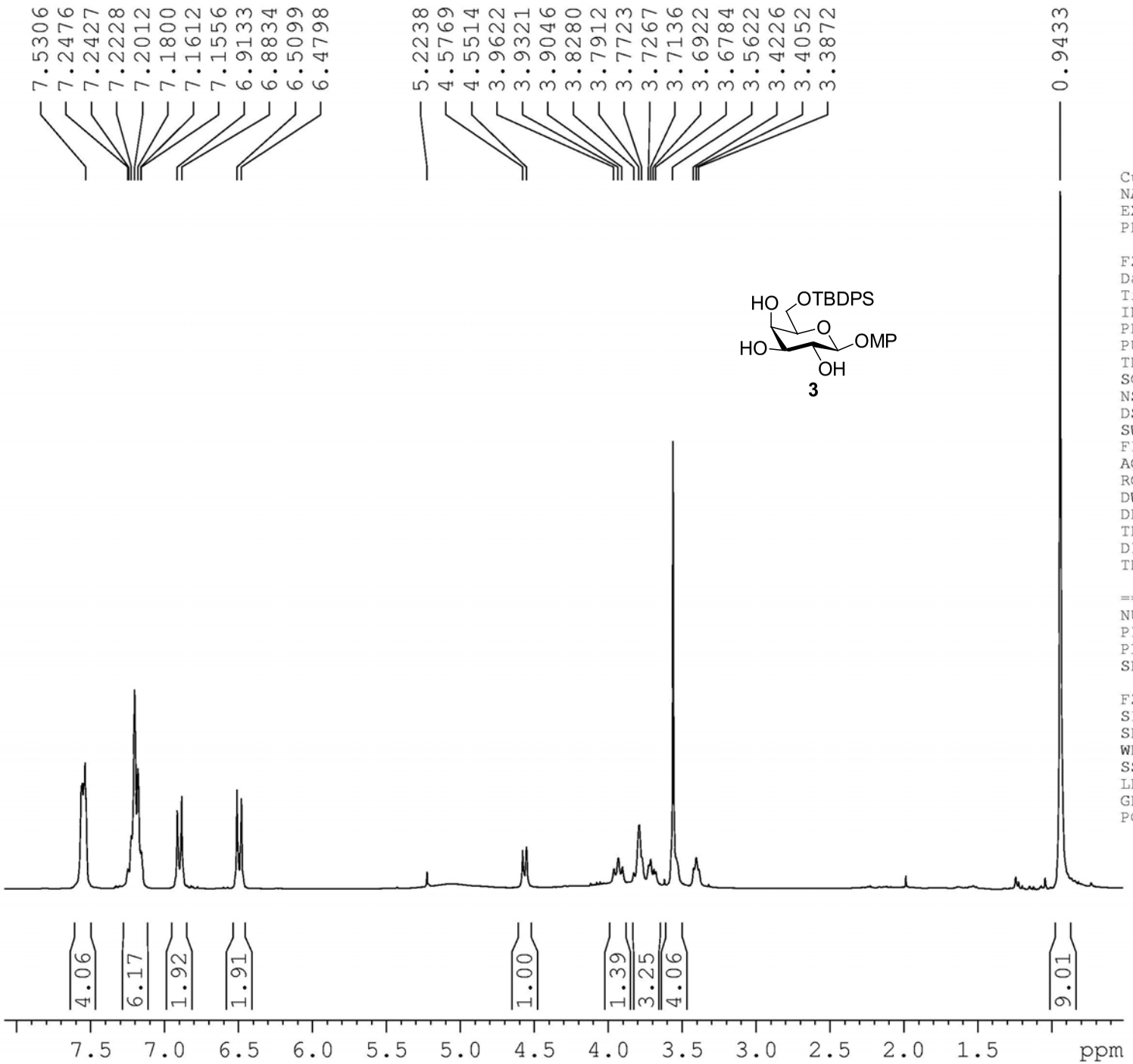
(*C*OOH), 170.1(2), 170.0(4), 169.9(2), 169.7, 169.6, 169.5, 169.4 (12×*C*OCH$_3$), 101.0 (C-1^a), 100.7 (C-1^b), 98.9 (C-1^c), 98.8 (C-1^d), 97.1 (C-1^e), 81.2, 80.6, 79.5, 79.3, 78.3 (OCH2–*C*≡CH), 75.6, 75.4, 74.9, 74.7, 71.4, 71.3, 70.7, 70.4, 70.3, 70.1, 69.9, 69.5, 69.4, 68.8, 68.6, 68.2, 67.0, 66.8, 66.6, 61.5 (C-6^b), 61.3(C-6^c), 57.5, 20.7(4), 20.6(4), 20.5(2), 20.4(2) (12×CO*C*H$_3$), 17.5, 17.2 (2×C-*C*H$_3$). HRMS Calcd. for $C_{57}H_{76}O_{37}Na$ $(M+Na)^+$: 1375.3963; found 1375.3960.

Propargyl α-L-rhamnopyranosyl-(1→2)-α-L-rhamnopyranosyl-(1→2)-β-D-galactopyranosyl-(1→3)-2-*O*-(β-D-galactopyranosyl)-β-D-glucopyranosiduronic acid (1). To a solution of the protected pentasaccharide **23** (450 mg, 0.33 mmol) in dry MeOH (5 mL) was added NaOMe in MeOH (0.5 M, 0.5 mL) and the solution was stirred at room temperature for 4 hours. The solution was neutralized with DOWEX 50W H^+ resin and filtered through a cotton plug. The filtrate was evaporated and washed with CH_2Cl_2 (5ml) removing TEMPO salt to afford compound **1** (240 mg, 86%) in 99% yield. ^{1}H NMR (D_2O, 400 MHz) δ: 5.80 (d, 1H, *J* 7.2 Hz, H-1^c), 5.18 (bs, 1H, H-1^d), 4.98 (d, 1H, *J* 1.6 Hz, H-1^e), 4.38 (d, 1H, *J* 7.2 Hz, H-1^b), 4.34 (d, 1H, *J* 7.6 Hz, H-1^a), 4.06-3.96 (m, 8H, H-2^d, H-2^e, H-3^a, H-4^b, H-5^e, H-6^c, C*H*$_2$-C≡CH), 3.94-3.55 (m, 16H, H-2^a, H-2^b, H-2^c, H-3^b, H-3^c, H-3^d, H-3^e, H-4^a, H-4^c, H-5^a, H-5^b, H-5^c, H-5^d, H-6^b, H-6'^{b}, H-6'^{c}), 3.48 (t, 2H, *J* 9.6 Hz, H-4^d, H-4^e), 1.94 (bs, 1H, CH$_2$-C≡C*H*), 1.37 (d, 3H, *J* 6.0 Hz, C-C*H*$_3$), 1.32 (d, 3H, *J* 6.0 Hz, C-C*H*$_3$). ^{13}C NMR (D_2O, 100 MHz) δ: 175.4 (*C*OOH), 102.8 (C-1^a), 101.9 (C-1^b), 100.8 (C-1^c), 99.4 (C-1^d), 96.0 (C-1^e), 79.6, 78.2, 77.5, 76.3, 75.7, 75.0, 74.6(2), 74.5, 73.2, 73.0, 72.3, 72.1, 71.4, 70.8, 70.2(2), 69.8, 69.3, 68.6(2), 61.3, 60.4, 56.3, 17.1, 16.7 (2 × CO*C*H$_3$). HRMS Calcd. for $C_{33}H_{52}O_{25}Na$ $(M+Na)^+$:871.2695; found 871.2698.



1

i. Perrin, D. D.; Amarego, W. L.; Perrin, D. R. *Purification of Laboratory Chemicals*; (Pergamon, London, 1996).

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EXPNO                       60
PROCNO                       1

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Time                     15.45
INSTRUM                  spect
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PULPROG                   zg30
TD                       32768
SOLVENT                  CDCl3
NS                          16
DS                           2
SWH                  6188.119 Hz
FIDRES               0.188846 Hz
AQ                   2.6477044 sec
RG                        71.8
DW                     80.800 usec
DE                      6.00 usec
TE                      300.0 K
D1                1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                     11.60 usec
PL1                    -1.00 dB
SFO1             300.1318534 MHz

F2 - Processing parameters
SI                       16384
SF               300.1300000 MHz
WDW                         EM
SSB                          0
LB                     0.30 Hz
GB                           0
PC                        1.00
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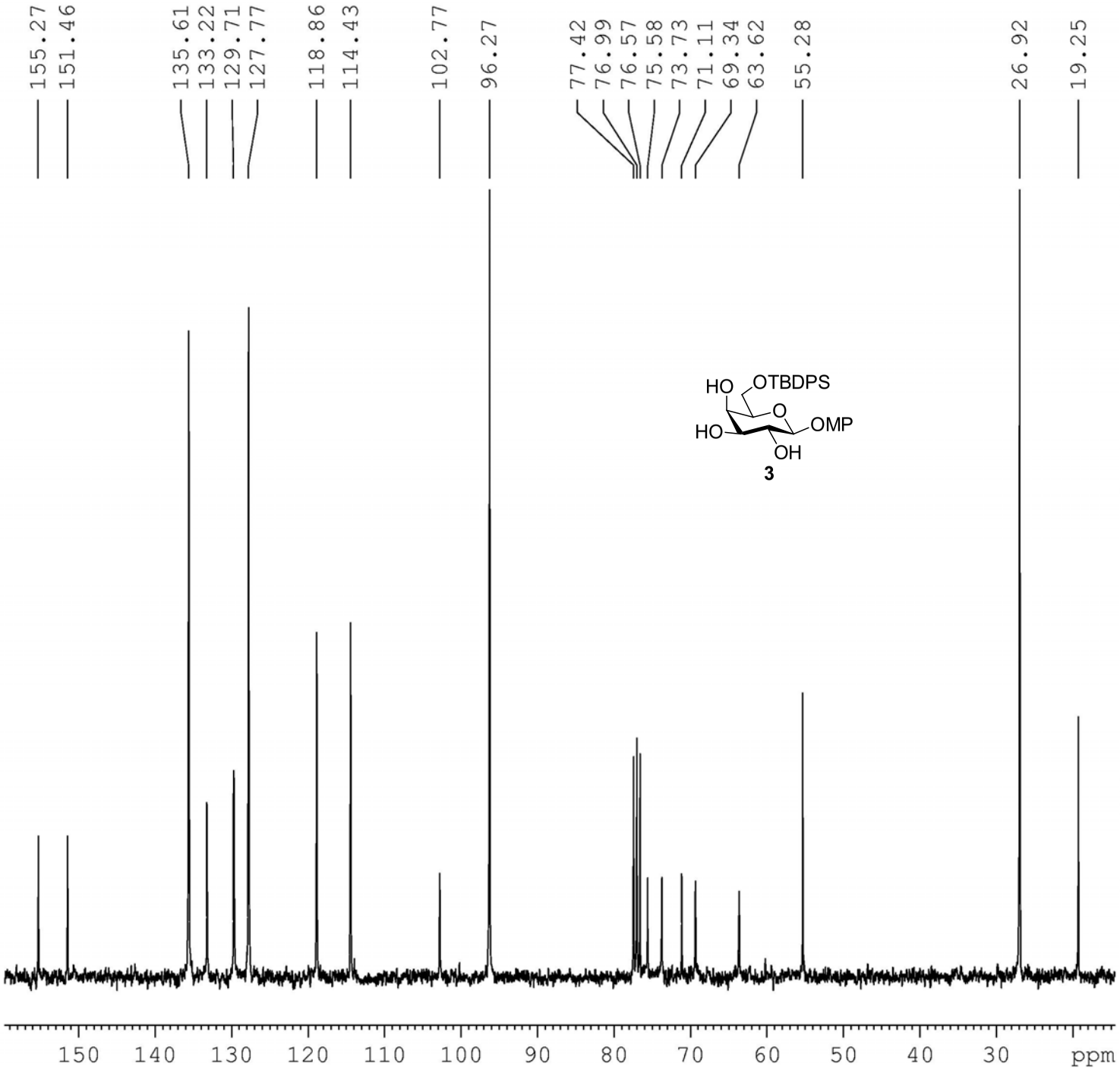


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NAME                 11.10.06
EXPNO                      90
PROCNO                      1

F2 - Acquisition Parameters
Date_              20061011
Time                   16.39
INSTRUM                spect
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PULPROG                zgpg30
TD                     65536
SOLVENT                CDCl3
NS                       512
DS                         0
SWH                19531.250 Hz
FIDRES             0.298023 Hz
AQ                 1.6777716 sec
RG                         9
DW                    25.600 usec
DE                     6.00 usec
TE                     300.0 K
D1                2.00000000 sec
d11               0.03000000 sec
DELTA             1.89999998 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     13C
P1                      8.70 usec
PL1                    -3.00 dB
SFO1              75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                     1H
PCPD2                 80.00 usec
PL2                    -1.00 dB
PL12                   17.00 dB
PL13                   21.00 dB
SFO2             300.1312005 MHz

F2 - Processing parameters
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SF                75.4677286 MHz
WDW                         EM
SSB                         0
LB                      3.00 Hz
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PC                       1.00
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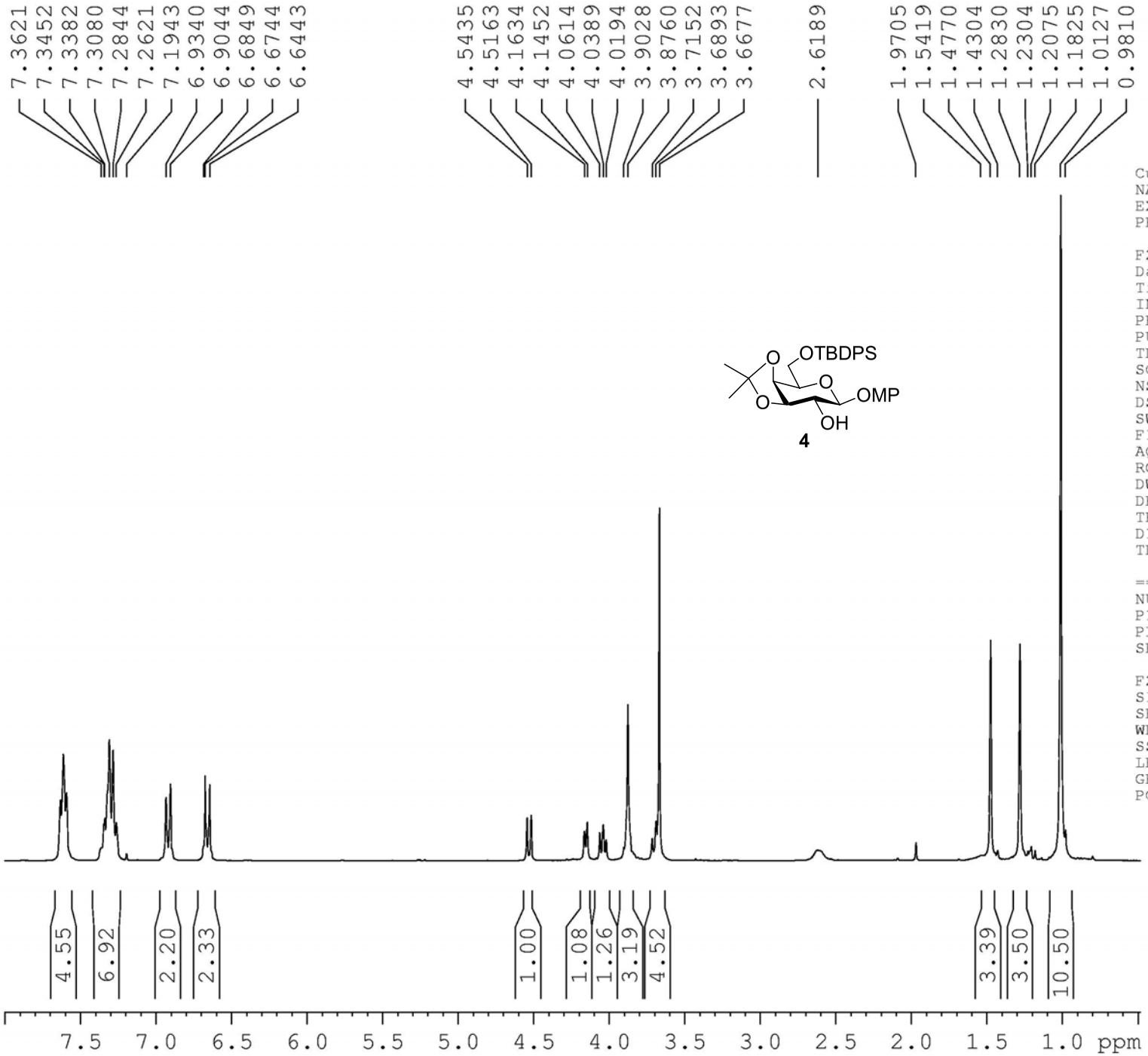


Current Data Parameters

NAME	06.10.06
EXPNO	130
PROCNO	1

F2 - Acquisition Parameters

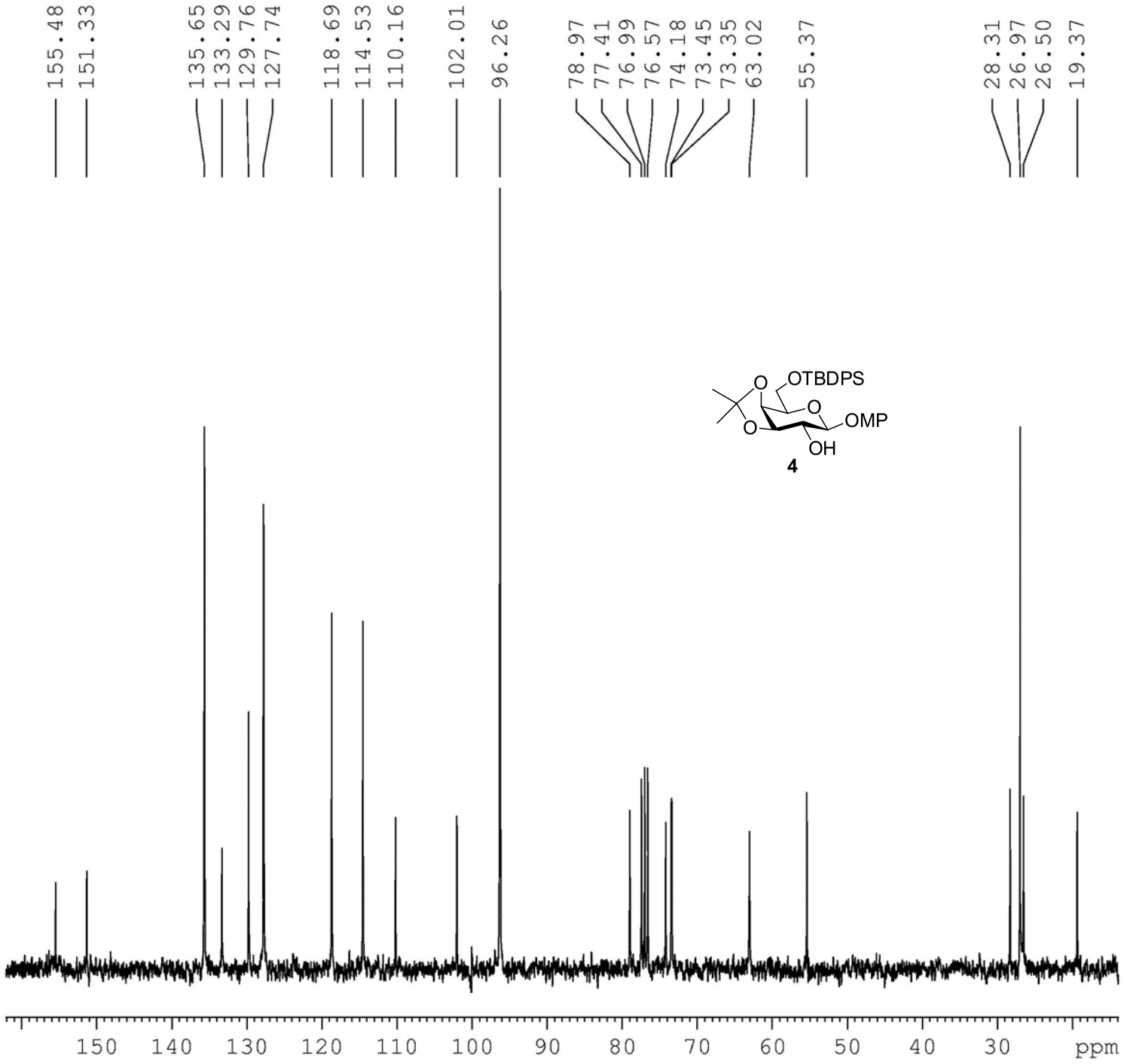
Date_	20061006
Time	15.24
INSTRUM	spect
PROBHD	5 mm QNP 1H/13
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	6188.119 Hz
FIDRES	0.188846 Hz
AQ	2.6477044 sec
RG	144
DW	80.800 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
TD0	1

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

NUC1	1H
P1	11.60 usec
PL1	-1.00 dB
SFO1	300.1318534 MHz

F2 - Processing parameters

SI	16384
SF	300.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



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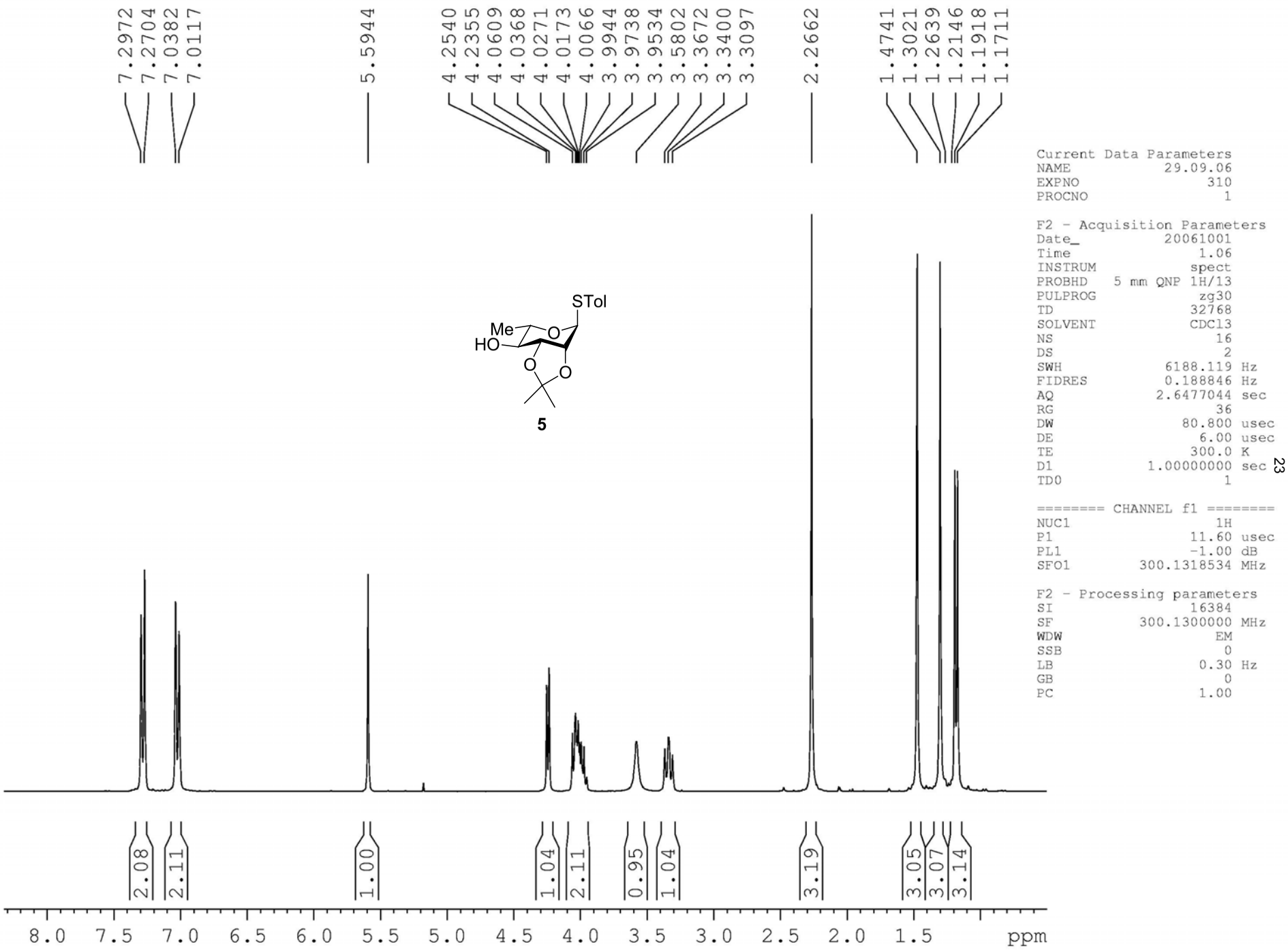
EXPNO 70
PROCNO 1

F2 - Acquisition Parameters
Date_ 20061016
Time 10.21
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 240
DS 0
SWH 19531.250 Hz
FIDRES 0.298023 Hz
AQ 1.6777716 sec
RG 9
DW 25.600 usec
DE 6.00 usec
TE 302.7 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 8.70 usec
PL1 -3.00 dB
SFO1 75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 17.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677424 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

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Current Data Parameters
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EXPNO                      310
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20061001
Time                      1.06
INSTRUM                  spect
PROBHD    5 mm QNP 1H/13
PULPROG                   zg30
TD                       32768
SOLVENT                  CDCl3
NS                          16
DS                           2
SWH                  6188.119 Hz
FIDRES               0.188846 Hz
AQ                   2.6477044 sec
RG                          36
DW                     80.800 usec
DE                      6.00 usec
TE                      300.0 K
D1                1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                     11.60 usec
PL1                    -1.00 dB
SFO1           300.1318534 MHz

F2 - Processing parameters
SI                       16384
SF             300.1300000 MHz
WDW                         EM
SSB                          0
LB                      0.30 Hz
GB                           0
PC                        1.00
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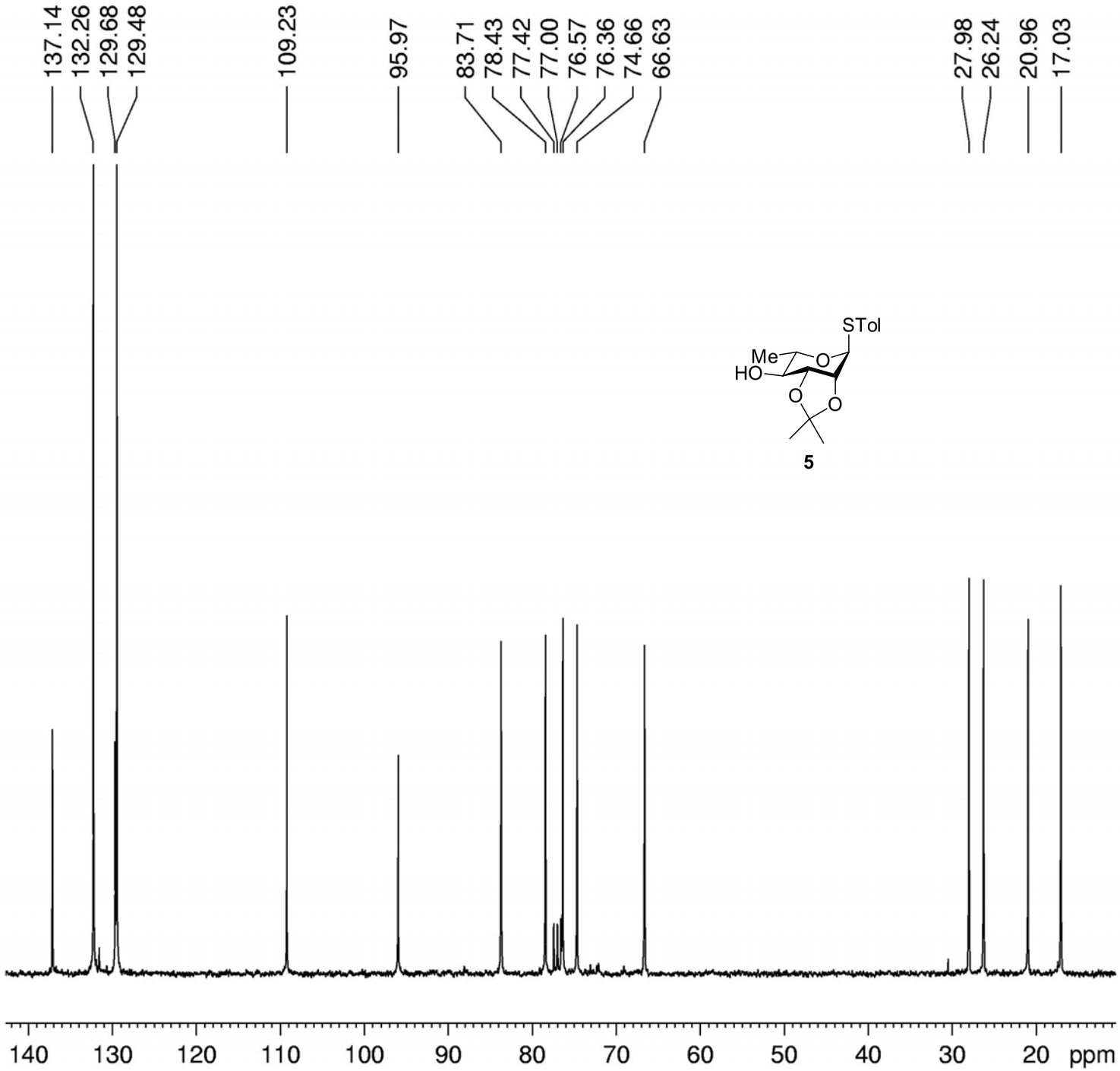

5

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Current Data Parameters
NAME                  10.10.06
EXPNO                      210
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20061011
Time                     12.23
INSTRUM                  spect
PROBHD    5 mm QNP 1H/13
PULPROG                zgpg30
TD                        65536
SOLVENT                  CDCl3
NS                         251
DS                           0
SWH                  19531.250 Hz
FIDRES                0.298023 Hz
AQ                    1.6777716 sec
RG                           9
DW                      25.600 usec
DE                       6.00 usec
TE                      300.0 K
D1                 2.00000000 sec
d11                0.03000000 sec
DELTA              1.89999998 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       13C
P1                        8.70 usec
PL1                      -3.00 dB
SFO1                75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                    80.00 usec
PL2                      -1.00 dB
PL12                     17.00 dB
PL13                     21.00 dB
SFO2               300.1312005 MHz

F2 - Processing parameters
SI                         32768
SF                  75.4677573 MHz
WDW                           EM
SSB                           0
LB                        3.00 Hz
GB                            0
PC                         1.00
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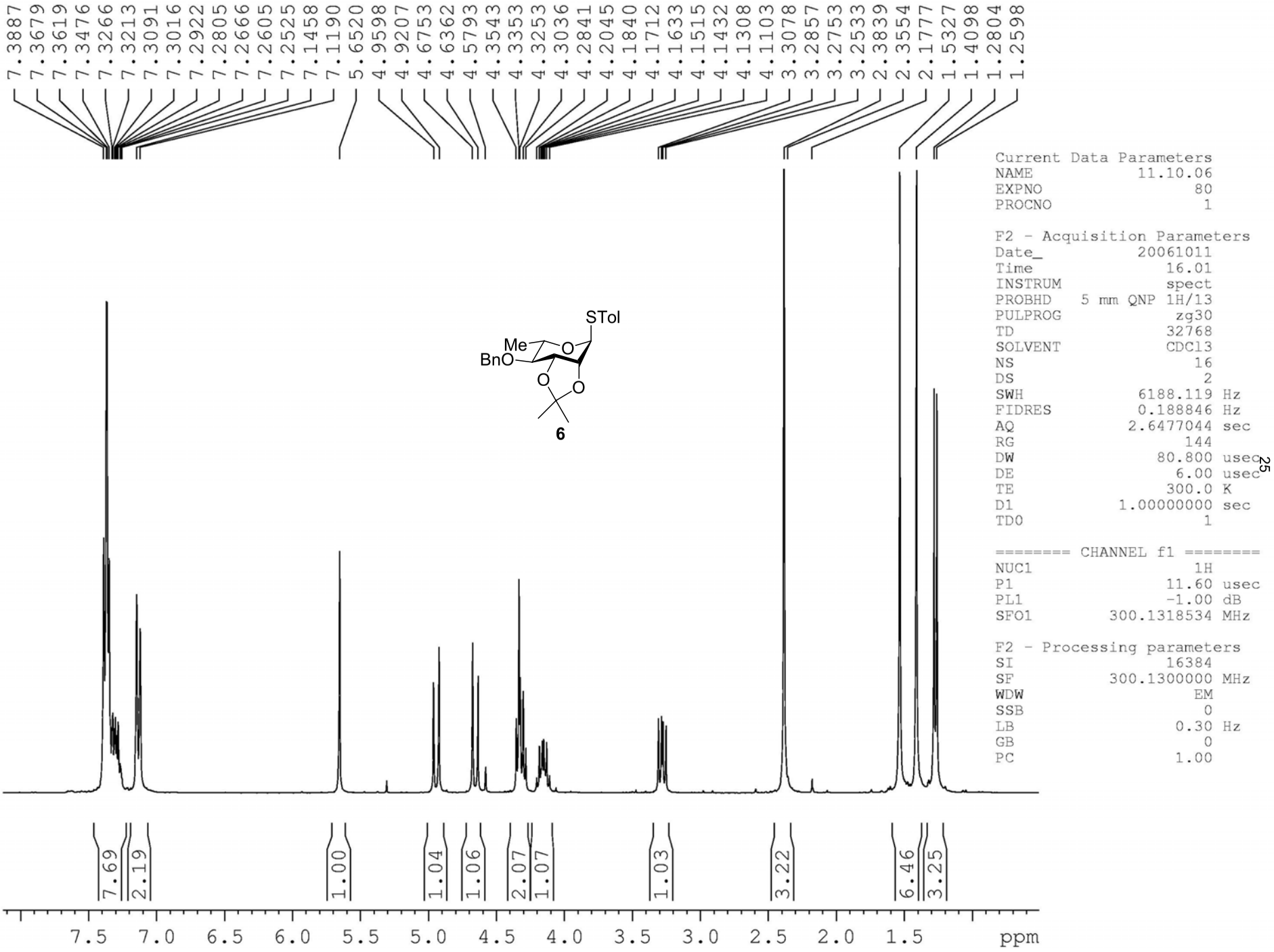
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7.2605
7.2525
7.1458
7.1190
5.6520
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4.6362
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4.3543
4.3353
4.3253
4.3036
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1.5327
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1.2804
1.2598
STol
Me
BnO
O
O
O
6
Current Data Parameters
NAME 11.10.06
EXPNO 80
PROCNO 1
F2 - Acquisition Parameters
Date_ 20061011
Time 16.01
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 144
DW 80.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.60 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 16384
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.69
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1.00
1.04
1.06
2.07
1.07
1.03
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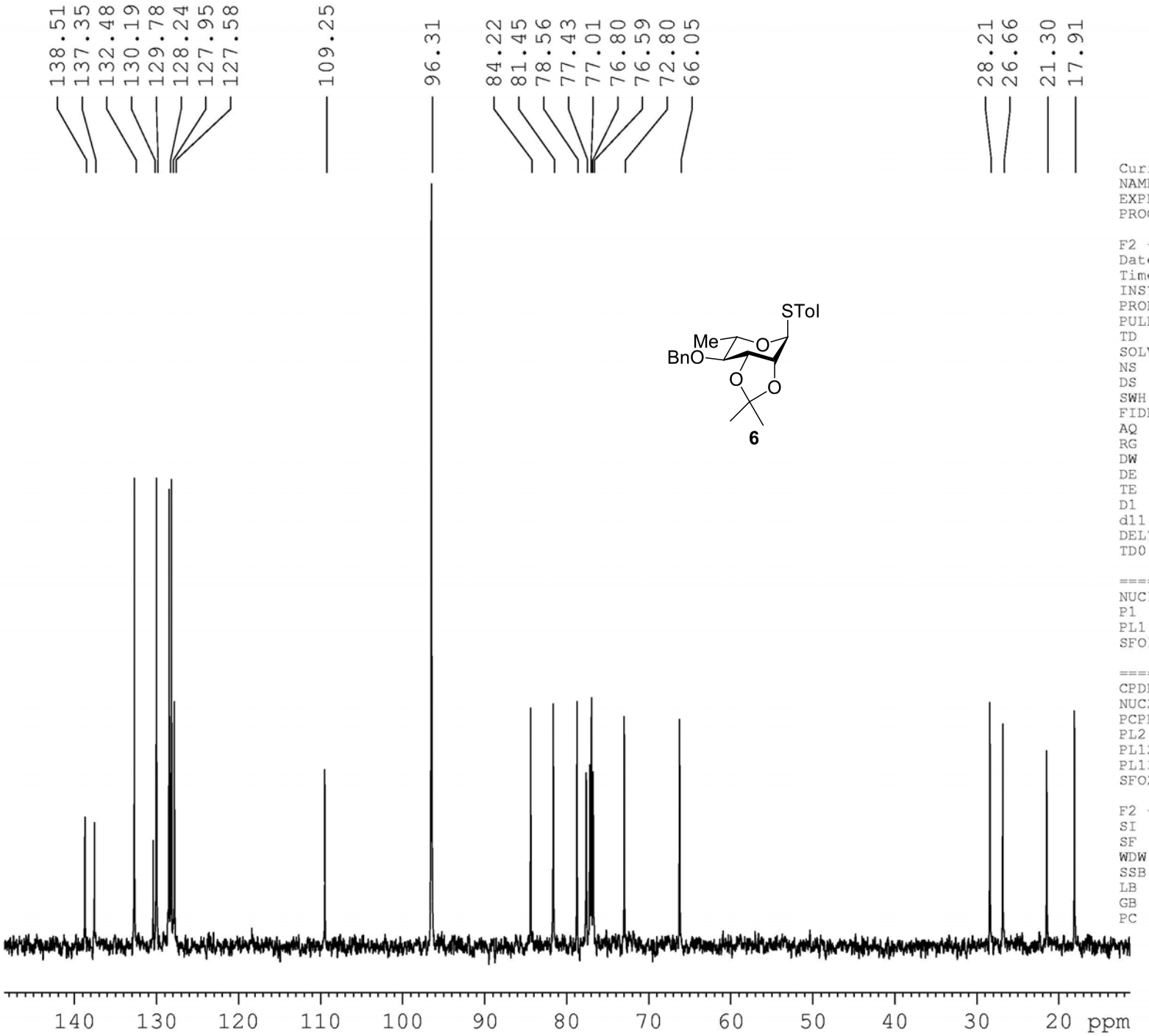
2.0
1.5
ppm

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Current Data Parameters
NAME                19.10.06
EXPNO                     20
PROCNO                     1

F2 - Acquisition Parameters
Date_                20061019
Time                    16.55
INSTRUM                 spect
PROBHD   5 mm QNP 1H/13
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                        512
DS                          0
SWH                 19531.250 Hz
FIDRES                0.298023 Hz
AQ                    1.6777716 sec
RG                          9
DW                     25.600 usec
DE                      6.00 usec
TE                     300.0 K
D1                  2.00000000 sec
d11                 0.03000000 sec
DELTA               1.89999998 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                      13C
P1                      8.70 usec
PL1                    -3.00 dB
SFO1               75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                  80.00 usec
PL2                    -1.00 dB
PL12                   17.00 dB
PL13                   21.00 dB
SFO2              300.1312005 MHz

F2 - Processing parameters
SI                        32768
SF                 75.4677262 MHz
WDW                        EM
SSB                         0
LB                      3.00 Hz
GB                          0
PC                      1.40
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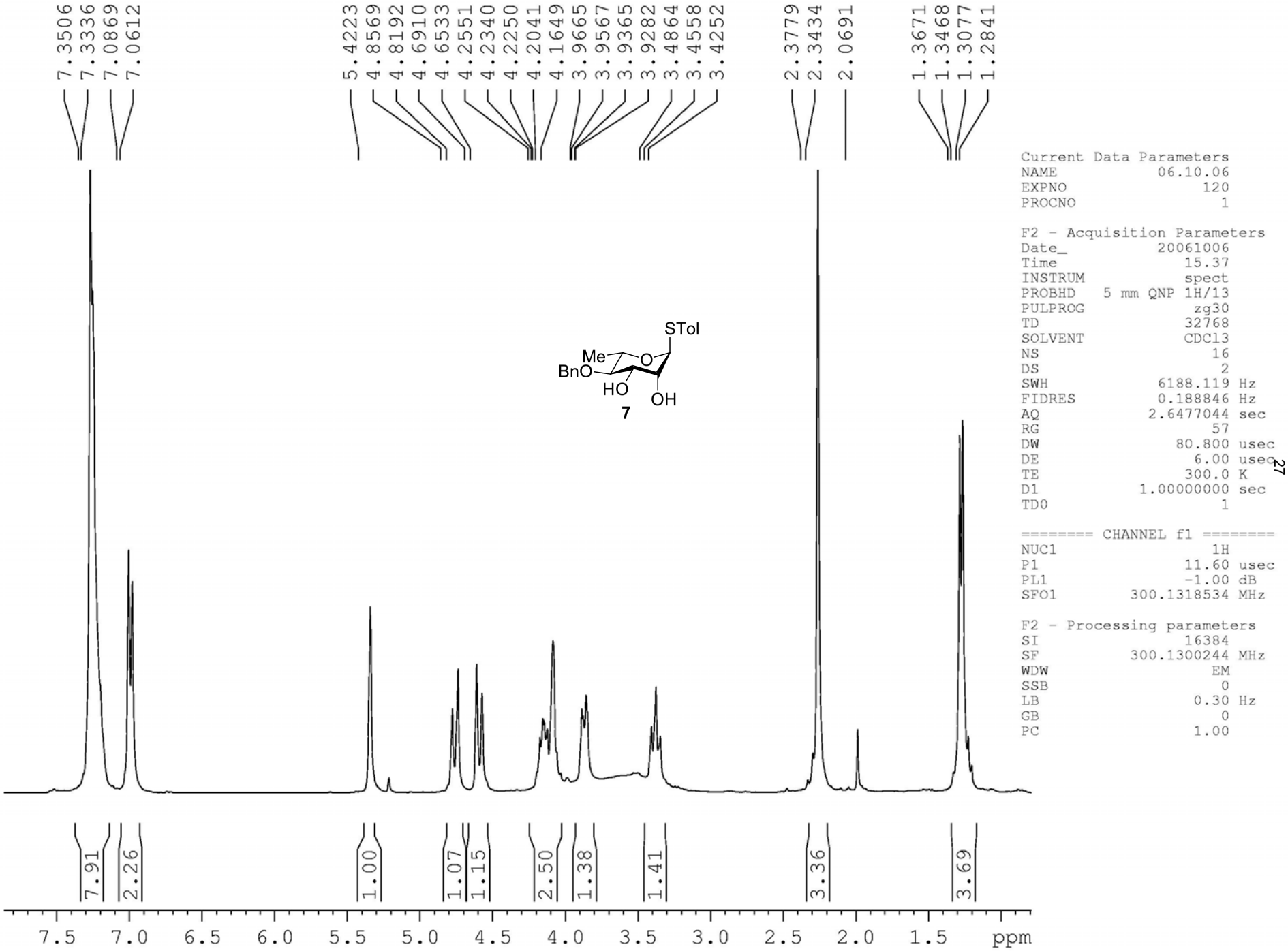
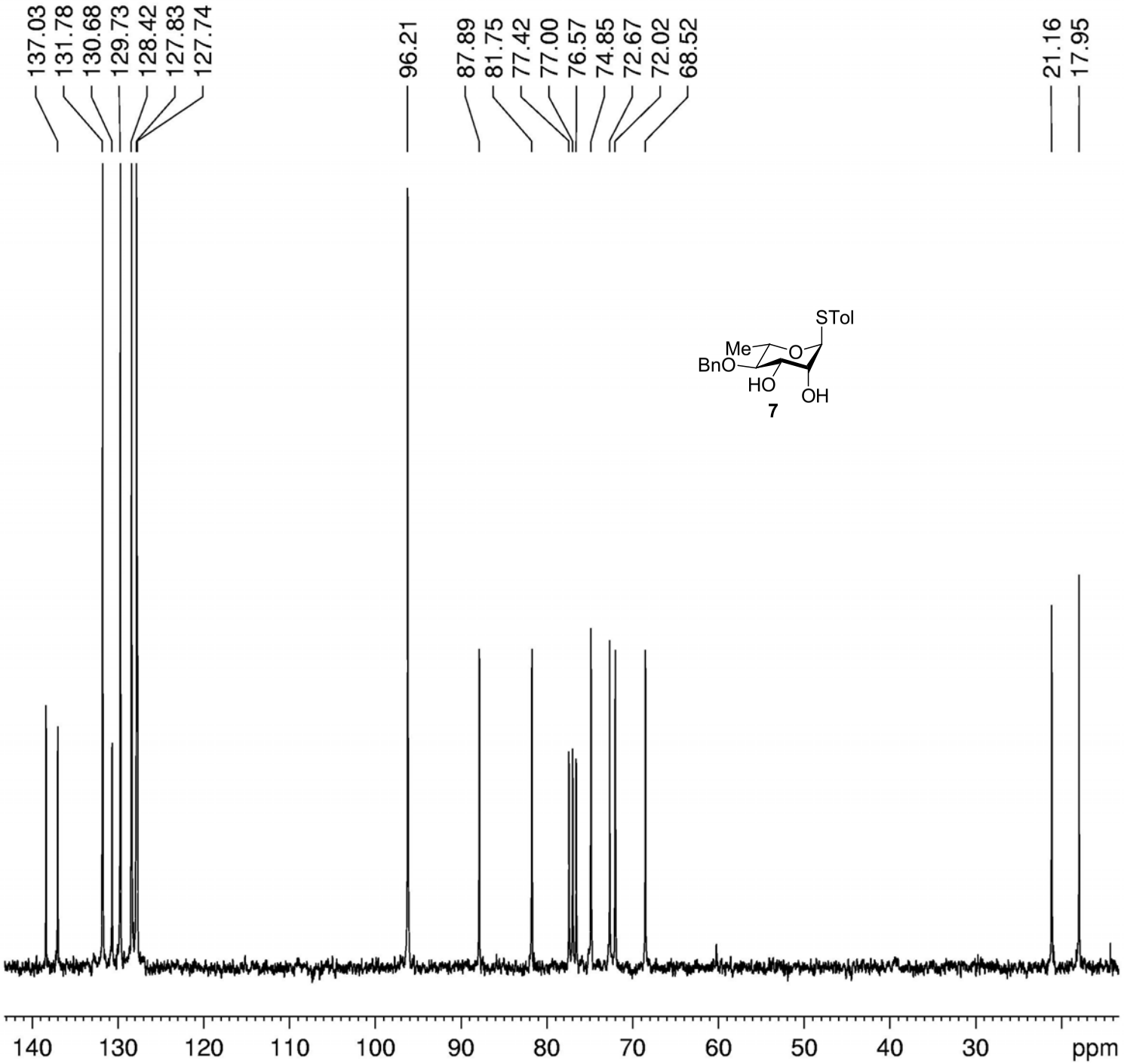


Current Data Parameters
NAME 06.10.06
EXPNO 120
PROCNO 1

F2 - Acquisition Parameters
Date_ 20061006
Time 15.37
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 57
DW 80.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 11.60 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 16384
SF 300.1300244 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

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Current Data Parameters
NAME                  10.10.06
EXPNO                      220
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20061010
Time                     22.20
INSTRUM                  spect
PROBHD    5 mm QNP 1H/13
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                        512
DS                          0
SWH                 19531.250 Hz
FIDRES                0.298023 Hz
AQ                    1.6777716 sec
RG                          9
DW                     25.600 usec
DE                      6.00 usec
TE                      300.0 K
D1                  2.00000000 sec
d11                 0.03000000 sec
DELTA               1.89999998 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                      13C
P1                     8.70 usec
PL1                   -3.00 dB
SFO1               75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                 80.00 usec
PL2                   -1.00 dB
PL12                  17.00 dB
PL13                  21.00 dB
SFO2              300.1312005 MHz

F2 - Processing parameters
SI                        32768
SF                 75.4677417 MHz
WDW                          EM
SSB                          0
LB                    3.00 Hz
GB                           0
PC                    1.00
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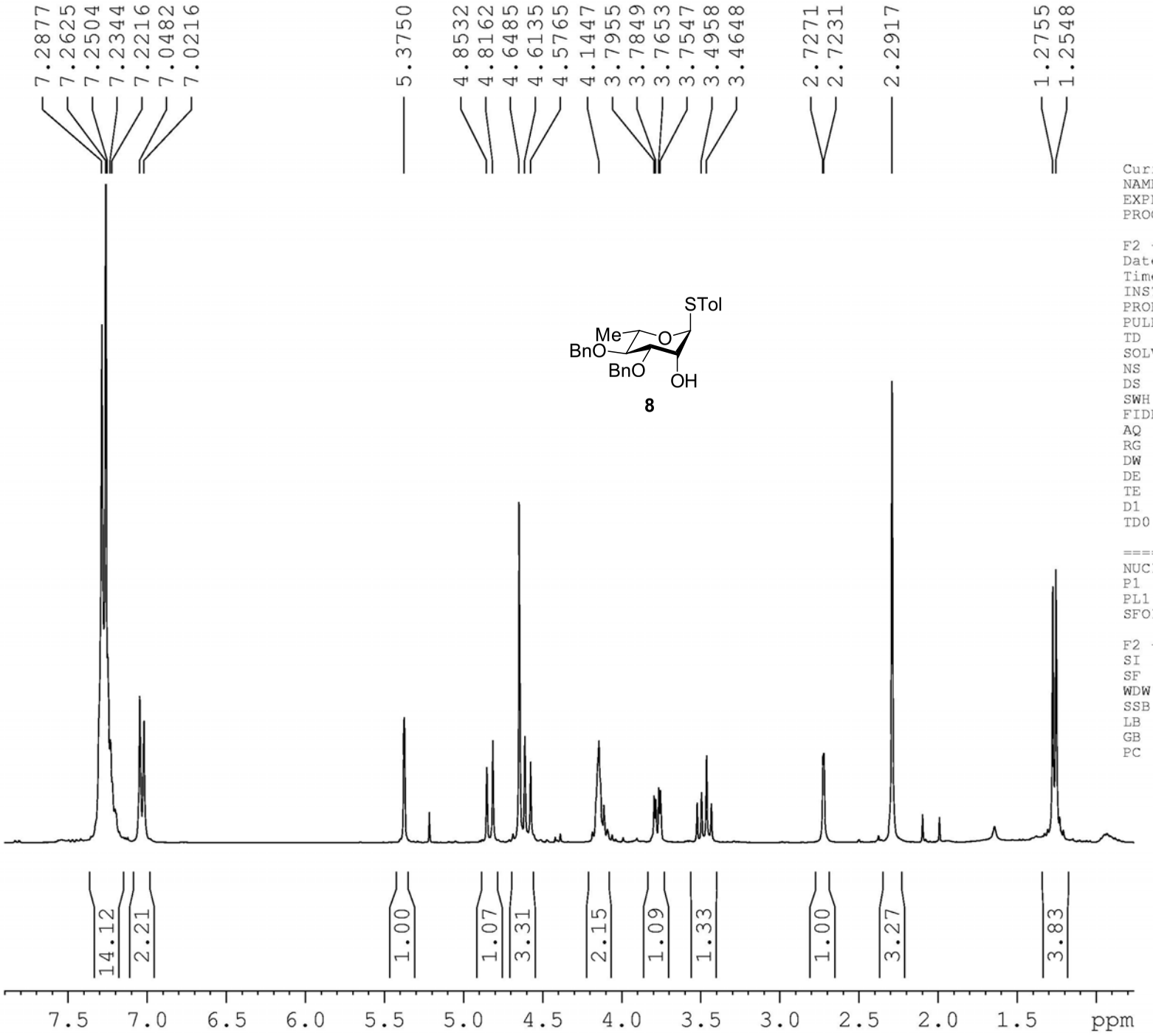


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Current Data Parameters
NAME                  09.10.06
EXPNO                       60
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20061009
Time                     16.51
INSTRUM                  spect
PROBHD    5 mm QNP 1H/13
PULPROG                  zg30
TD                      32768
SOLVENT                 CDCl3
NS                         16
DS                          2
SWH                6188.119 Hz
FIDRES            0.188846 Hz
AQ                2.6477044 sec
RG                       80.6
DW                   80.800 usec
DE                    6.00 usec
TE                    300.0 K
D1                1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                    11.60 usec
PL1                   -1.00 dB
SFO1           300.1318534 MHz

F2 - Processing parameters
SI                      16384
SF             300.1300000 MHz
WDW                        EM
SSB                         0
LB                    0.30 Hz
GB                          0
PC                       1.00
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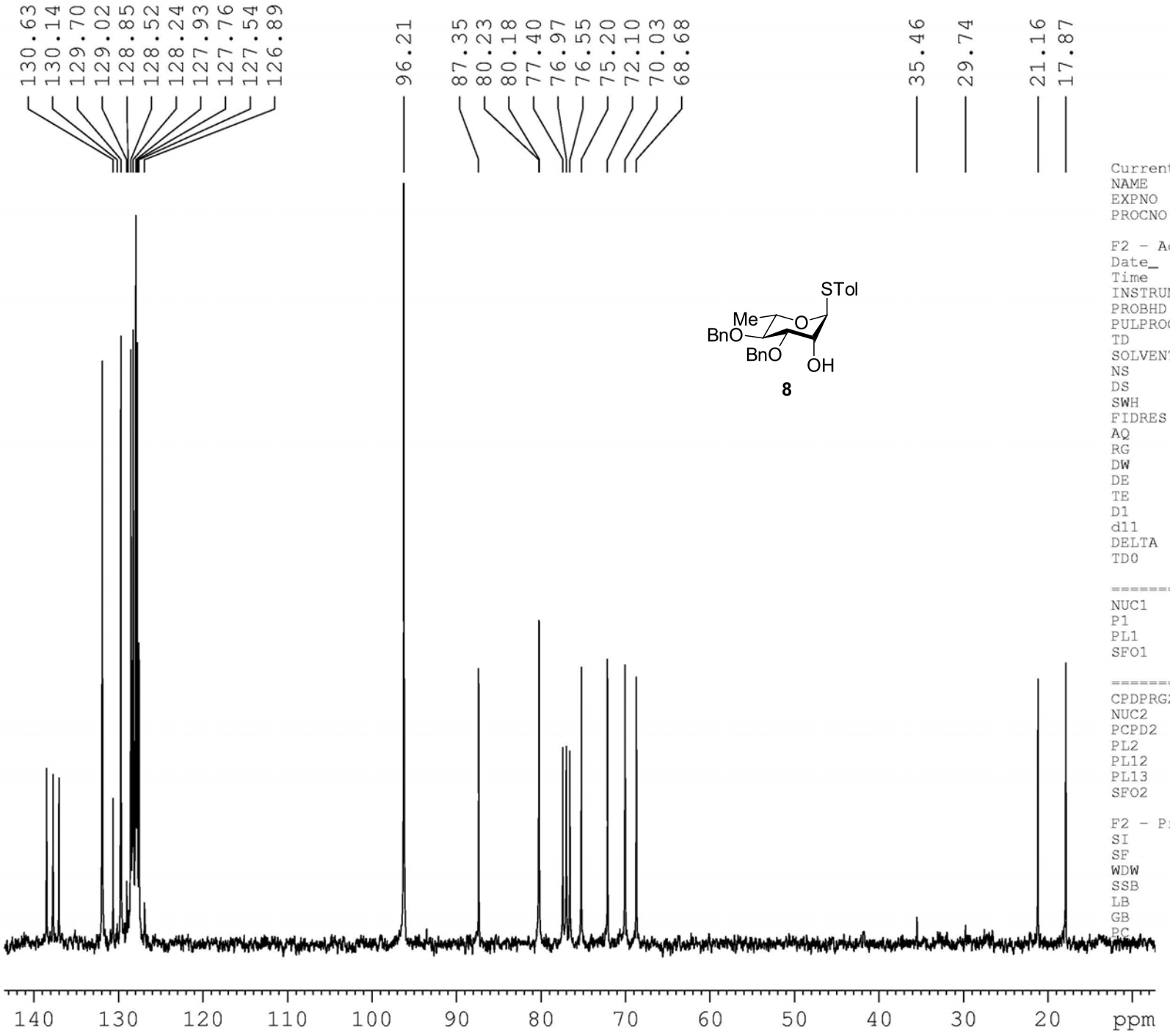
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80.23
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77.40
76.97
76.55
75.20
72.10
70.03
68.68
35.46
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17.87
STol
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BnO
BnO
OH
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Current Data Parameters
NAME 19.10.06
EXPNO 30
PROCNO 1
F2 - Acquisition Parameters
Date_ 20061019
Time 17.46
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 19531.250 Hz
FIDRES 0.298023 Hz
AQ 1.6777716 sec
RG 9
DW 25.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.70 usec
PL1 -3.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 17.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677484 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
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120
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80
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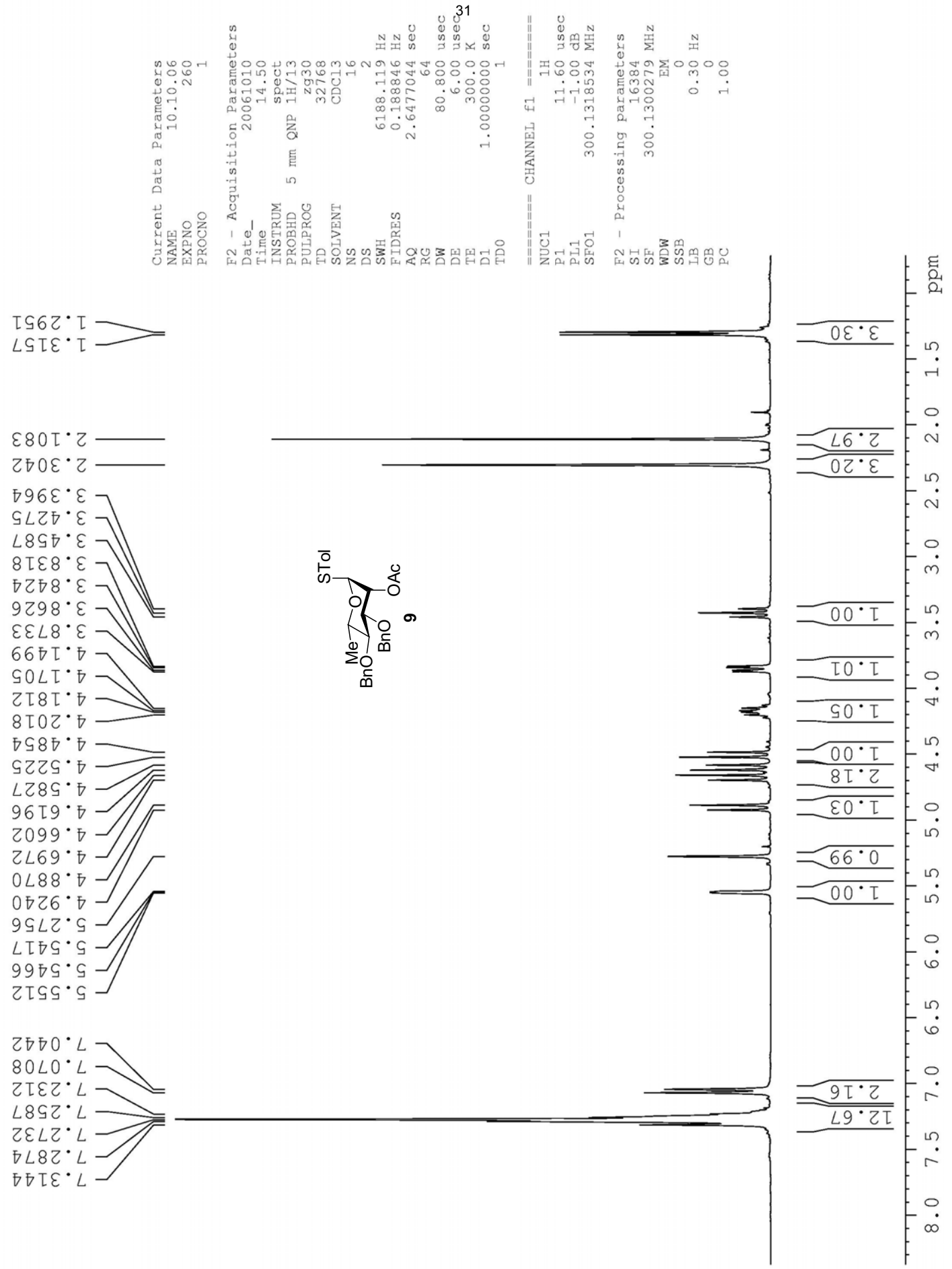
60
50
40
30
20
ppm


Current Data Parameters
NAME 10.10.06
EXPNO 260
PROCNO 1
F2 - Acquisition Parameters
Date_ 20061010
Time 14.50
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDC13
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 64
DW 80.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.60 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 16384
SF 300.1300279 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
STol
OAc
BnO
Me
BnO
9
1.2951
1.3157
2.1083
2.3042
3.3964
3.4275
3.4587
3.8318
3.8424
3.8626
3.8733
4.1499
4.1705
4.1812
4.2018
4.4854
4.5225
4.5827
4.6196
4.6602
4.6972
4.8870
4.9240
5.2756
5.5417
5.5466
5.5512
7.0442
7.0708
7.2312
7.2587
7.2732
7.2874
7.3144
3.30
2.97
3.20
1.00
1.01
1.05
1.00
2.18
1.03
0.99
1.00
2.16
12.67
8.0
7.5
7.0
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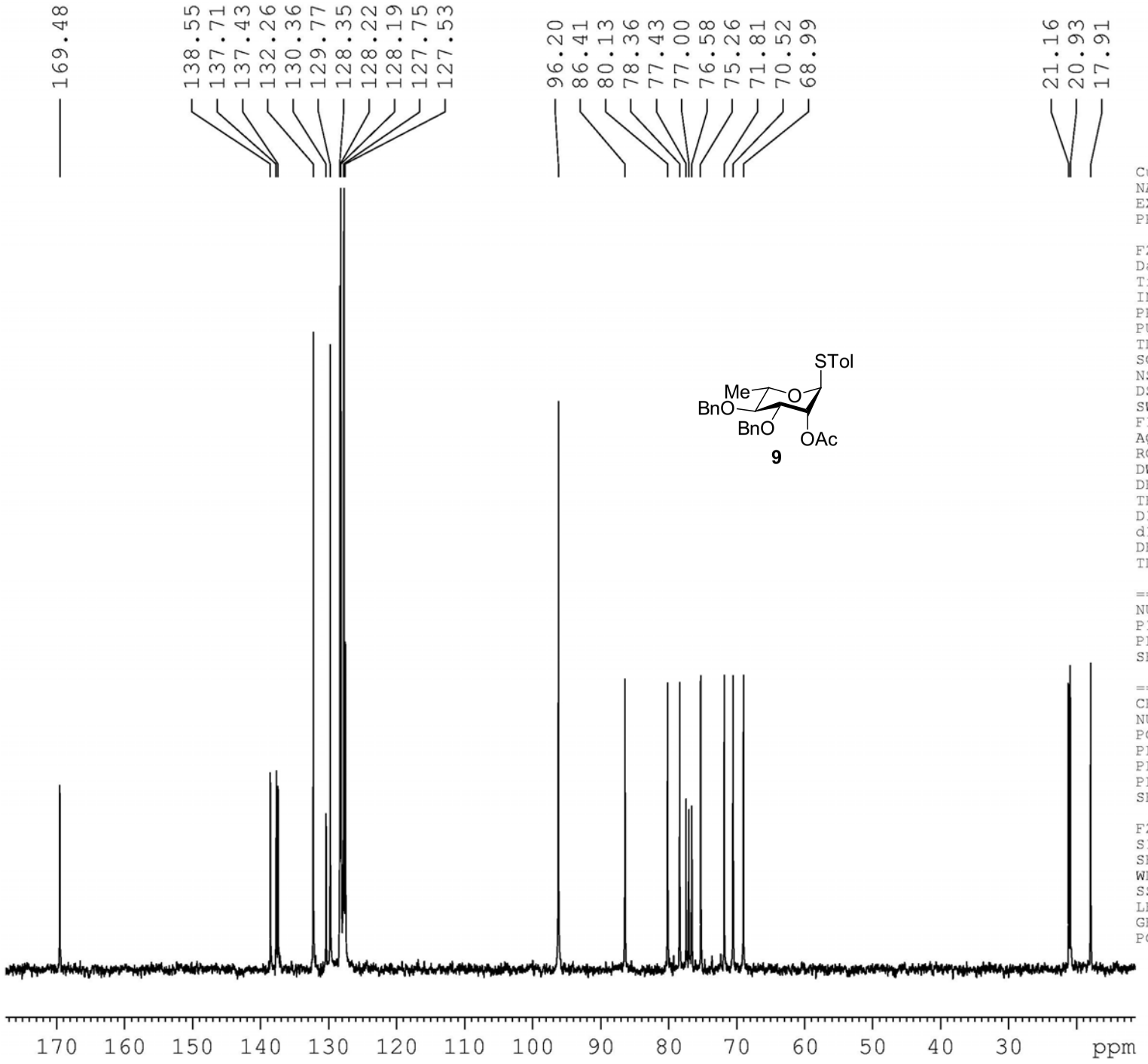
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm



Current Data Parameters

NAME	13.10.06
EXPNO	80
PROCNO	1

F2 - Acquisition Parameters

Date_	20061013
Time	18.59
INSTRUM	spect
PROBHD	5 mm QNP 1H/13
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	512
DS	0
SWH	19531.250 Hz
FIDRES	0.298023 Hz
AQ	1.6777716 sec
RG	9
DW	25.600 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

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

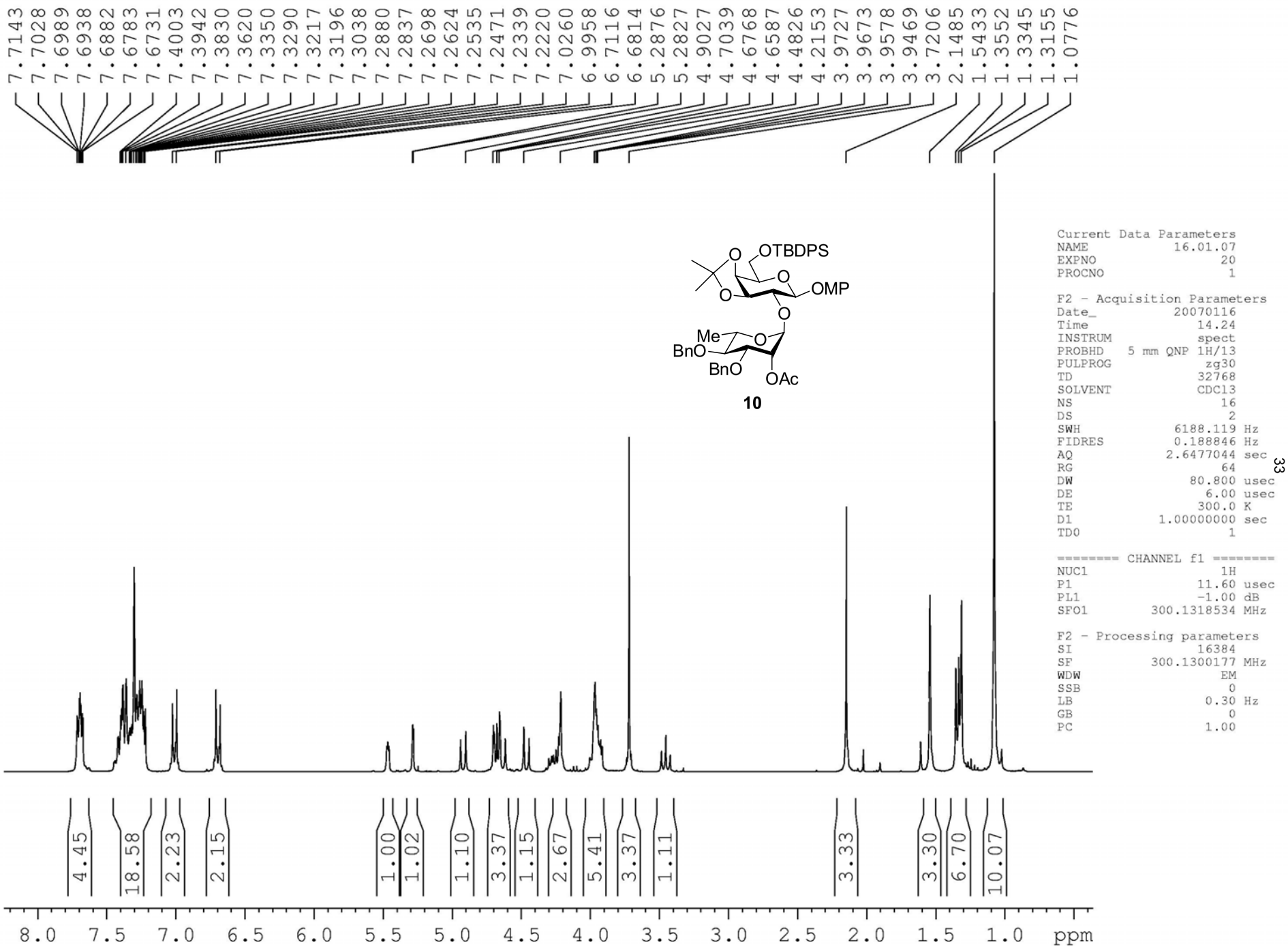
NUC1	13C
P1	8.70 usec
PL1	-3.00 dB
SFO1	75.4752953 MHz

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

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-1.00 dB
PL12	17.00 dB
PL13	21.00 dB
SFO2	300.1312005 MHz

F2 - Processing parameters

SI	32768
SF	75.4677521 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40


7.7143
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7.6731
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7.3942
7.3830
7.3620
7.3350
7.3290
7.3217
7.3196
7.3038
7.2880
7.2837
7.2698
7.2624
7.2535
7.2471
7.2339
7.2220
7.0260
6.9958
6.7116
6.6814
5.2876
5.2827
4.9027
4.7039
4.6768
4.6587
4.4826
4.2153
3.9727
3.9673
3.9578
3.9469
3.7206
2.1485
1.5433
1.3552
1.3345
1.3155
1.0776
OTBDPS
OMP
Me
BnO
BnO
OAc
10
Current Data Parameters
NAME 16.01.07
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070116
Time 14.24
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 64
DW 80.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.60 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 16384
SF 300.1300177 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
4.45
18.58
2.23
2.15
1.00
1.02
1.10
3.37
1.15
2.67
5.41
3.37
1.11
3.33
3.30
6.70
10.07
8.0
7.5
7.0
6.5
6.0
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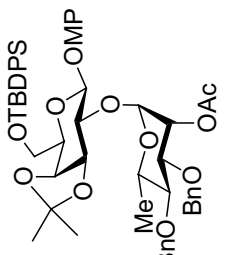
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm



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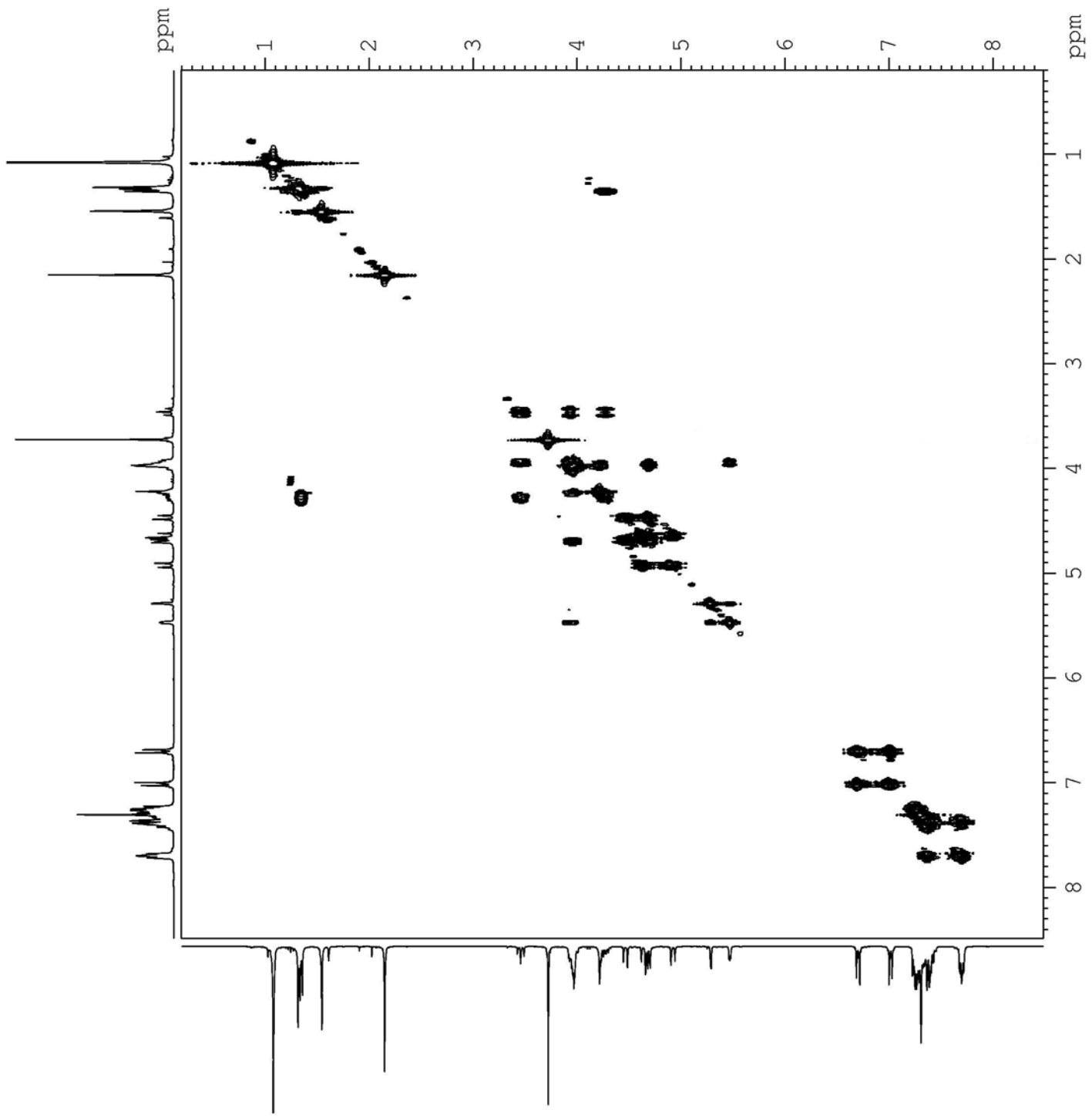
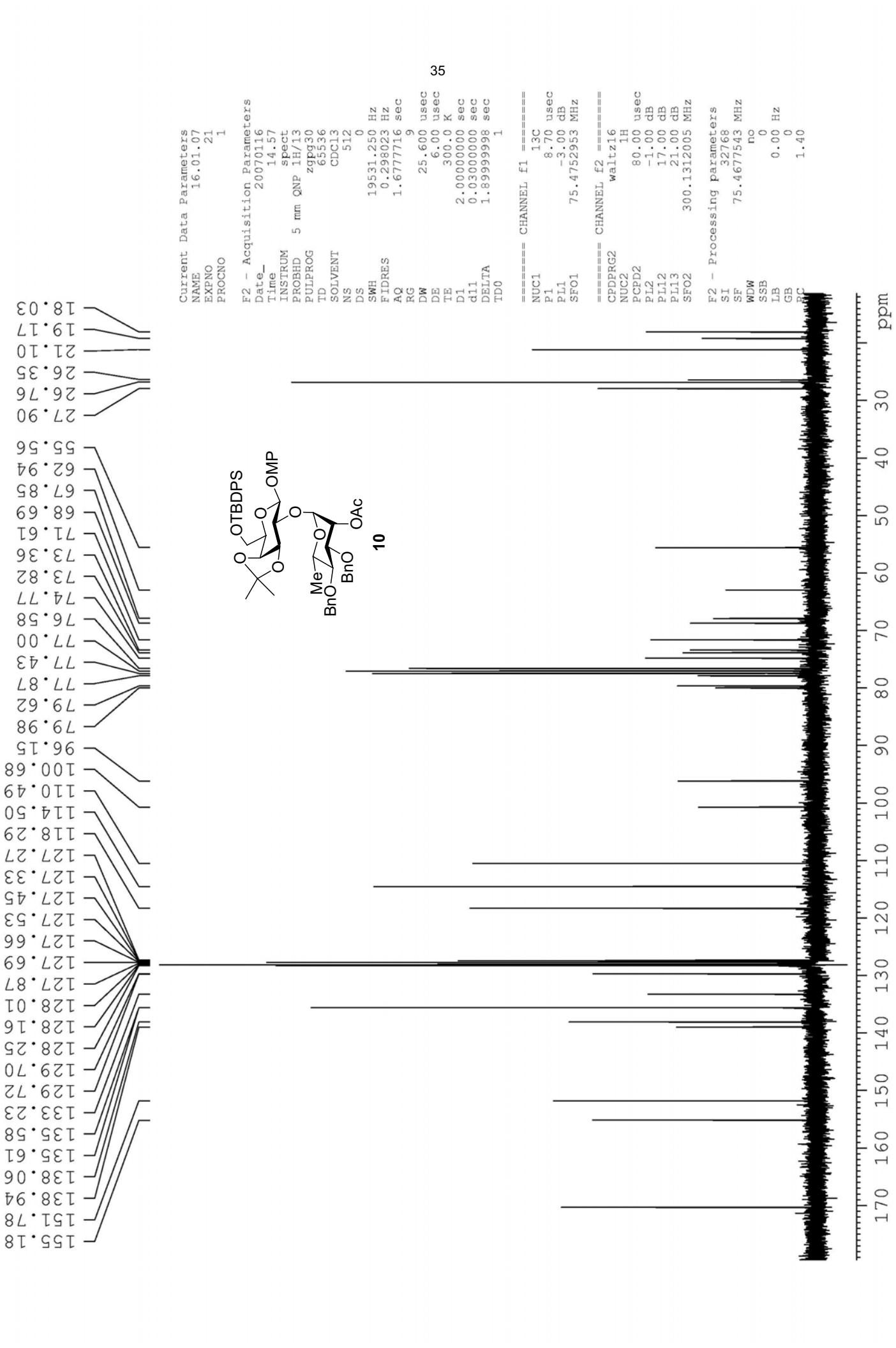
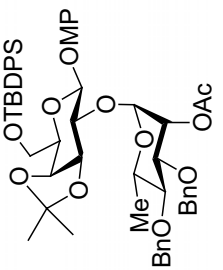
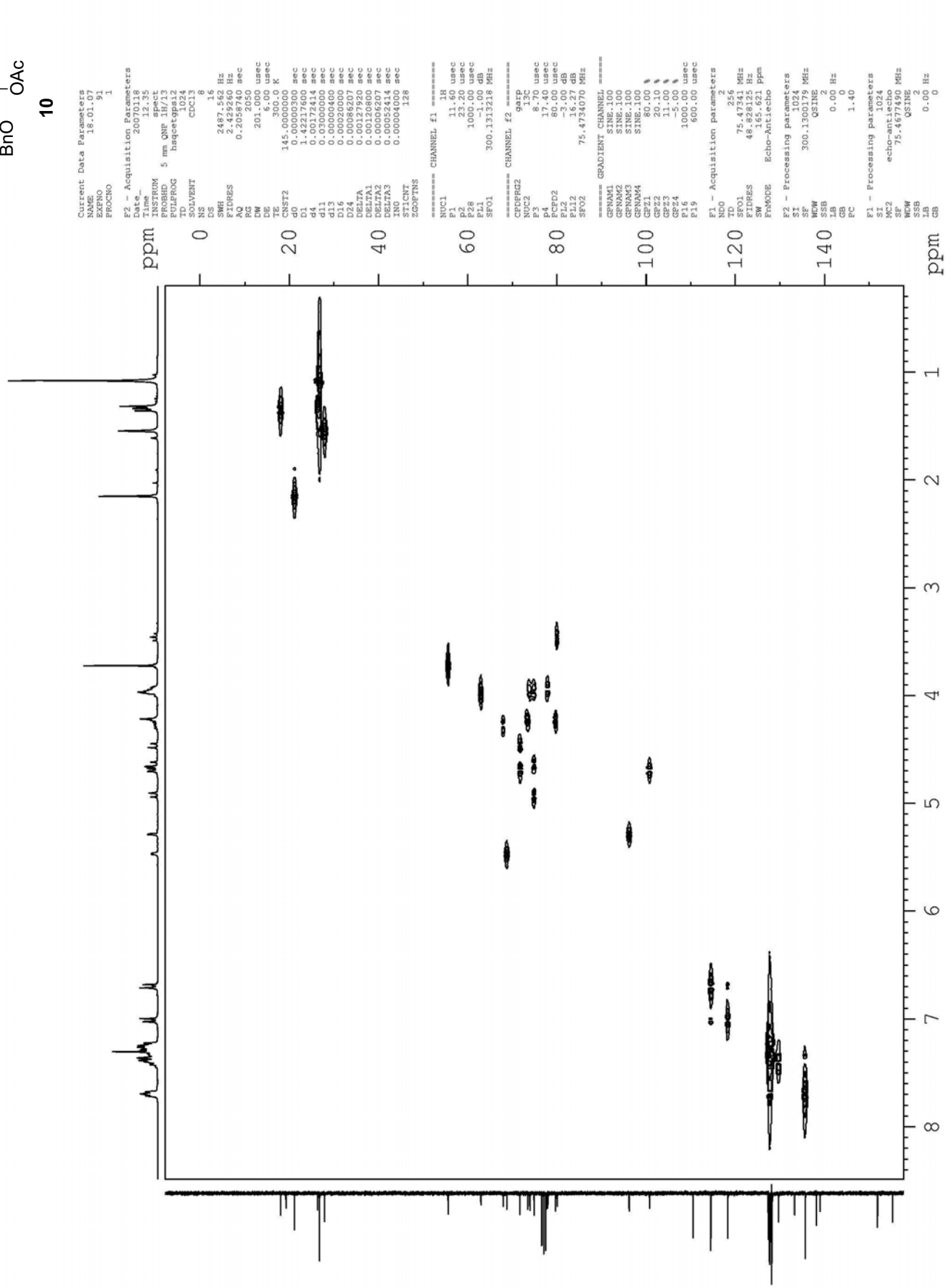


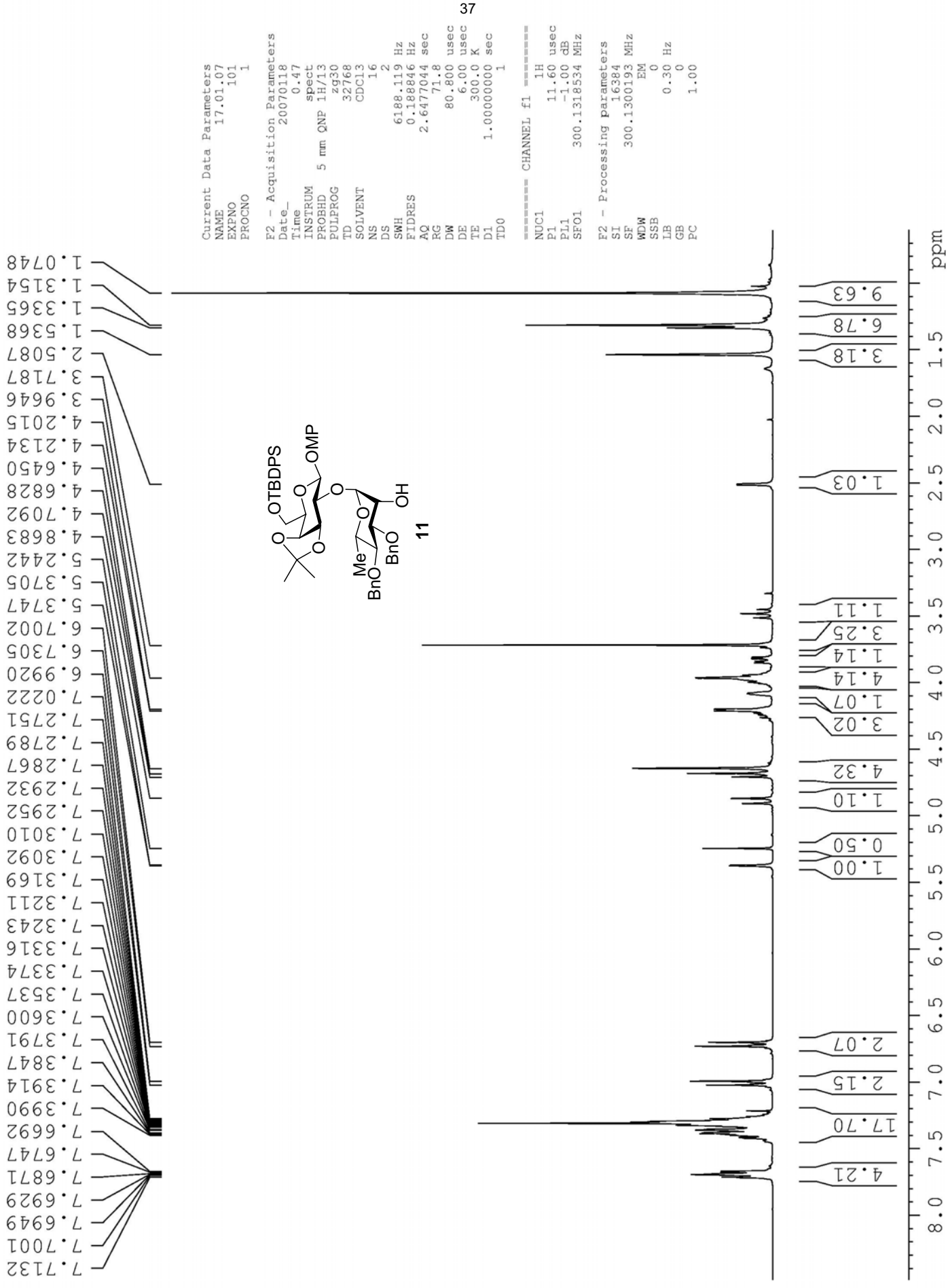

```
Current Data Parameters
NAME          16.01.07
EXPNO               22
PROCNO               1

F2 - Acquisition Parameters
Date_         20070116
Time_            14.58
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG     cosygpqf
TD              2048
SOLVENT         CDCl3
NS                 8
DS                 8
SWH          2487.562 Hz
FIDRES       1.214630 Hz
AQ           0.4116980 sec
RG              40.3
DW             201.000 usec
DE               6.00 usec
TE             300.0 K
d0           0.00000300 sec
D1           1.33083403 sec
d13          0.00000400 sec
D16          0.00020000 sec
IN0          0.00040200 sec

======== CHANNEL f1 ========
NUC1               1H
P0              11.60 usec
P1              11.60 usec
PL1             -1.00 dB
SFO1     300.1313205 MHz

====== GRADIENT CHANNEL =====
GPNAM1        SINE.100
GPNAM2        SINE.100
GPZ1             10.00 %
GPZ2             10.00 %
P16            1000.00 usec

F1 - Acquisition parameters
ND0                1
TD               128
SFO1     300.1313 MHz
FIDRES    19.434080 Hz
SW            8.288 ppm
FnMODE           QF

F2 - Processing parameters
SI              1024
SF       300.1300177 MHz
WDW           SINE
SSB              0
LB            0.00 Hz
GB               0
PC            1.40

F1 - Processing parameters
SI              1024
MC2               QF
SF       300.1300177 MHz
WDW           SINE
SSB              0
LB            0.00 Hz
GB               0
```

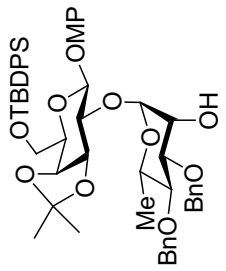

Current Data Parameters
NAME 16.01.07
EXPNO 21
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070116
Time 14.57
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 19531.250 Hz
FIDRES 0.298023 Hz
AQ 1.6777716 sec
RG 9
DW 25.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.70 usec
PL1 -3.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 17.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677543 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
OTBDPS
OMP
OAc
Me
BnO
BnO
10
155.18
151.78
138.94
138.06
135.61
135.58
133.23
129.72
129.70
128.25
128.16
128.01
127.87
127.69
127.66
127.53
127.45
127.33
127.27
118.29
114.50
110.49
100.68
96.15
79.98
79.62
77.87
77.43
77.00
76.58
74.77
73.82
73.36
71.61
68.69
67.85
62.94
55.56
27.90
26.76
26.35
21.10
19.17
18.03
ppm


OTBDPS
OMP
Me
BnO
BnO
OAc

10


Current Data Parameters
NAME 18.01.07
EXPNO 91
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070118
Time 12.35
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG hsqcetgpsi2
TD 1024
SOLVENT CDCl3
NS 8
DS 16
SWH 2487.562 Hz
FIDRES 2.429260 Hz
AQ 0.2058740 sec
RG 2050
DW 201.000 usec
DE 6.00 usec
TE 300.0 K
CNST2 145.0000000
d0 0.00000300 sec
D1 1.42217600 sec
d4 0.00172414 sec
d11 0.03000000 sec
d13 0.00000400 sec
D16 0.00020000 sec
D24 0.00086207 sec
DELTA 0.00127920 sec
DELTA1 0.00128000 sec
DELTA2 0.00006207 sec
DELTA3 0.00052414 sec
INO 0.00004000 sec
ST1CNT 128
ZGOPTNS
======== CHANNEL f1 ========
NUC1 1H
P1 11.60 usec
P2 23.20 usec
P28 1000.00 usec
PL1 0.00 dB
SFO1 300.1313218 MHz
======== CHANNEL f2 ========
CPDPRG2 garp
NUC2 13C
P3 8.70 usec
P4 17.40 usec
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 16.27 dB
SFO2 75.4734070 MHz
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPNAM3 SINE.100
GPNAM4 SINE.100
GPZ1 80.00 %
GPZ2 20.10 %
GPZ3 11.00 %
GPZ4 -5.00 %
P16 1000.00 usec
P19 600.00 usec
F1 - Acquisition parameters
NDO 2
TD 256
SFO1 75.47341 MHz
FIDRES 48.828125 Hz
SW 165.621 ppm
FnMODE Echo-Antiecho
F2 - Processing parameters
SI 1024
SF 300.1300173 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.40
F1 - Processing parameters
SI 1024
MC2 echo-antiecho
SF 75.4677490 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
ppm
ppm


Current Data Parameters
NAME 17.01.07
EXPNO 101
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070118
Time 0.47
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 71.8
DW 80.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.60 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 16384
SF 300.1300193 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OTBDPS
OMP
OH
BnO
Me
BnO
11
1.0748
1.3154
1.3365
1.5368
2.5087
3.7187
3.9646
4.2015
4.2134
4.6450
4.6828
4.7092
4.8683
5.2442
5.3705
5.3747
6.7002
6.7305
6.9920
7.0222
7.2751
7.2789
7.2867
7.2932
7.2952
7.3010
7.3092
7.3169
7.3211
7.3243
7.3316
7.3374
7.3537
7.3600
7.3791
7.3847
7.3914
7.3990
7.6692
7.6747
7.6871
7.6929
7.6949
7.7001
7.7132
9.63
6.78
3.18
1.03
1.11
3.25
1.14
4.14
1.07
3.02
4.32
1.10
1.00
0.50
2.07
2.15
17.70
4.21
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm



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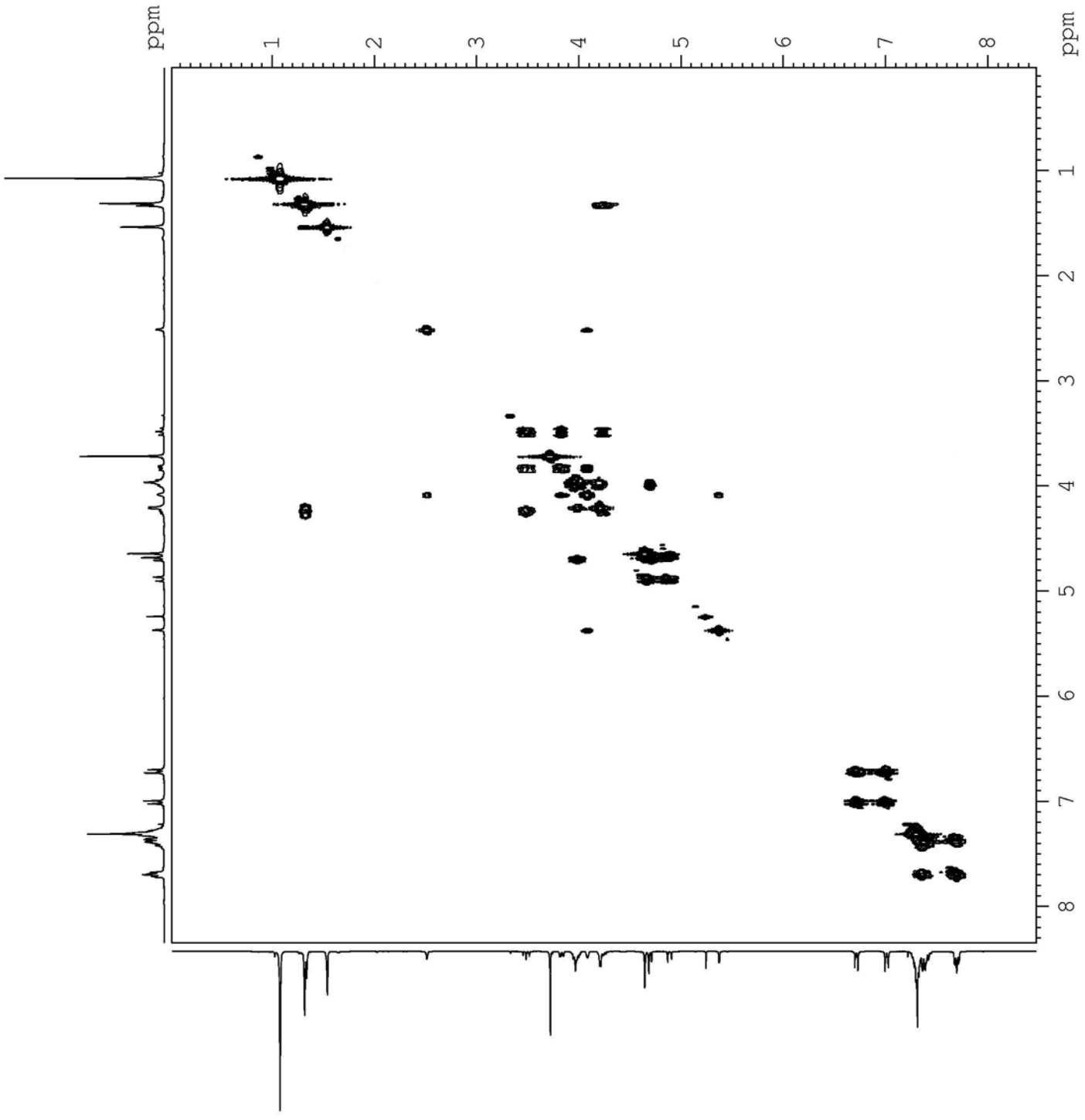
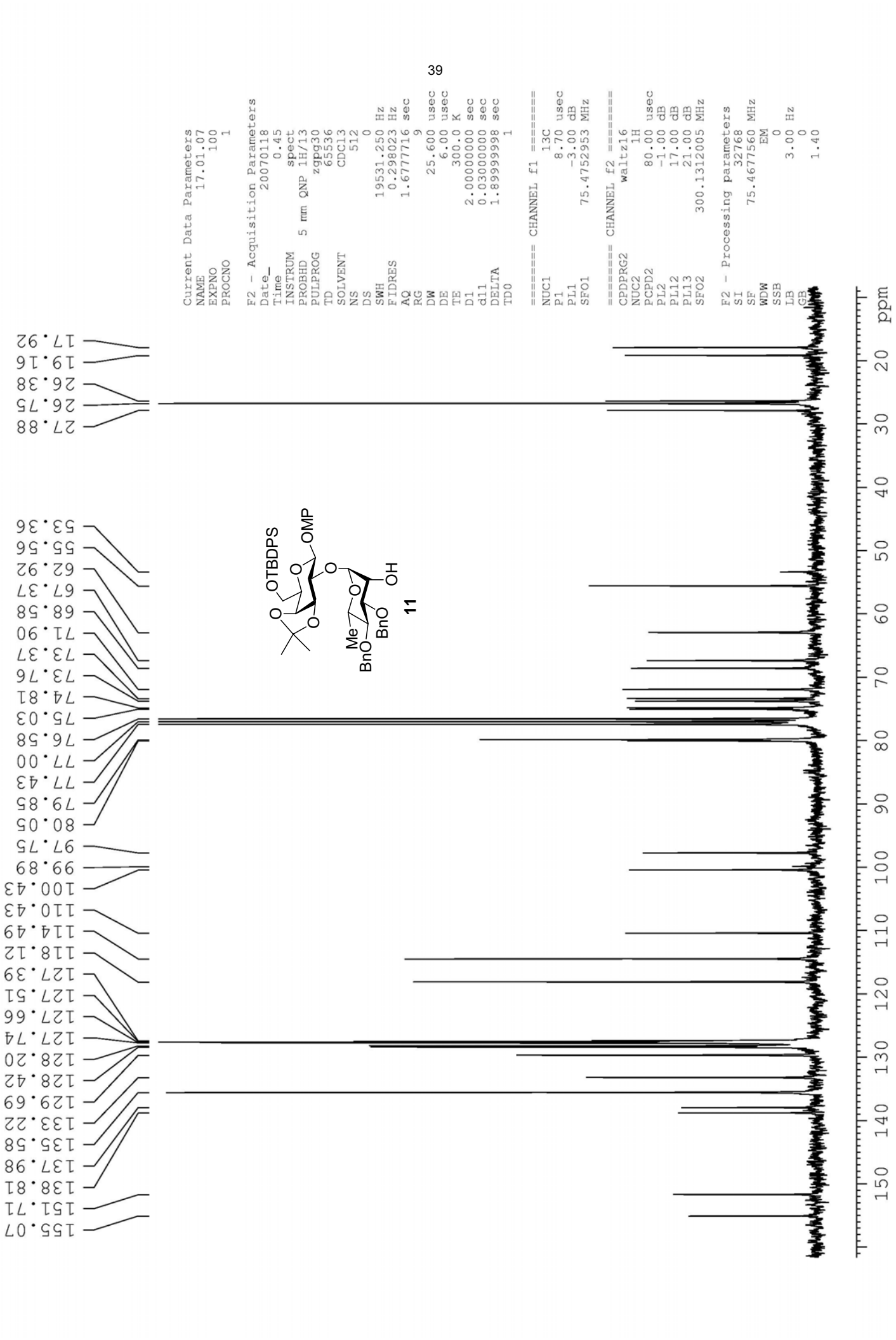
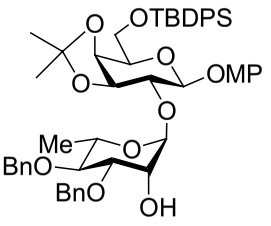
```
Current Data Parameters
NAME                  19.01.07
EXPNO                       21
PROCNO                       1

F2 - Acquisition Parameters
Date_              20070119
Time_                 17.00
INSTRUM                spect
PROBHD    5 mm QNP 1H/13
PULPROG          cosygpqf
TD                   2048
SOLVENT              CDCl3
NS                       8
DS                       8
SWH               2538.071 Hz
FIDRES            1.239293 Hz
AQ                0.4035060 sec
RG                      36
DW                197.000 usec
DE                  6.00 usec
TE                  300.0 K
d0                0.00000300 sec
D1                1.33902597 sec
d13               0.00000400 sec
D16               0.00020000 sec
IN0               0.00039400 sec

======== CHANNEL f1 ========
NUC1                    1H
P0                  11.60 usec
P1                  11.60 usec
PL1                 -1.00 dB
SFO1          300.1312946 MHz

====== GRADIENT CHANNEL =====
GPNAM1           SINE.100
GPNAM2           SINE.100
GPZ1                10.00 %
GPZ2                10.00 %
P16               1000.00 usec

F1 - Acquisition parameters
ND0                      1
TD                     128
SFO1          300.1313 MHz
FIDRES         19.828680 Hz
SW                 8.457 ppm
FnMODE                  QF

F2 - Processing parameters
SI                    1024
SF            300.1300197 MHz
WDW                  SINE
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.40

F1 - Processing parameters
SI                    1024
MC2                     QF
SF            300.1300197 MHz
WDW                  SINE
SSB                     0
LB                   0.00 Hz
GB                      0
```




Current Data Parameters
NAME 17.01.07
EXPNO 100
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070118
Time 0.45
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 19531.250 Hz
FIDRES 0.298023 Hz
AQ 1.6777716 sec
RG 9
DW 25.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.70 usec
PL1 -3.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 17.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677560 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
1.40
OTBDPS
OMP
OH
BnO
Me
BnO
11
ppm



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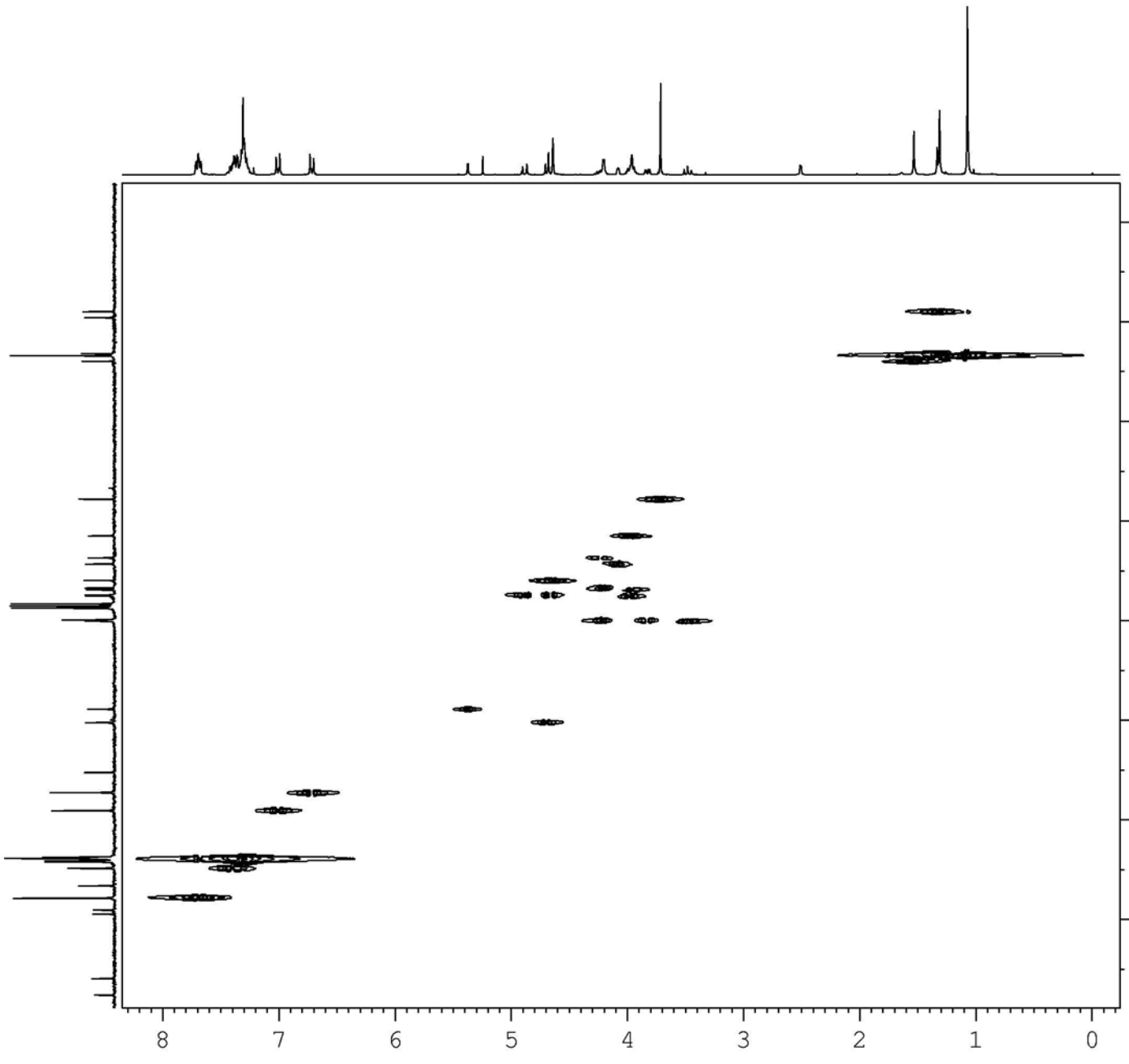
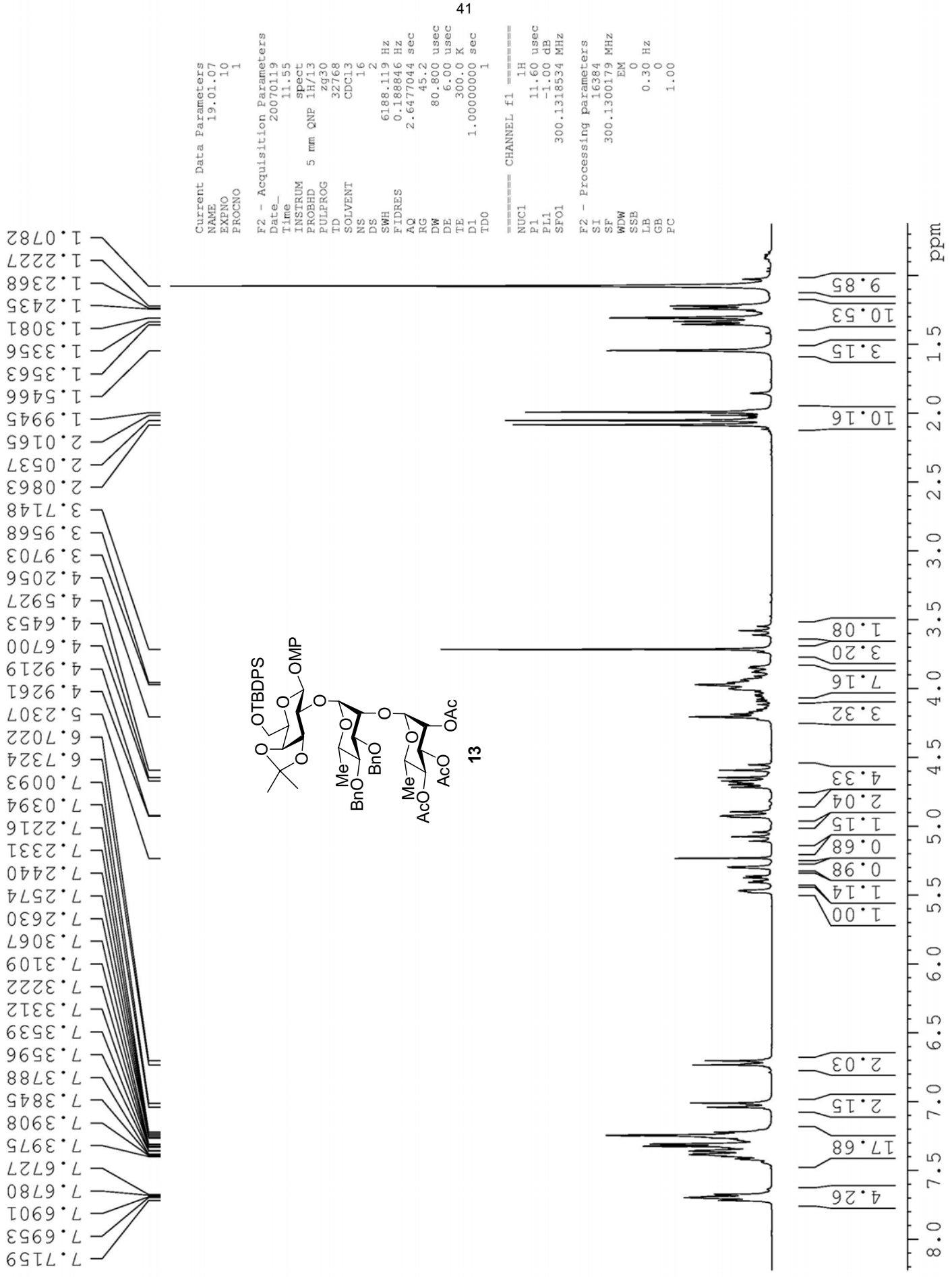
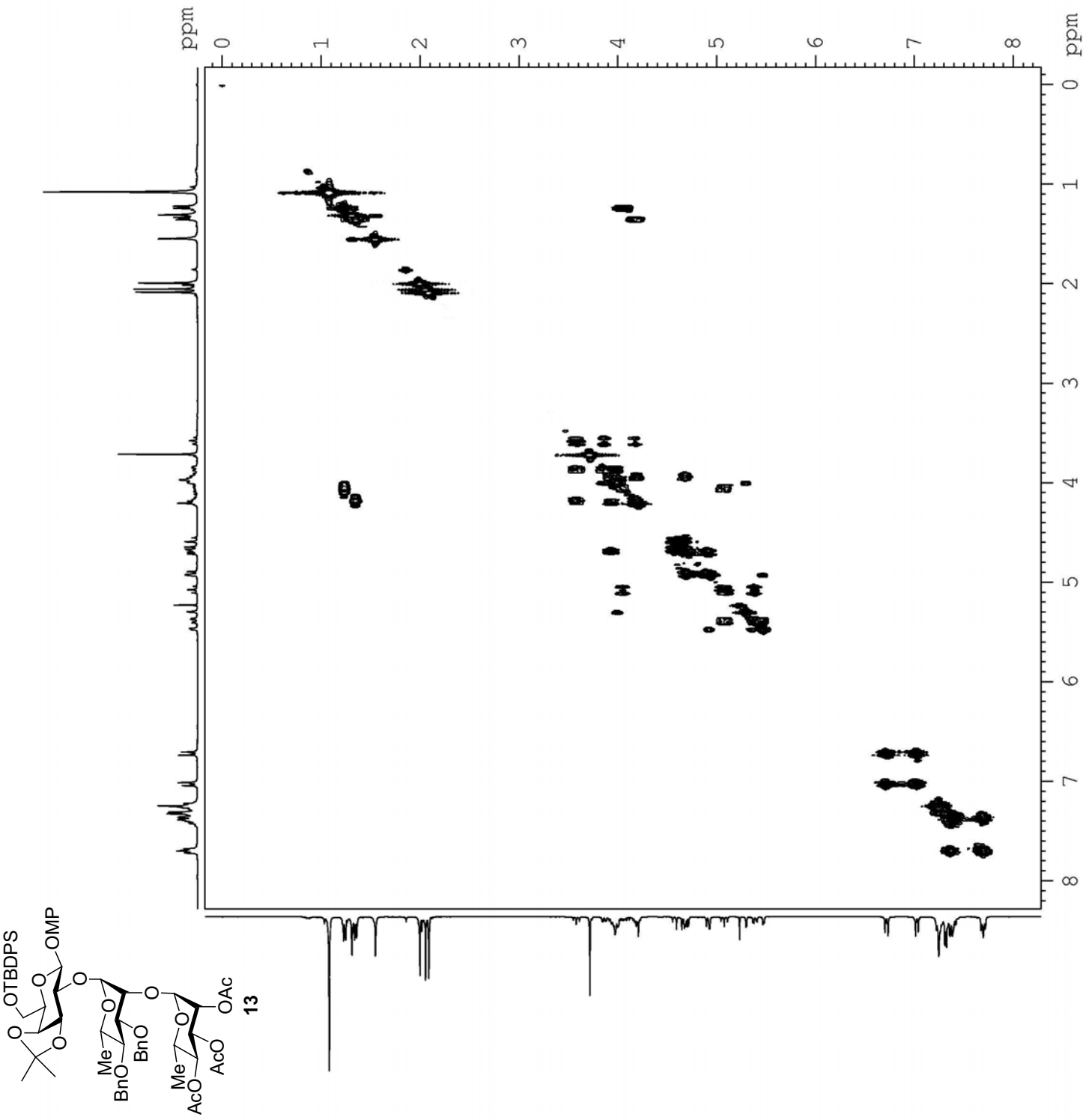
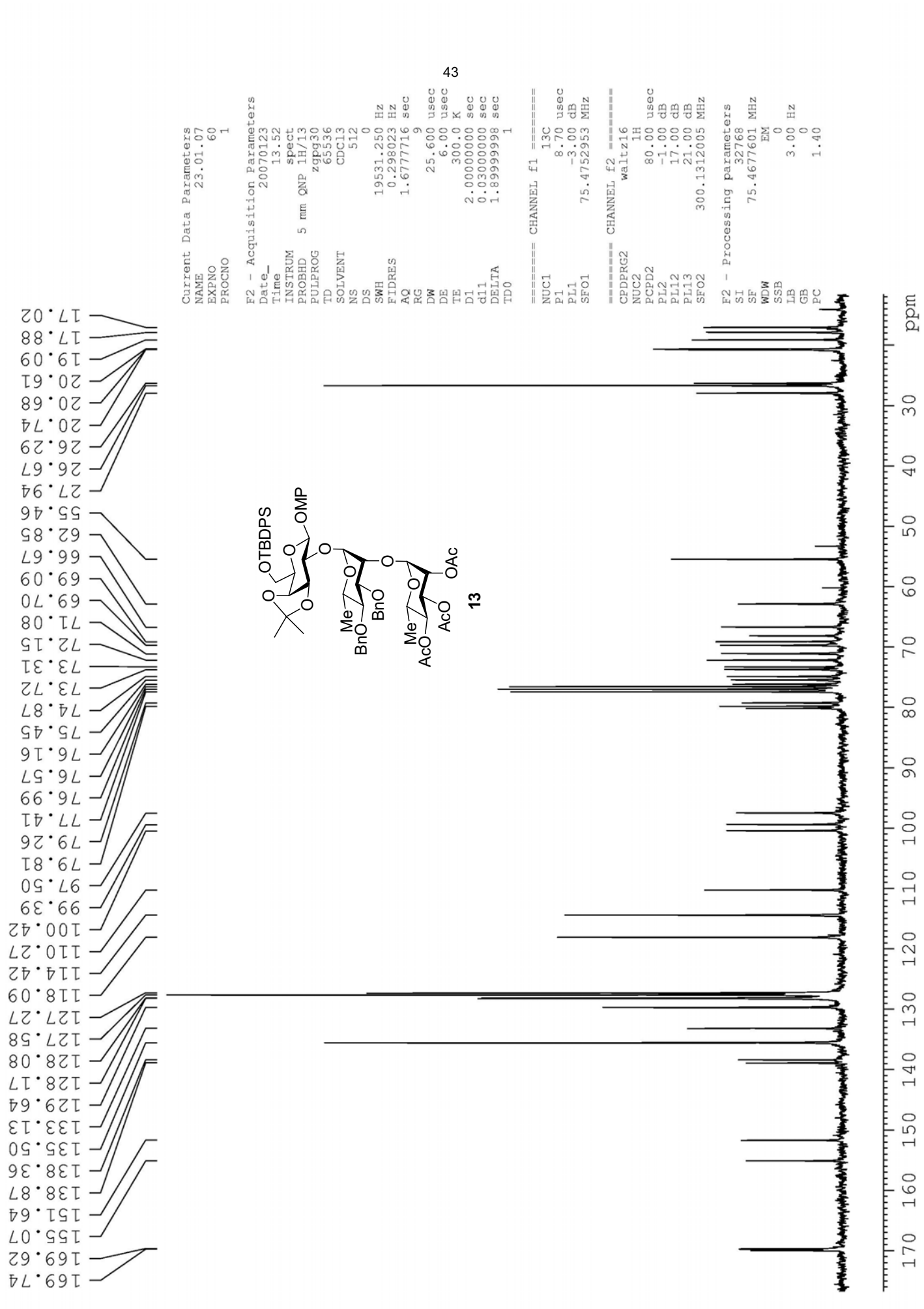
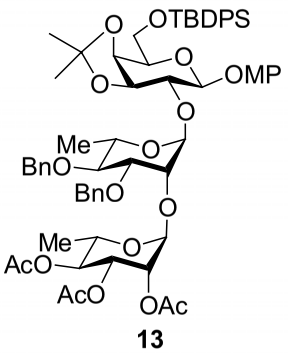



```
Current Data Parameters
NAME                 17.01.07
EXPNO                     102
PROCNO                      1

F2 - Acquisition Parameters
Date_              20070118
Time                   0.49
INSTRUM               spect
PROBHD    5 mm QNP 1H/13
PULPROG       hsqcetgpsi2
TD                      1024
SOLVENT              CDCl3
NS                         8
DS                        16
SWH                2577.320 Hz
FIDRES             2.516914 Hz
AQ                 0.1987060 sec
RG                      2050
DW                  194.000 usec
DE                    6.00 usec
TE                   300.0 K
CNST2             145.0000000
d0                 0.00000300 sec
D1                 1.42934406 sec
d4                 0.00172414 sec
d11                0.03000000 sec
d13                0.00000400 sec
D16                0.00020000 sec
D24                0.00086207 sec
DELTA              0.00127920 sec
DELTA1             0.00120800 sec
DELTA2             0.00006207 sec
DELTA3             0.00052414 sec
IN0                0.00004000 sec
ST1CNT                   128
ZGOPTNS

======== CHANNEL f1 ========
NUC1                       1H
P1                      11.60 usec
p2                      23.20 usec
P28                   1000.00 usec
PL1                     -1.00 dB
SFO1            300.1312363 MHz

======== CHANNEL f2 ========
CPDPRG2                 garp
NUC2                      13C
P3                       8.70 usec
p4                      17.40 usec
PCPD2                   80.00 usec
PL2                     -3.00 dB
PL12                    16.27 dB
SFO2             75.4734070 MHz

====== GRADIENT CHANNEL =====
GPNAM1               SINE.100
GPNAM2               SINE.100
GPNAM3               SINE.100
GPNAM4               SINE.100
GPZ1                    80.00 %
GPZ2                    20.10 %
GPZ3                    11.00 %
GPZ4                    -5.00 %
P16                   1000.00 usec
P19                    600.00 usec

F1 - Acquisition parameters
ND0                         2
TD                        256
SFO1             75.47341 MHz
FIDRES           48.828125 Hz
SW                  165.621 ppm
FnMODE       Echo-Antiecho

F2 - Processing parameters
SI                       1024
SF              300.1300193 MHz
WDW                     QSINE
SSB                         2
LB                       0.00 Hz
GB                          0
PC                       1.40

F1 - Processing parameters
SI                       1024
MC2             echo-antiecho
SF               75.4677490 MHz
WDW                     QSINE
SSB                         2
LB                       0.00 Hz
GB                          0
```

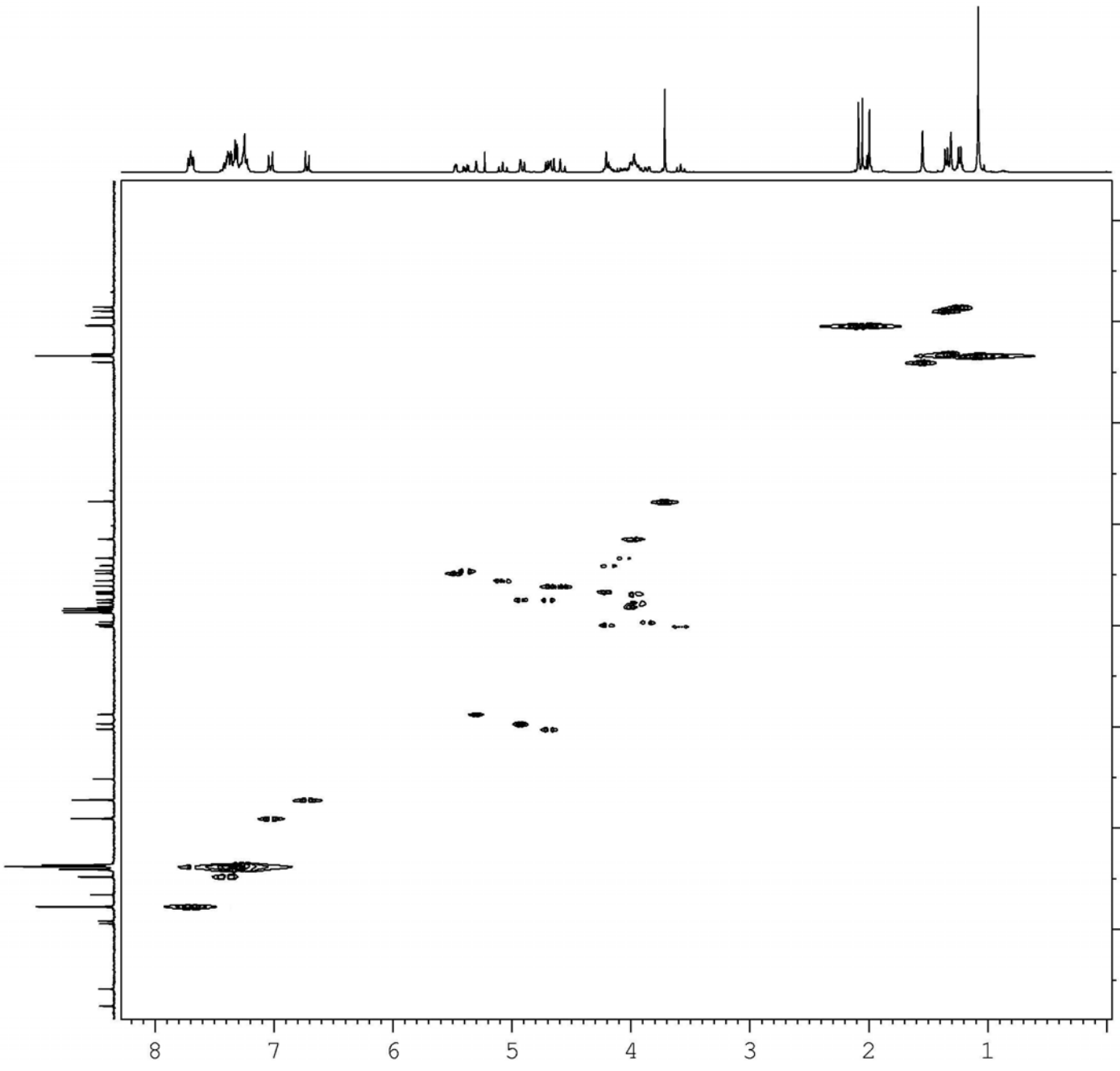

Current Data Parameters
NAME 19.01.07
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070119
Time 11.55
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 45.2
DW 80.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.60 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 16384
SF 300.1300179 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OTBDPS
OMP
Me
BnO
BnO
Me
AcO
AcO
OAc
13
7.7159
7.6953
7.6901
7.6780
7.6727
7.3975
7.3908
7.3845
7.3788
7.3596
7.3539
7.3312
7.3222
7.3109
7.3067
7.2630
7.2574
7.2440
7.2331
7.2216
7.0394
7.0093
6.7324
6.7022
5.2307
4.9261
4.9219
4.6700
4.6453
4.5927
4.2056
3.9703
3.9568
3.7148
2.0863
2.0537
2.0165
1.9945
1.5466
1.3563
1.3356
1.3081
1.2435
1.2368
1.2227
1.0782
4.26
17.68
2.15
2.03
1.00
1.14
0.98
0.68
1.15
2.04
4.33
3.32
7.16
3.20
1.08
10.16
3.15
10.53
9.85
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm


OTBDPS
OMP
Me
BnO
BnO
Me
AcO
AcO
OAc
13
ppm
0
1
2
3
4
5
6
7
8
ppm
Current Data Parameters
NAME 19.01.07
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070119
Time_ 11.57
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG cosygpqf
TD 2048
SOLVENT CDCl3
NS 8
DS 8
SWH 2538.071 Hz
FIDRES 1.239293 Hz
AQ 0.4035060 sec
RG 25.4
DW 197.000 usec
DE 6.00 usec
TE 300.0 K
d0 0.00000300 sec
D1 1.33902597 sec
d13 0.00000400 sec
D16 0.00020000 sec
IN0 0.00039400 sec
======== CHANNEL f1 ========
NUC1 1H
P0 11.60 usec
P1 11.60 usec
PL1 -1.00 dB
SFO1 300.1312344 MHz
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPZ1 10.00 %
GPZ2 10.00 %
P16 1000.00 usec
F1 - Acquisition parameters
ND0 1
TD 128
SFO1 300.1312 MHz
FIDRES 19.828680 Hz
SW 8.457 ppm
FnMODE QF
F2 - Processing parameters
SI 1024
SF 300.1300179 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
F1 - Processing parameters
SI 1024
MC2 QF
SF 300.1300179 MHz
WDW SINE
SSB 0
LB 0.00 Hz
GB 0

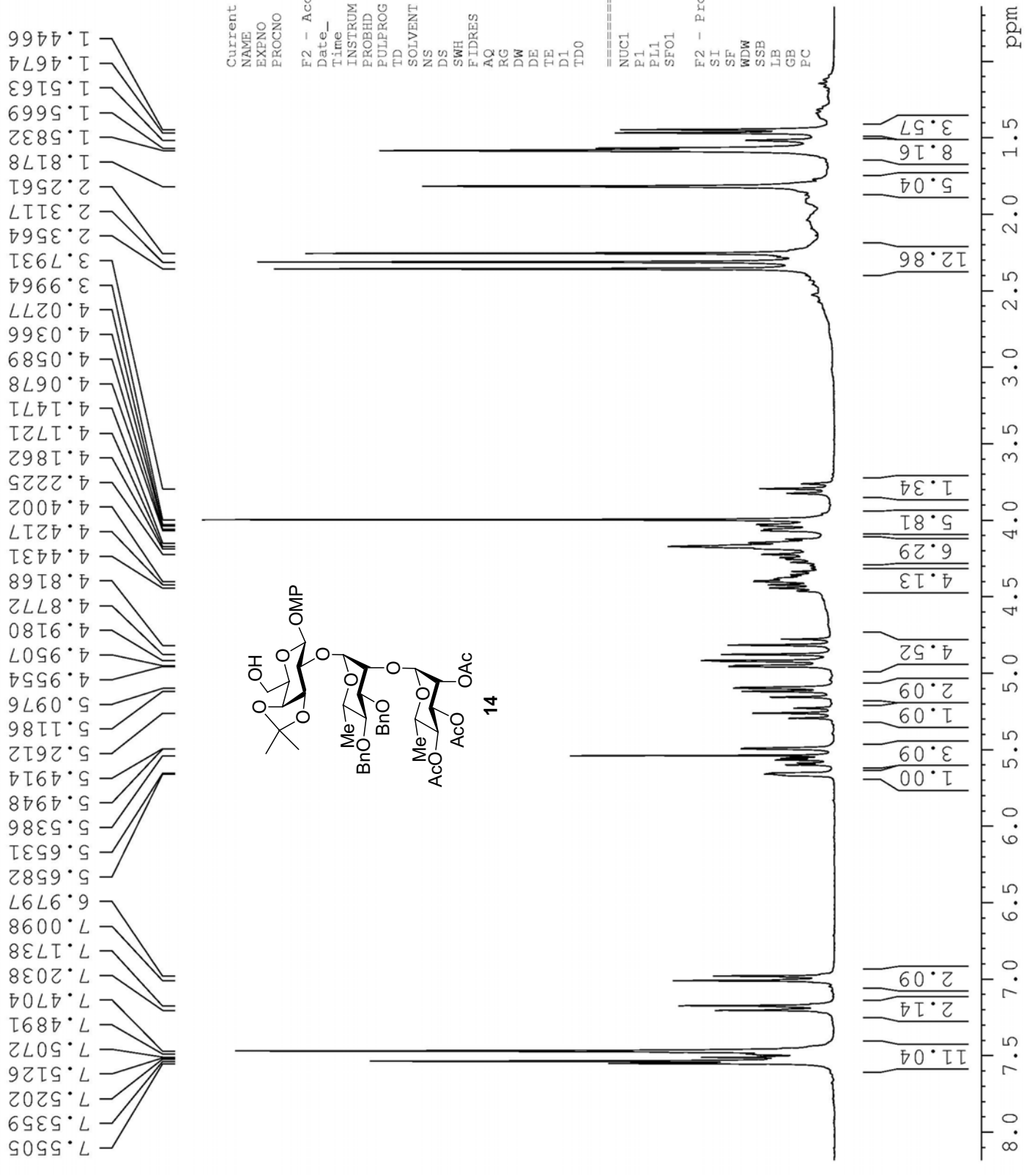

OTBDPS
OMP
Me
BnO
BnO
Me
AcO
AcO
OAc
13
Current Data Parameters
NAME 23.01.07
EXPNO 60
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070123
Time 13.52
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 19531.250 Hz
FIDRES 0.298023 Hz
AQ 1.6777716 sec
RG 9
DW 25.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.70 usec
PL1 -3.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 17.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677601 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
169.74
169.62
155.07
151.64
138.87
138.36
135.50
133.13
129.64
128.17
128.08
127.58
127.27
118.09
114.42
110.27
100.42
99.39
97.50
79.81
79.26
77.41
76.99
76.57
76.16
75.45
74.87
73.72
73.31
72.15
71.08
69.70
69.09
66.67
62.85
55.46
27.94
26.67
26.29
20.74
20.68
20.61
19.09
17.88
17.02
ppm

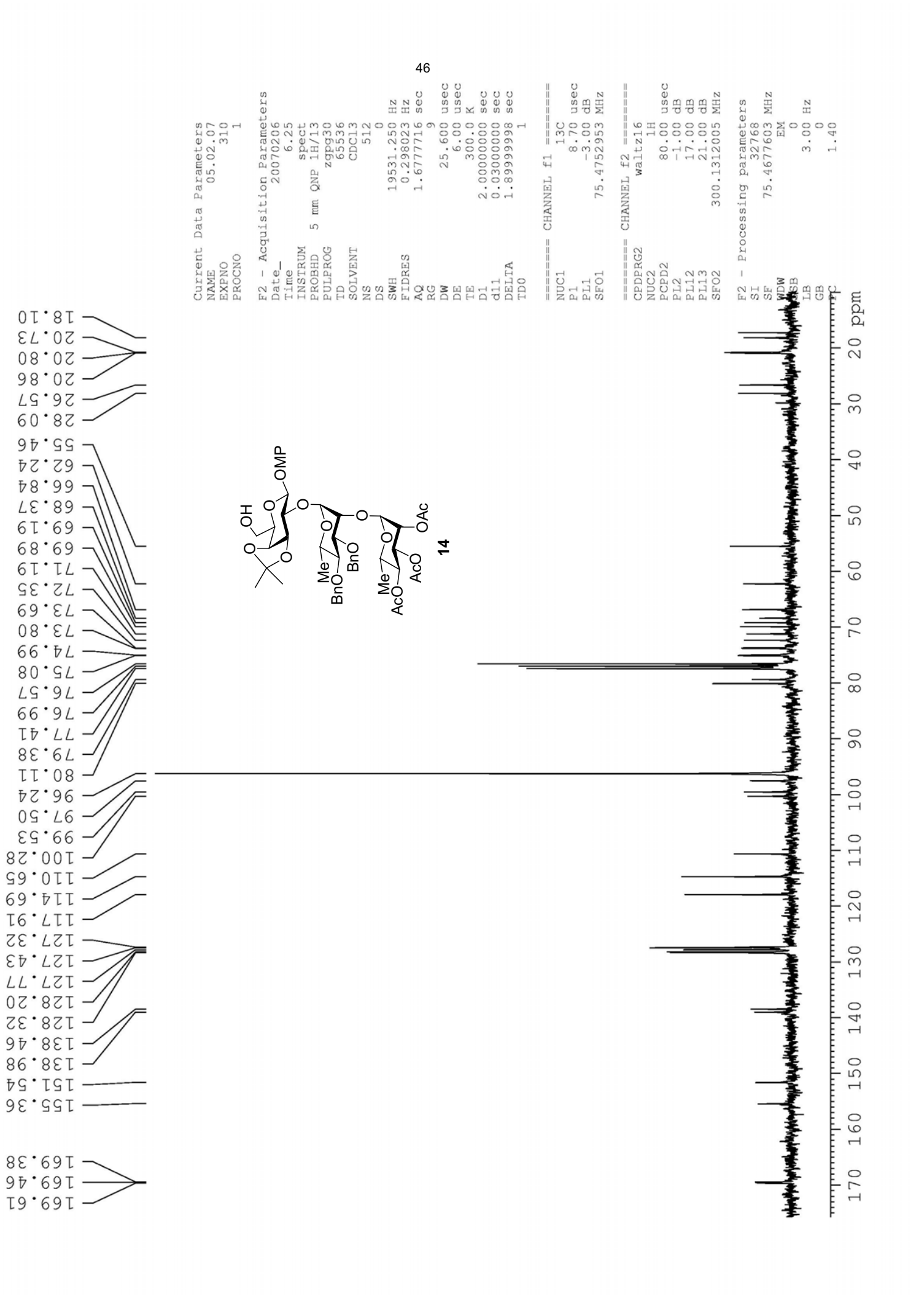


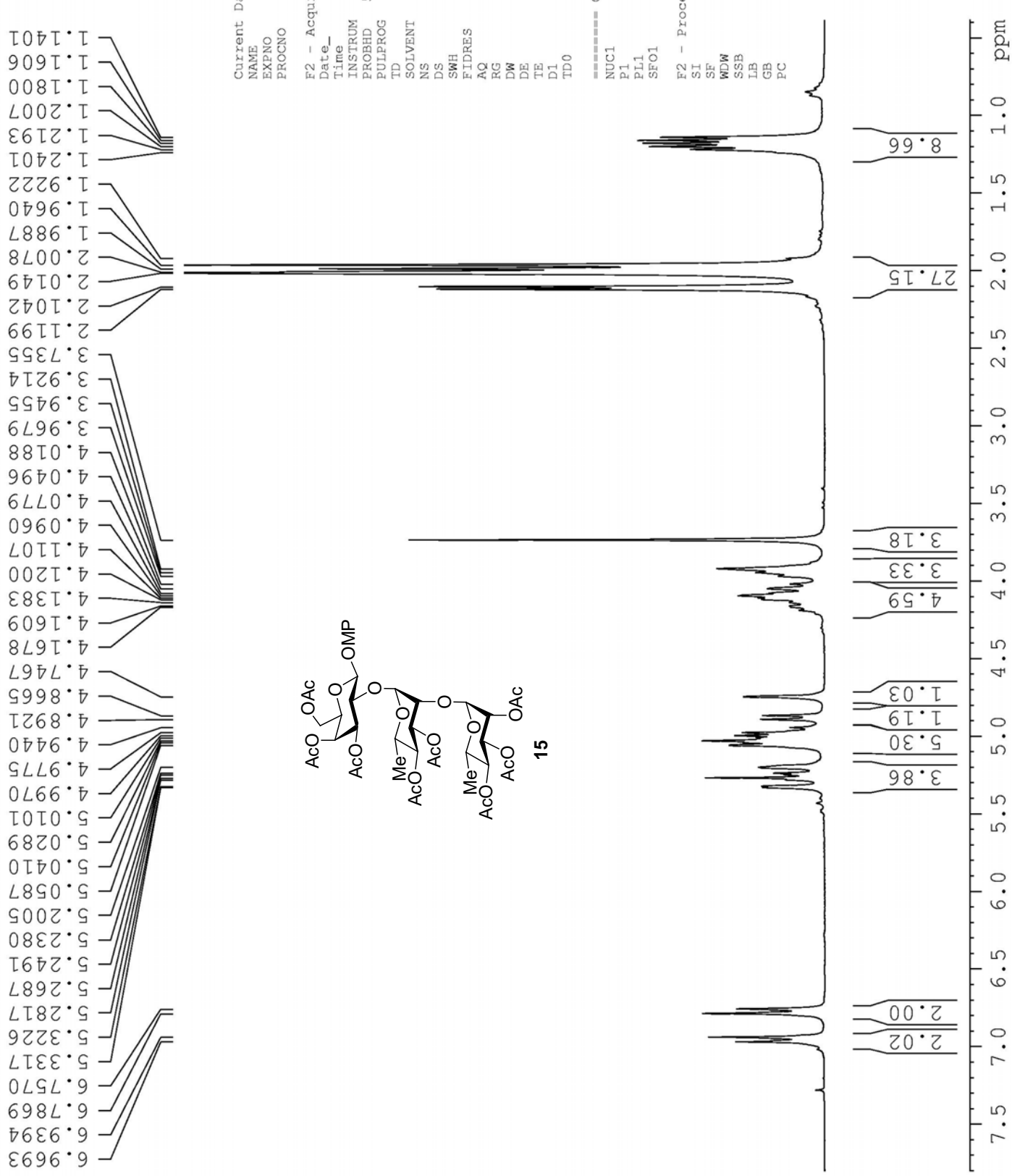
13

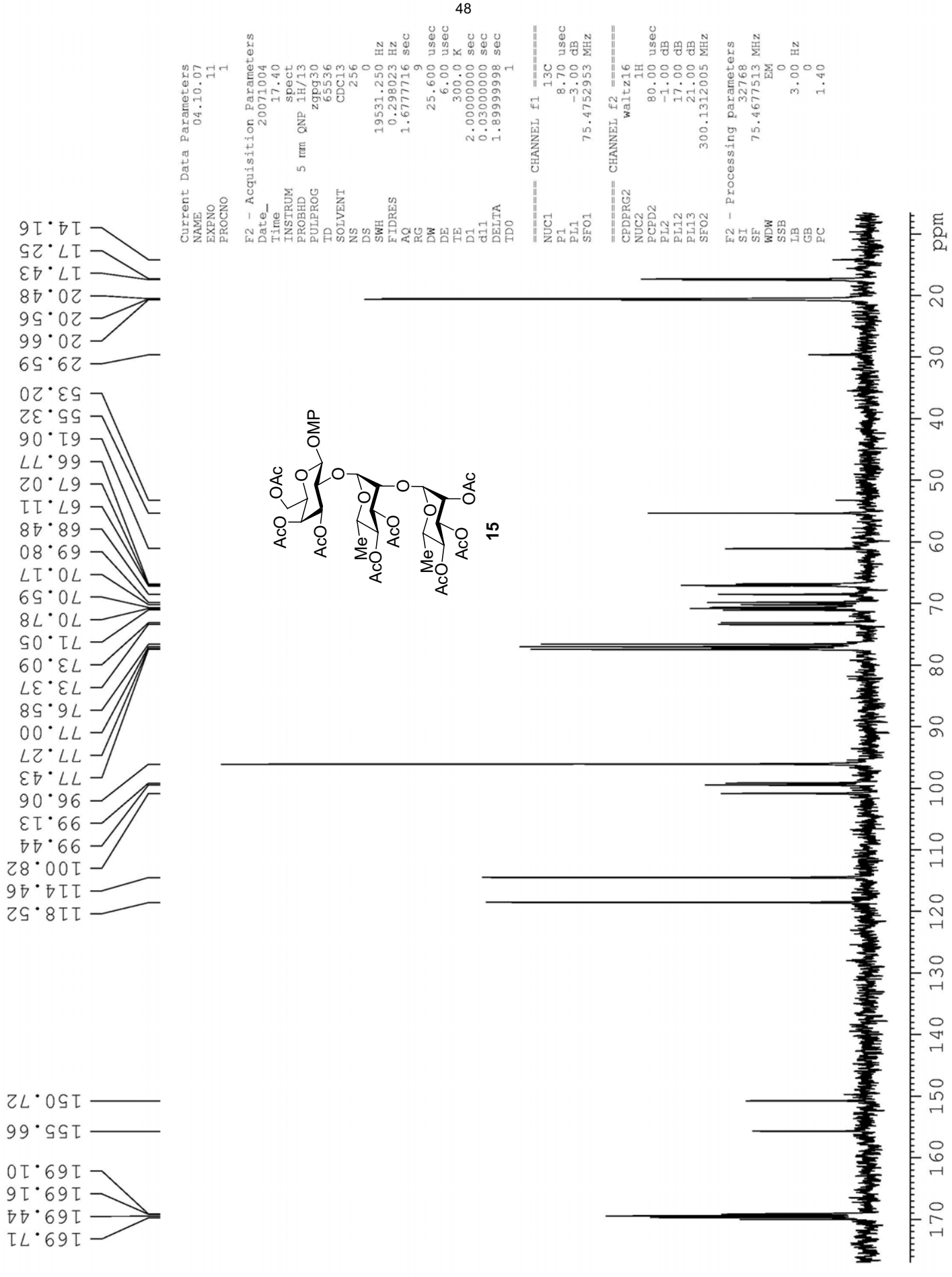


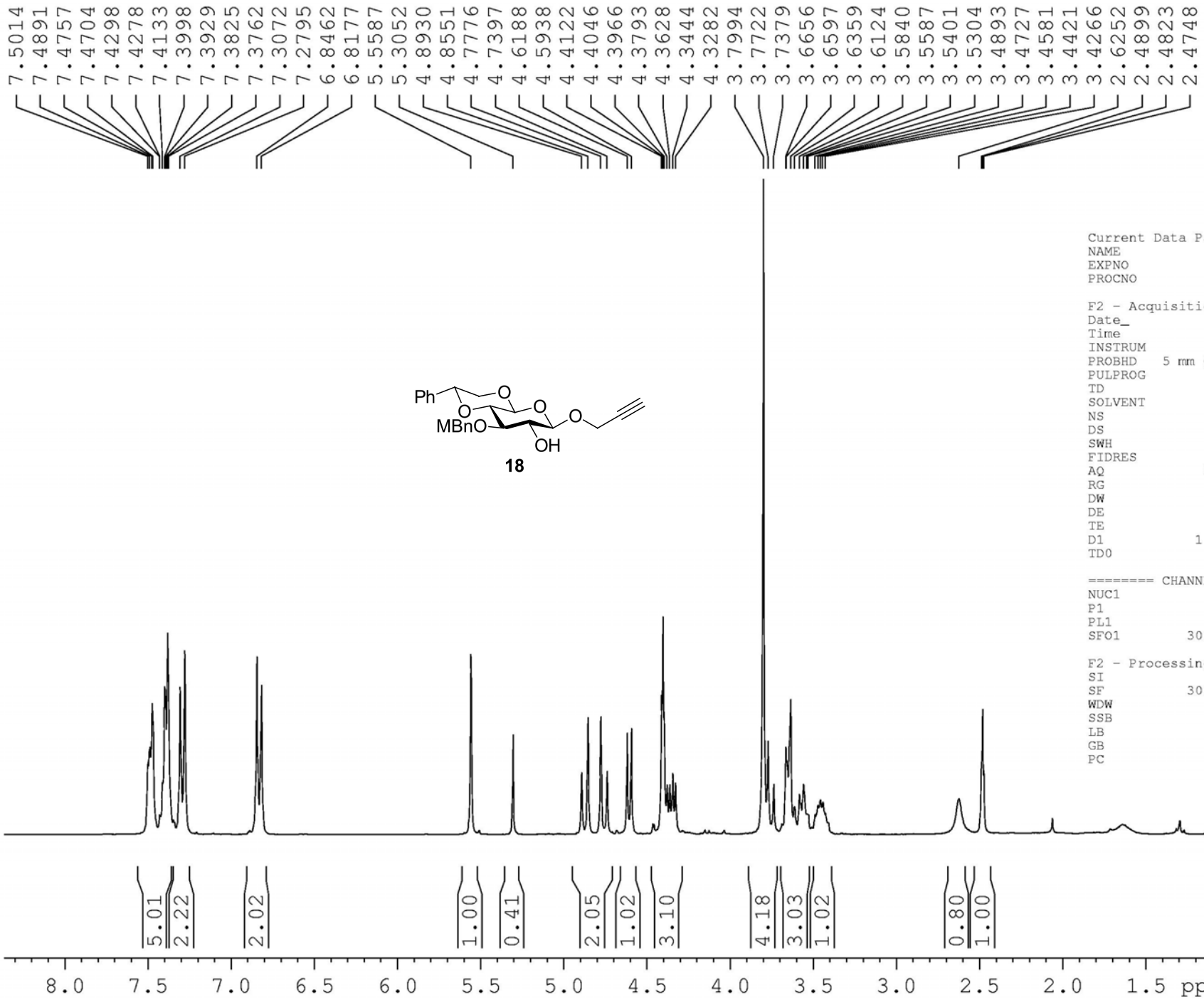

```
Current Data Parameters
NAME                 23.01.07
EXPNO                      62
PROCNO                      1

F2 - Acquisition Parameters
Date_              20070123
Time                   13.56
INSTRUM                spect
PROBHD    5 mm QNP 1H/13
PULPROG       hsqcetgpsi2
TD                       1024
SOLVENT                CDCl3
NS                          8
DS                         16
SWH                2500.000 Hz
FIDRES             2.441406 Hz
AQ                 0.2048500 sec
RG                      2050
DW                  200.000 usec
DE                    6.00 usec
TE                    300.0 K
CNST2             145.0000000
d0                 0.00000300 sec
D1                 1.42320001 sec
d4                 0.00172414 sec
d11                0.03000000 sec
d13                0.00000400 sec
D16                0.00020000 sec
D24                0.00086207 sec
DELTA              0.00127920 sec
DELTA1             0.00120800 sec
DELTA2             0.00006207 sec
DELTA3             0.00052414 sec
IN0                0.00004000 sec
ST1CNT                   128
ZGOPTNS

======== CHANNEL f1 ========
NUC1                       1H
P1                     11.60 usec
p2                     23.20 usec
P28                  1000.00 usec
PL1                    -1.00 dB
SFO1            300.1312551 MHz

======== CHANNEL f2 ========
CPDPRG2                 garp
NUC2                     13C
P3                      8.70 usec
p4                     17.40 usec
PCPD2                  80.00 usec
PL2                    -3.00 dB
PL12                   16.27 dB
SFO2             75.4734070 MHz

====== GRADIENT CHANNEL =====
GPNAM1              SINE.100
GPNAM2              SINE.100
GPNAM3              SINE.100
GPNAM4              SINE.100
GPZ1                   80.00 %
GPZ2                   20.10 %
GPZ3                   11.00 %
GPZ4                   -5.00 %
P16                  1000.00 usec
P19                   600.00 usec

F1 - Acquisition parameters
ND0                         2
TD                        256
SFO1              75.47341 MHz
FIDRES           48.828125 Hz
SW                  165.621 ppm
FnMODE       Echo-Antiecho

F2 - Processing parameters
SI                       1024
SF              300.1300180 MHz
WDW                     QSINE
SSB                         2
LB                     0.00 Hz
GB                          0
PC                     1.40

F1 - Processing parameters
SI                       1024
MC2           echo-antiecho
SF              75.4677490 MHz
WDW                     QSINE
SSB                         2
LB                     0.00 Hz
GB                          0
```

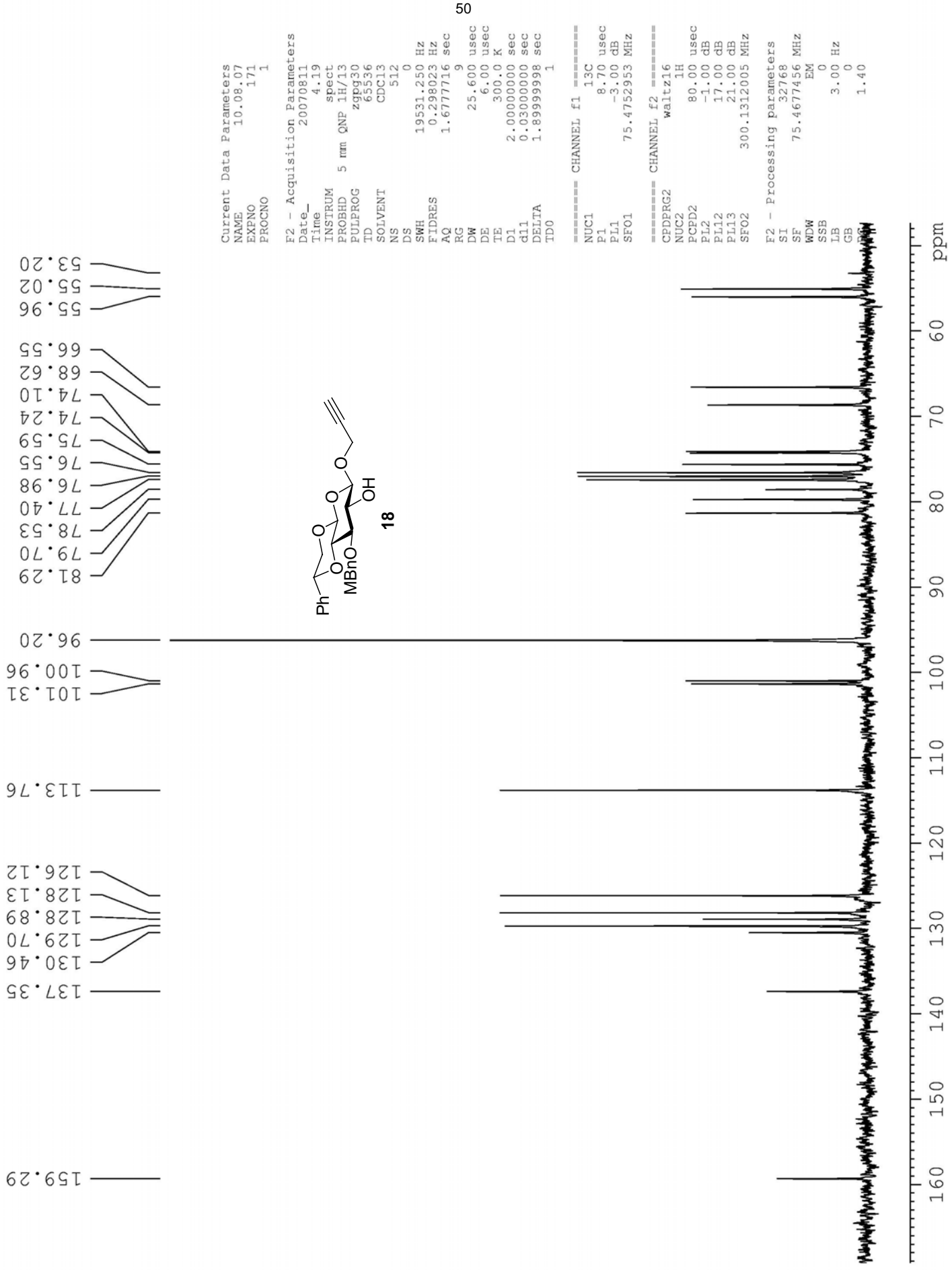

Current Data Parameters
NAME 05.02.07
EXPNO 311
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070206
Time 6.27
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDC13
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 144
DW 80.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.60 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 16384
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OMP
OH
BnO
Me
AcO
OAc
14
11.04
2.14
2.09
1.00
3.09
1.09
2.09
4.52
4.13
6.29
5.81
1.34
12.86
5.04
8.16
3.57
ppm

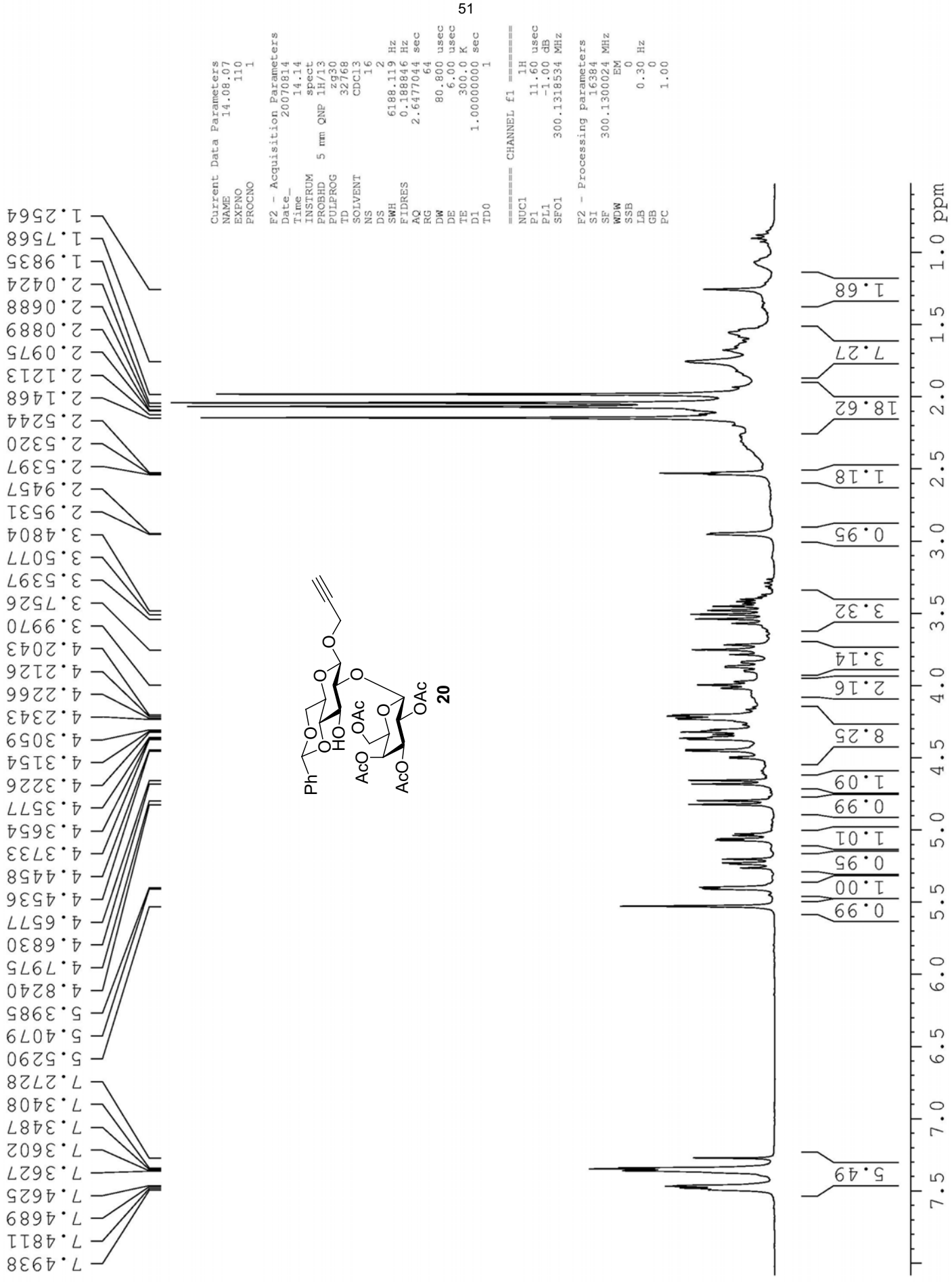

Current Data Parameters
NAME 05.02.07
EXPNO 310
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070206
Time 6.25
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 19531.250 Hz
FIDRES 0.298023 Hz
AQ 1.6777716 sec
RG 9
DW 25.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.70 usec
PL1 -3.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 17.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677603 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
OH
OMP
BnO
Me
AcO
OAc
14
ppm

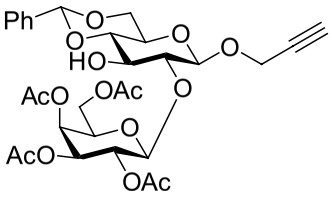

Current Data Parameters
NAME 04.10.07
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20071004
Time 17.25
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 50.8
DW 80.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.60 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 16384
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OMP
OAc
AcO
Me
15
8.66
27.15
4.59
3.33
3.18
1.03
1.19
5.30
3.86
2.02
2.00
ppm


Current Data Parameters
NAME 04.10.07
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20071004
Time 17.40
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 0
SWH 19531.250 Hz
FIDRES 0.298023 Hz
AQ 1.6777716 sec
RG 9
DW 25.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.70 usec
PL1 -3.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 17.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677513 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
OMP
AcO
OAc
Me
15
ppm

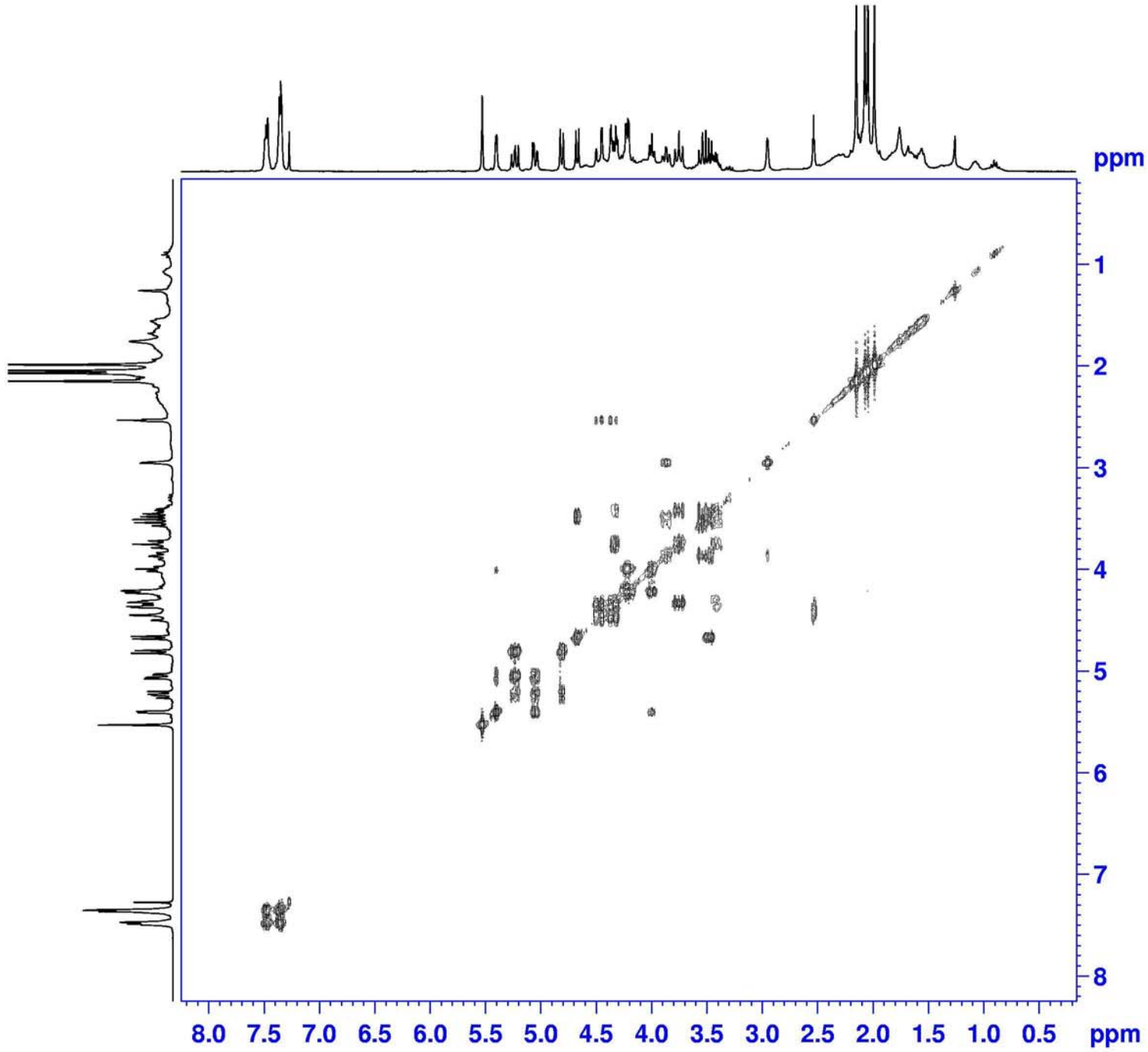

7.5014
7.4891
7.4757
7.4704
7.4298
7.4278
7.4133
7.3998
7.3929
7.3825
7.3762
7.3072
7.2795
6.8462
6.8177
5.5587
5.3052
4.8930
4.8551
4.7776
4.7397
4.6188
4.5938
4.4122
4.4046
4.3966
4.3793
4.3628
4.3444
4.3282
3.7994
3.7722
3.7379
3.6656
3.6597
3.6359
3.6124
3.5840
3.5587
3.5401
3.5304
3.4893
3.4727
3.4581
3.4421
3.4266
2.6252
2.4899
2.4823
2.4748
Ph
O
O
O
O
MBnO
OH
18
Current Data Parameters
NAME 10.08.07
EXPNO 170
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070811
Time 3.45
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 144
DW 80.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.60 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 16384
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
5.01
2.22
2.02
1.00
0.41
2.05
1.02
3.10
4.18
3.03
1.02
0.80
1.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm


Current Data Parameters
NAME 10.08.07
EXPNO 171
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070811
Time 4.19
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 512
DS 0
SWH 19531.250 Hz
FIDRES 0.298023 Hz
AQ 1.6777716 sec
RG 9
DW 25.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.70 usec
PL1 -3.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 17.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677456 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
Ph
MBnO
OH
18
159.29
137.35
130.46
129.70
128.89
128.13
126.12
113.76
101.31
100.96
96.20
81.29
79.70
78.53
77.40
76.98
76.55
75.59
74.24
74.10
68.62
66.55
55.96
55.02
53.20
ppm

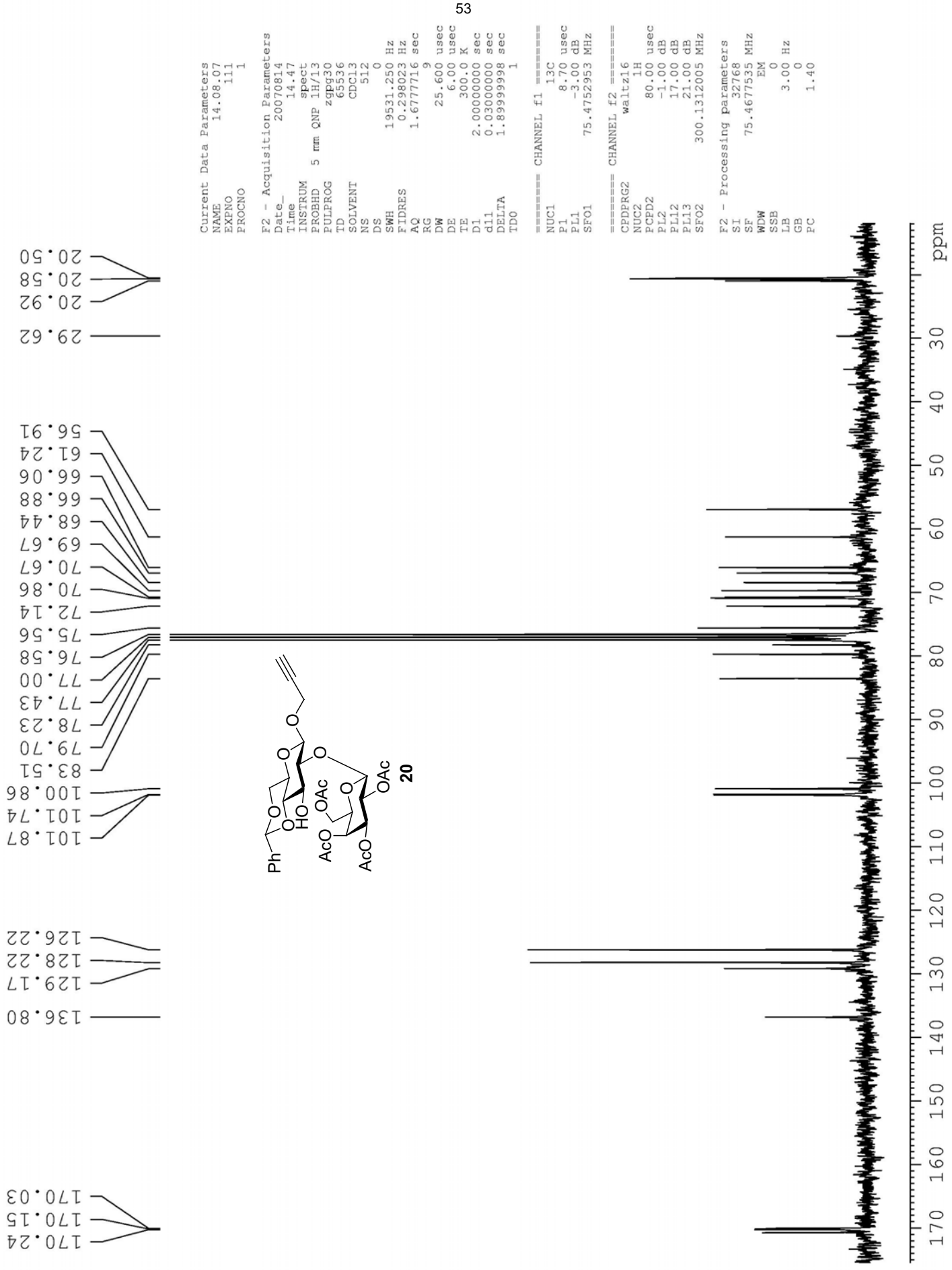

Current Data Parameters
NAME 14.08.07
EXPNO 110
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070814
Time 14.14
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6188.119 Hz
FIDRES 0.188846 Hz
AQ 2.6477044 sec
RG 64
DW 80.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.60 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 16384
SF 300.1300024 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Ph
HO
AcO
OAc
AcO
OAc
20
7.4938
7.4811
7.4689
7.4625
7.3627
7.3602
7.3487
7.3408
7.2728
5.5290
5.4079
5.3985
4.8240
4.7975
4.6830
4.6577
4.4536
4.4458
4.3733
4.3654
4.3577
4.3226
4.3154
4.3059
4.2343
4.2266
4.2126
4.2043
3.9970
3.7526
3.5397
3.5077
3.4804
2.9531
2.9457
2.5397
2.5320
2.5244
2.1468
2.1213
2.0975
2.0889
2.0688
2.0424
1.9835
1.7568
1.2564
5.49
0.99
1.00
0.95
1.01
0.99
1.09
8.25
2.16
3.14
3.32
0.95
1.18
18.62
7.27
1.68
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0 ppm

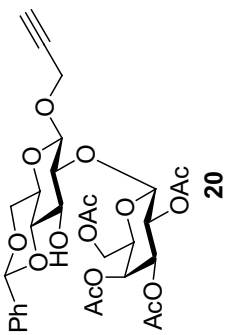


20

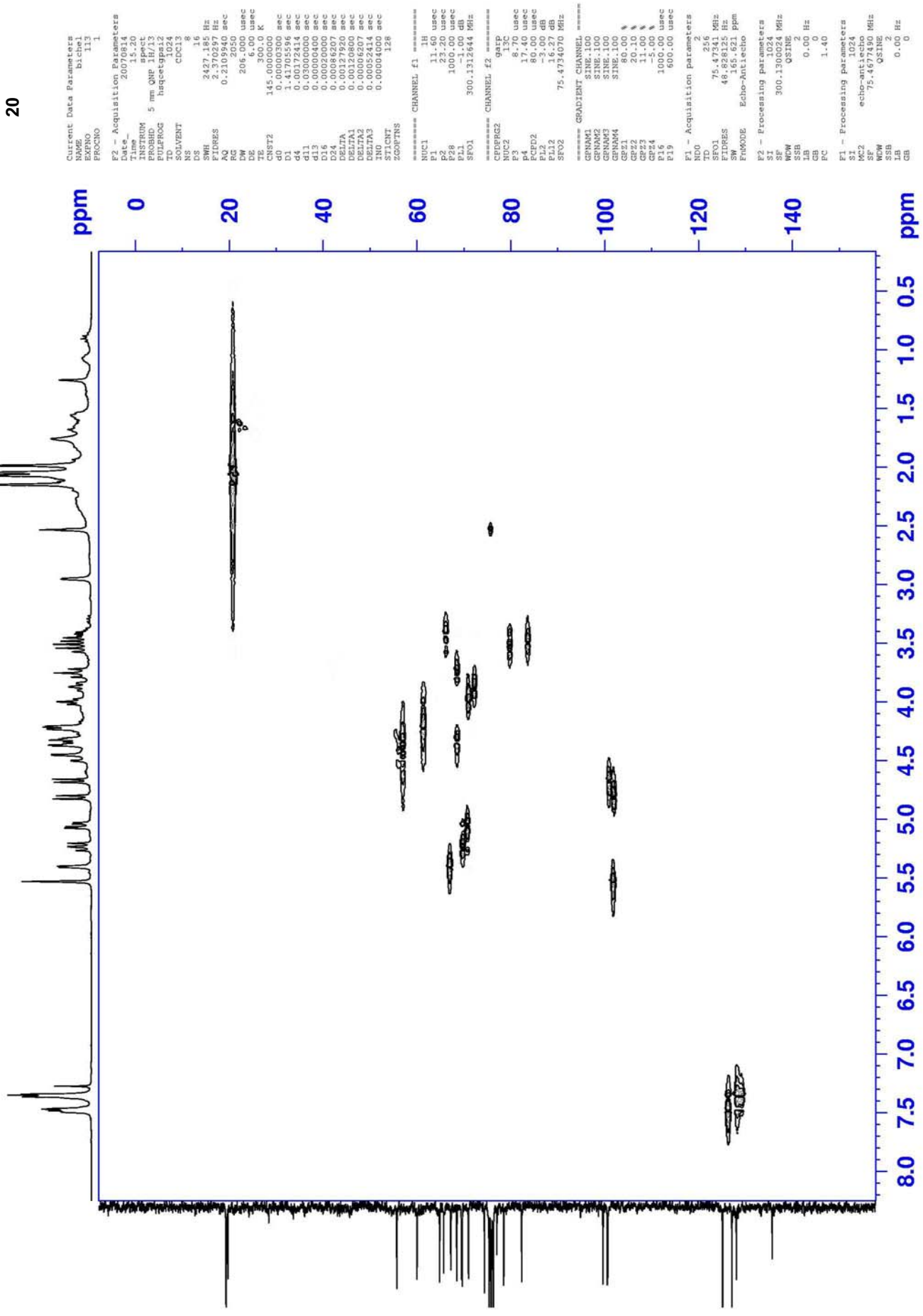


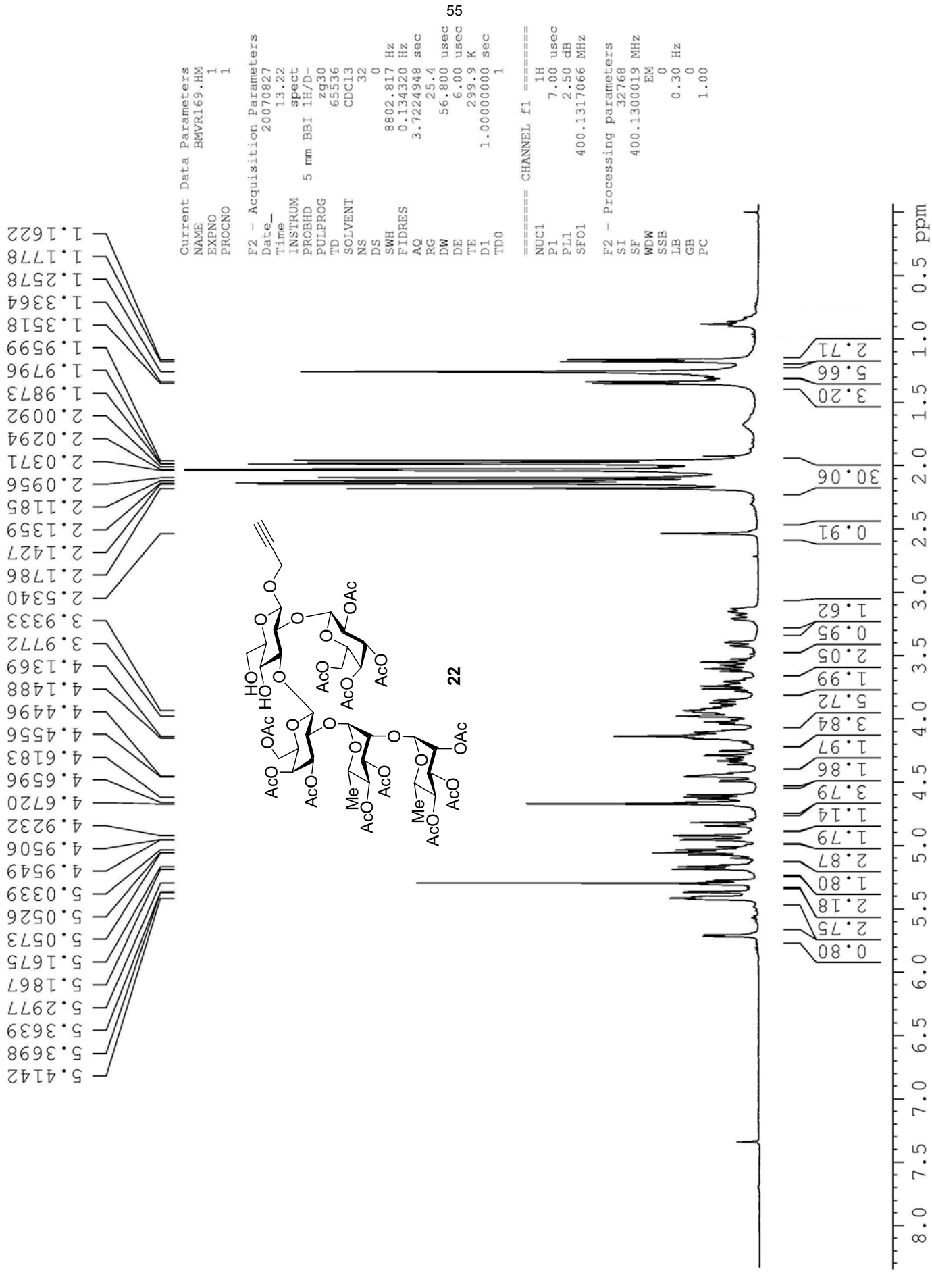

```
Current Data Parameters
NAME                  bichel
EXPNO                    112
PROCNO                     1

F2 - Acquisition Parameters
Date_              20070814
Time                  14.48
INSTRUM               spect
PROBHD    5 mm QNP 1H/13
PULPROG           cosygpqf
TD                     2048
SOLVENT               CDC13
NS                        8
DS                        8
SWH                2427.185 Hz
FIDRES             1.185149 Hz
AQ                 0.4219380 sec
RG                      45.2
DW                   206.000 usec
DE                     6.00 usec
TE                    300.0 K
d0                 0.00000300 sec
D1                 1.32059395 sec
d13                0.00000400 sec
D16                0.00020000 sec
IN0                0.00041200 sec

========== CHANNEL f1 ========
NUC1                      1H
P0                     11.60 usec
P1                     11.60 usec
PL1                    -1.00 dB
SFO1           300.1312644 MHz

====== GRADIENT CHANNEL =====
GPNAM1              SINE.100
GPNAM2              SINE.100
GPZ1                  10.00 %
GPZ2                  10.00 %
P16                 1000.00 usec

F1 - Acquisition parameters
ND0                       1
TD                      128
SFO1           300.1313 MHz
FIDRES          18.962379 Hz
SW                  8.087 ppm
FnMODE                 QF

F2 - Processing parameters
SI                     1024
SF             300.1300024 MHz
WDW                    SINE
SSB                       0
LB                     0.00 Hz
GB                        0
PC                     1.40

F1 - Processing parameters
SI                     1024
MC2                    QF
SF             300.1300024 MHz
WDW                    SINE
SSB                       0
LB                     0.00 Hz
GB                        0
```

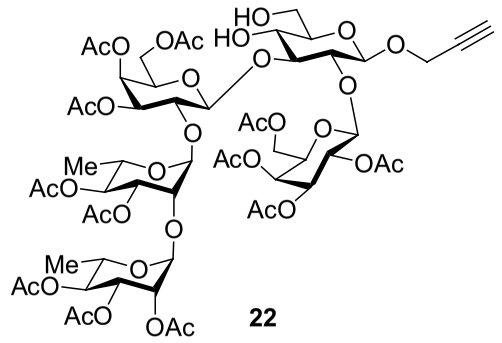

Current Data Parameters
NAME 14.08.07
EXPNO 111
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070814
Time 14.47
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 19531.250 Hz
FIDRES 0.298023 Hz
AQ 1.6777716 sec
RG 9
DW 25.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.70 usec
PL1 -3.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 17.00 dB
PL13 21.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677535 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
170.24
170.15
170.03
136.80
129.17
128.22
126.22
101.87
101.74
100.86
83.51
79.70
78.23
77.43
77.00
76.58
75.56
72.14
70.86
70.67
69.67
68.44
66.88
66.06
61.24
56.91
29.62
20.92
20.58
20.50
Ph
HO
AcO
OAc
AcO
OAc
20
ppm


Ph
O
O
O
HO
AcO
OAc
O
O
AcO
OAc

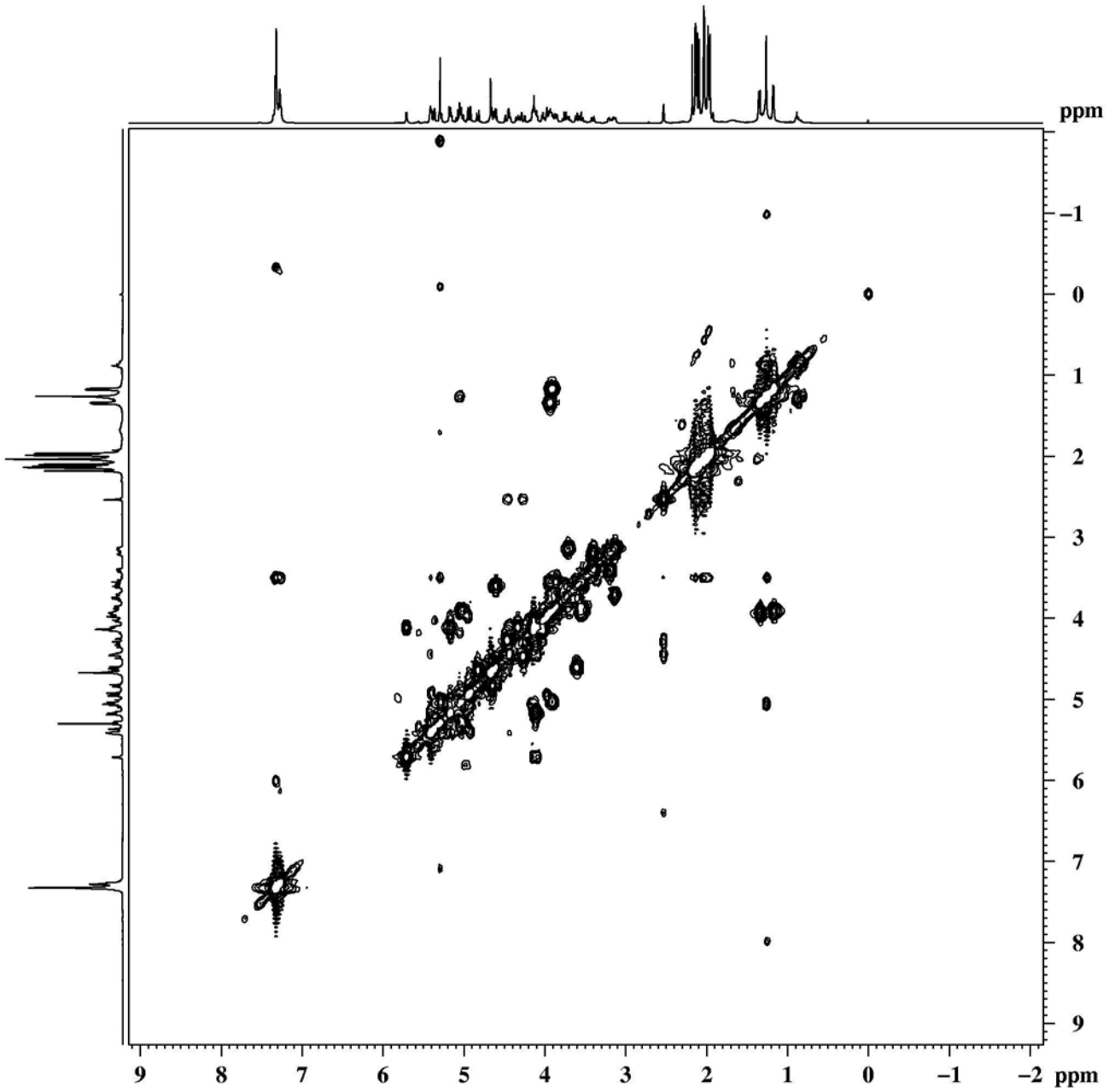
20


Current Data Parameters
NAME bichel
EXPNO 113
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070814
Time 15.20
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG hsqcetgpsi2
TD 1024
SOLVENT CDCl3
NS 8
DS 16
SWH 2427.185 Hz
FIDRES 2.370297 Hz
AQ 0.2109940 sec
RG 2050
DW 206.000 usec
DE 6.00 usec
TE 300.0 K
CNST2 145.0000000
d0 0.00000300 sec
D1 1.41705596 sec
d4 0.00172414 sec
d11 0.03000000 sec
d13 0.00000400 sec
D16 0.00020000 sec
D24 0.00006207 sec
DELTA 0.00127920 sec
DELTA1 0.00206207 sec
DELTA2 0.00006207 sec
DELTA3 0.00052414 sec
IN0 0.00004000 sec
ST1CNT 128
ZGOPTNS
======== CHANNEL f1 ========
NUC1 1H
P1 11.60 usec
P2 23.20 usec
P28 1000.00 usec
PL1 -1.00 dB
SFO1 300.1312644 MHz
======== CHANNEL f2 ========
CPDPRG2 garp
NUC2 13C
P3 8.70 usec
p4 17.40 usec
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 16.27 dB
SFO2 75.4734070 MHz
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPNAM3 SINE.100
GPNAM4 SINE.100
GPZ1 80.00 %
GPZ2 20.10 %
GPZ3 11.00 %
GPZ4 -5.00 %
P16 1000.00 usec
P19 600.00 usec
F1 - Acquisition parameters
NDO 2
TD 256
SFO1 75.47341 MHz
FIDRES 48.828125 Hz
SW 165.621 ppm
FnMODE Echo-Antiecho
F2 - Processing parameters
SI 1024
SF 300.1300024 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.40
F1 - Processing parameters
SI 1024
MC2 echo-antiecho
SF 75.4677490 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
ppm
0
20
40
60
80
100
120
140
ppm
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5

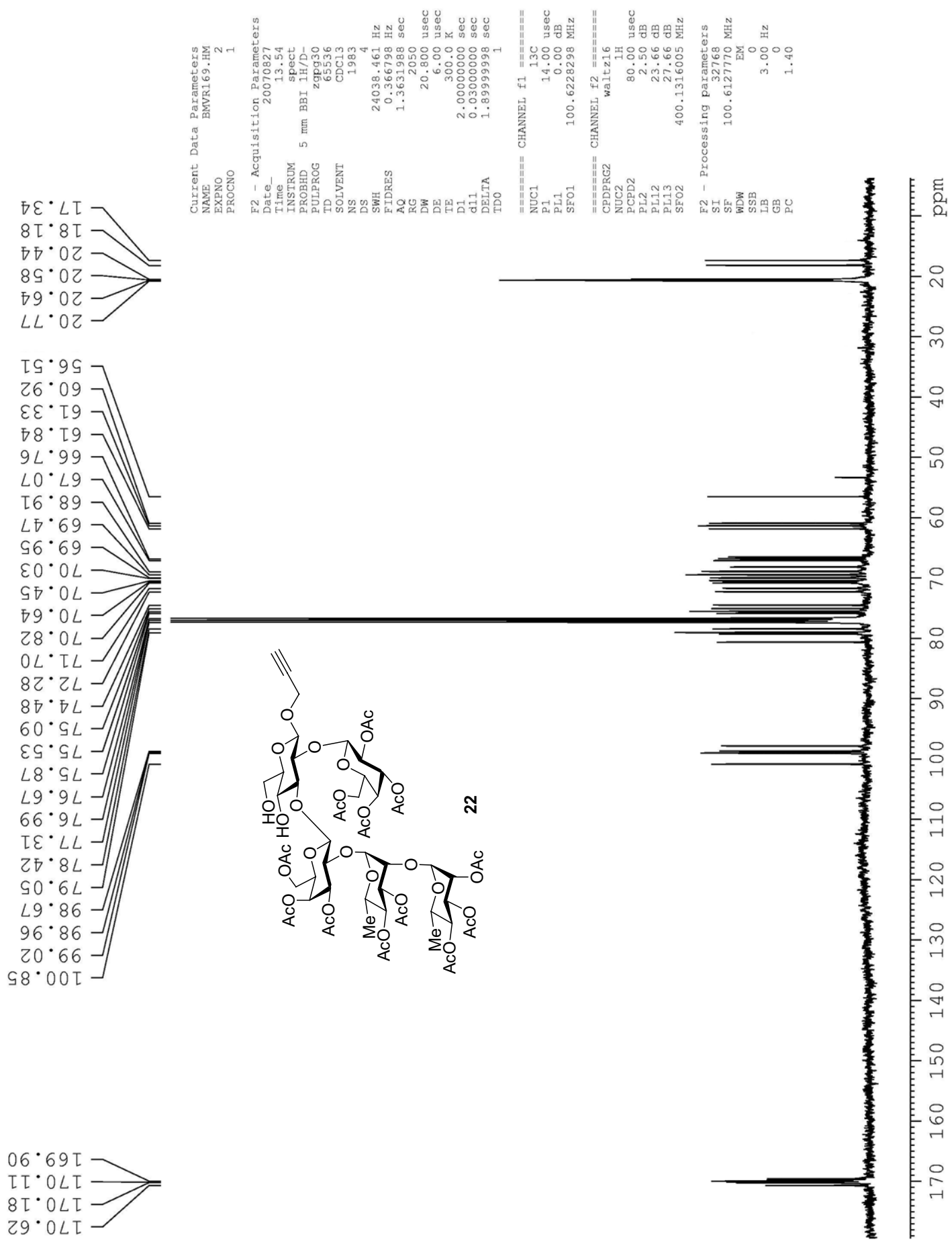

Current Data Parameters
NAME BMVR169.HM
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070827
Time 13.22
INSTRUM spect
PROBHD 5 mm BBI 1H/D-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 0
SWH 8802.817 Hz
FIDRES 0.134320 Hz
AQ 3.7224948 sec
RG 25.4
DW 56.800 usec
DE 6.00 usec
TE 299.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.00 usec
PL1 2.50 dB
SFO1 400.1317066 MHz
F2 - Processing parameters
SI 32768
SF 400.1300019 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
22
5.4142
5.3698
5.3639
5.2977
5.1867
5.1675
5.0573
5.0526
5.0339
4.9549
4.9506
4.9232
4.6720
4.6596
4.6183
4.4556
4.4496
4.1488
4.1369
3.9772
3.9333
2.5340
2.1786
2.1427
2.1359
2.1185
2.0956
2.0371
2.0294
2.0092
1.9873
1.9796
1.9599
1.3518
1.3364
1.2578
1.1778
1.1622
0.80
2.75
2.18
1.80
2.87
1.79
1.14
3.79
1.86
1.97
3.84
5.72
1.99
2.05
0.95
1.62
0.91
30.06
3.20
5.66
2.71
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

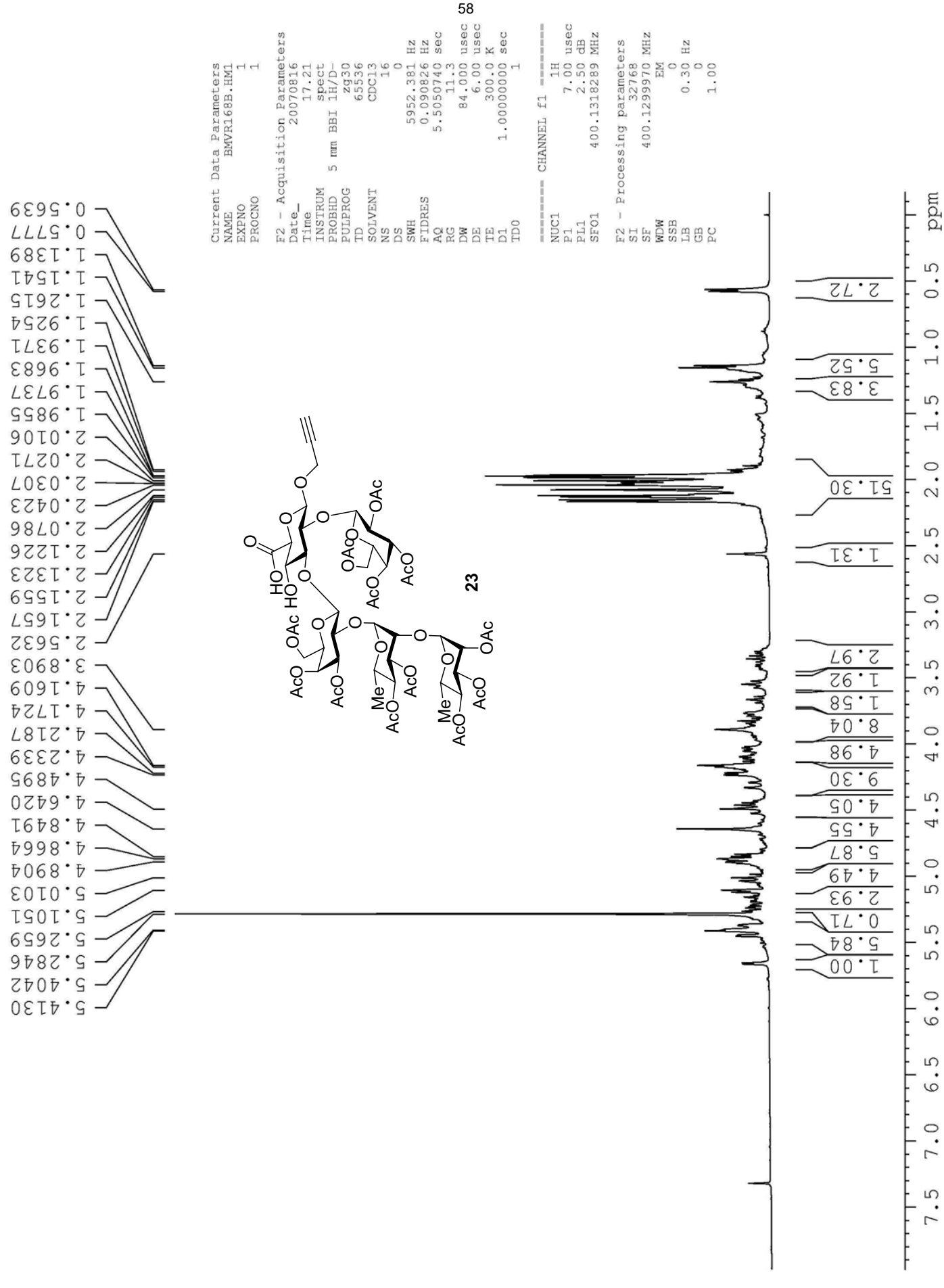


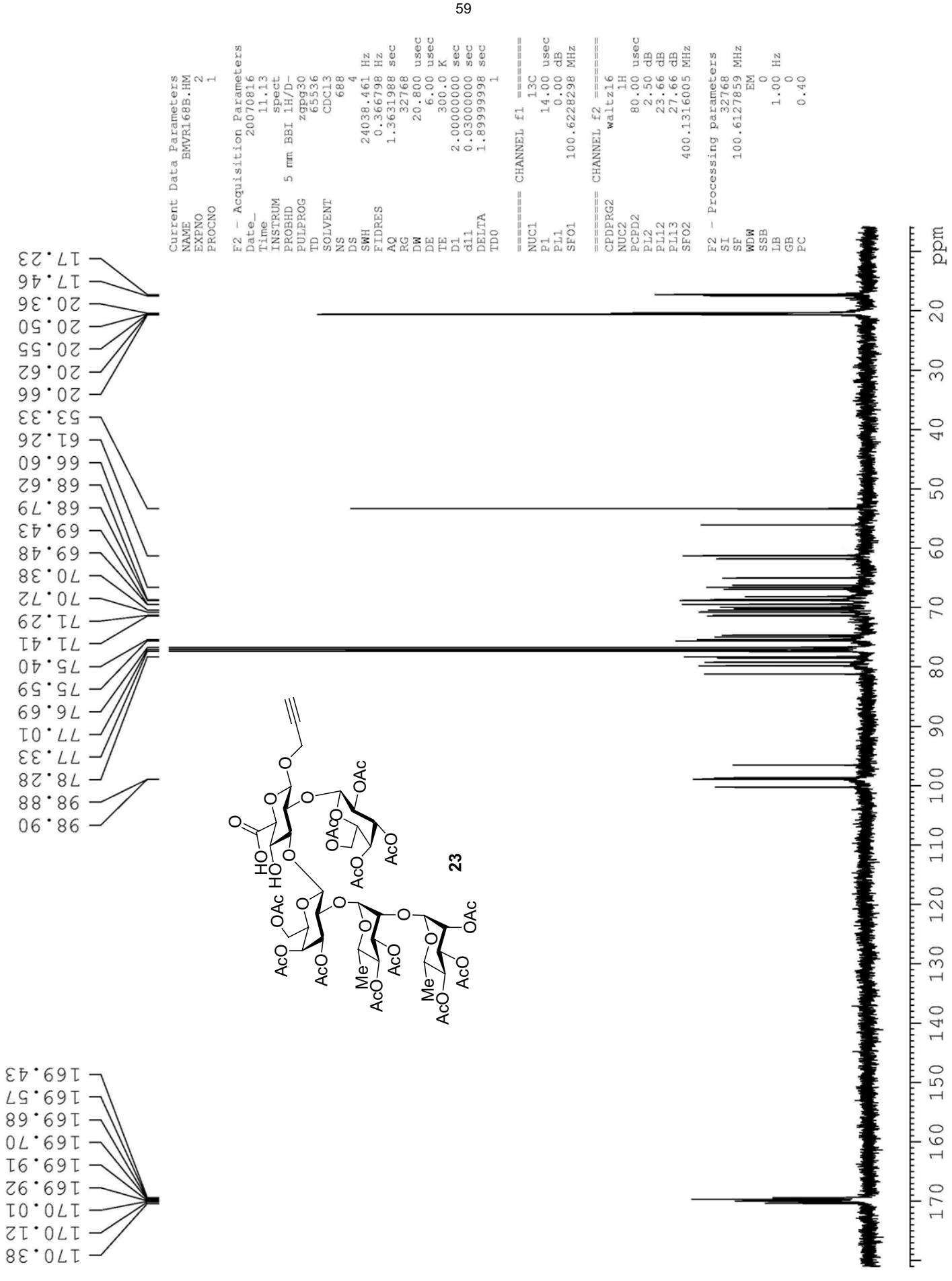
22

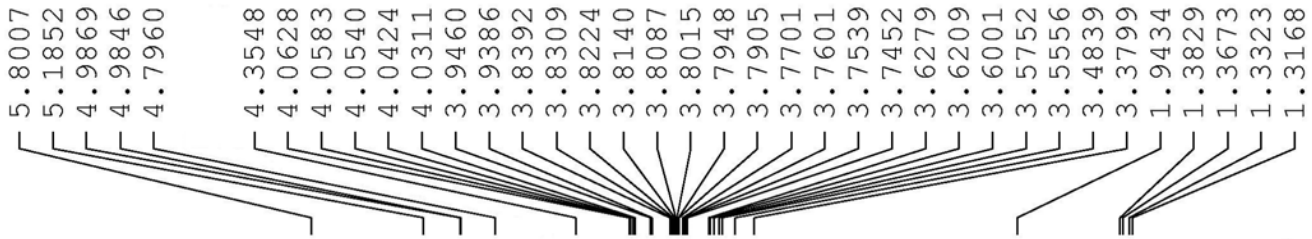


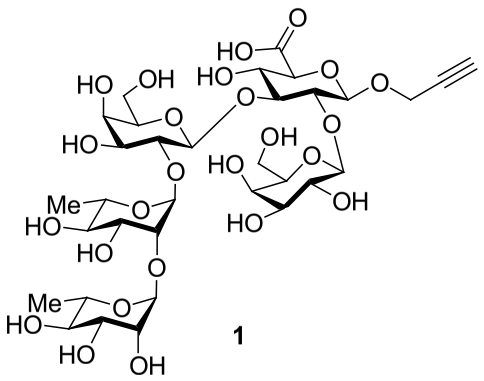

```
Current Data Parameters
NAME          BMVR169.HM
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070827
Time               15.25
INSTRUM            spect
PROBHD   5 mm BBI 1H/D-
PULPROG         cosyqf90
TD                 2048
SOLVENT            CDCl3
NS                   16
DS                    4
SWH             5597.015 Hz
FIDRES          2.732918 Hz
AQ              0.1830047 sec
RG                  22.6
DW                89.333 usec
DE                 6.00 usec
TE                300.0 K
d0              0.00000300 sec
D1              2.00000000 sec
IN0             0.00017865 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 7.00 usec
PL1                2.50 dB
SFO1         400.1314030 MHz

F1 - Acquisition parameters
ND0                   1
TD                  256
SFO1            400.1314 MHz
FIDRES         21.865379 Hz
SW                13.989 ppm
FnMODE                QF

F2 - Processing parameters
SI                  512
SF           400.1300000 MHz
WDW                 SINE
SSB                    0
LB                0.00 Hz
GB                     0
PC                 1.40

F1 - Processing parameters
SI                  512
MC2                  QF
SF           400.1300000 MHz
WDW                 SINE
SSB                    0
LB                0.00 Hz
GB                     0
```


Current Data Parameters
NAME BMVR169.HM
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070827
Time 13.54
INSTRUM spect
PROBHD 5 mm BBI 1H/D-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1983
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 14.00 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 23.66 dB
PL13 27.66 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127770 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
170.62
170.18
170.11
169.90
100.85
99.02
98.96
98.67
79.05
78.42
77.31
76.99
76.67
75.87
75.53
75.09
74.48
72.28
71.70
70.82
70.64
70.45
70.03
69.95
69.47
68.91
67.07
66.76
61.84
61.33
60.92
56.51
20.77
20.64
20.58
20.44
18.18
17.34
22
ppm


23
Current Data Parameters
NAME BMVR168B.HM1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070816
Time 17.21
INSTRUM spect
PROBHD 5 mm BBI 1H/D-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 5952.381 Hz
FIDRES 0.090826 Hz
AQ 5.5050740 sec
RG 11.3
DW 84.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.00 usec
PL1 2.50 dB
SFO1 400.1318289 MHz
F2 - Processing parameters
SI 32768
SF 400.1299970 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
5.4130
5.4042
5.2846
5.2659
5.1051
5.0103
4.8904
4.8664
4.8491
4.6420
4.4895
4.2339
4.2187
4.1724
4.1609
3.8903
2.5632
2.1657
2.1559
2.1323
2.1226
2.0786
2.0423
2.0307
2.0271
2.0106
1.9855
1.9737
1.9683
1.9371
1.9254
1.2615
1.1541
1.1389
0.5777
0.5639
1.00
5.84
0.71
2.93
4.49
5.87
4.55
4.05
9.30
4.98
8.04
1.58
1.92
2.97
1.31
51.30
3.83
5.52
2.72
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm


Current Data Parameters
NAME BMVR168B.HM
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070816
Time 11.13
INSTRUM spect
PROBHD 5 mm BBI 1H/D-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 688
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 32768
DW 20.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 14.00 usec
PL1 0.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 23.66 dB
PL13 27.66 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127859 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.40
98.90
98.88
78.28
77.33
77.01
76.69
75.59
75.40
71.41
71.29
70.72
70.38
69.48
69.43
68.79
68.62
66.60
61.26
53.33
20.66
20.62
20.55
20.50
20.36
17.46
17.23
170.38
170.12
170.01
169.92
169.91
169.70
169.68
169.57
169.43
ppm
23





1

Current Data Parameters

NAME	BMVR176.HM
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

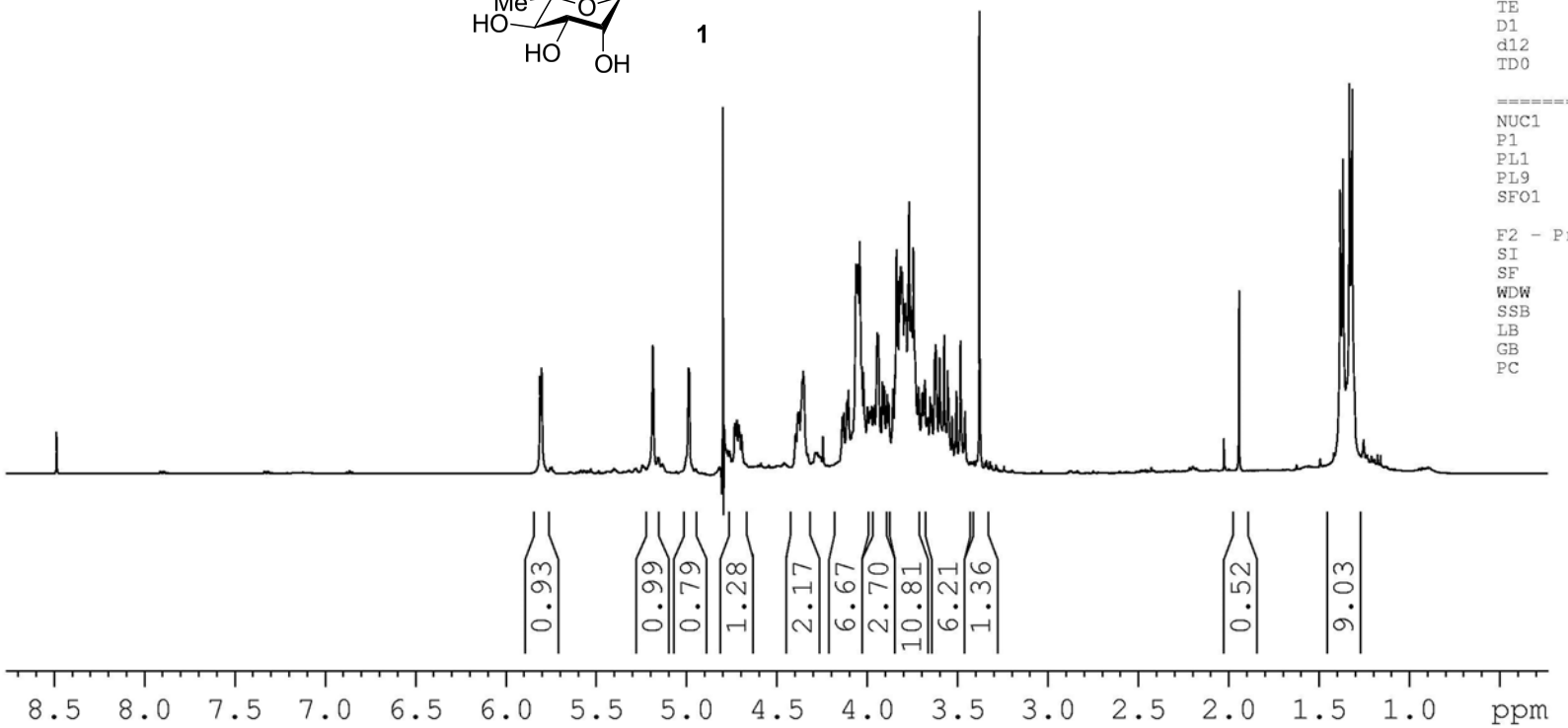
Date_	20070913
Time	11.24
INSTRUM	spect
PROBHD	5 mm BBI 1H/D-
PULPROG	zgpr
TD	65536
SOLVENT	D2O
NS	80
DS	0
SWH	9615.385 Hz
FIDRES	0.146719 Hz
AQ	3.4079220 sec
RG	114
DW	52.000 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
d12	0.00002000 sec
TD0	1

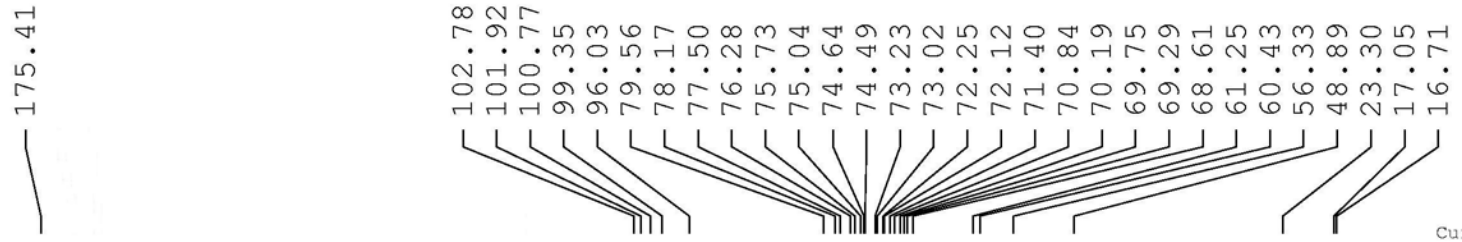
======== CHANNEL f1 ========

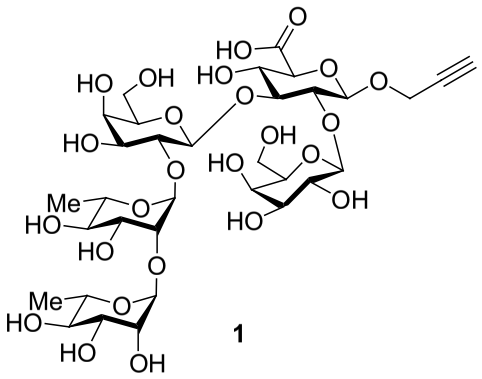
NUC1	1H
P1	7.00 usec
PL1	2.50 dB
PL9	60.00 dB
SFO1	400.1318840 MHz

F2 - Processing parameters

SI	32768
SF	400.1299651 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00







1

```
Current Data Parameters
NAME          BMVR176.HM
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070914
Time                9.43
INSTRUM            spect
PROBHD   5 mm BBI 1H/D-
PULPROG          zgpg30
TD                65536
SOLVENT             D2O
NS                16384
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631988 sec
RG                 2050
DW               20.800 usec
DE                6.00 usec
TE               300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                14.00 usec
PL1                0.00 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2                2.50 dB
PL12              23.66 dB
PL13              27.66 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB                    0
LB                 3.00 Hz
GB                     0
PC                 1.40
```