Supplementary Information

Synthesis of Heparin Oligosaccharides and Their Interaction with Eosinophil-Derived Neurotoxin

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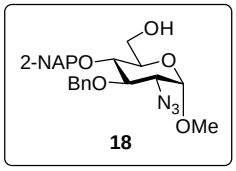
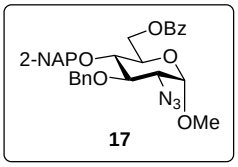


18

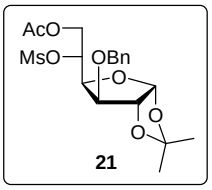
Methyl 2-azido-3-*O*-benzyl-2-deoxy-4-*O*-(2-naphthylmethyl)-α-D-glucopyranoside (18). BF_3/OEt_2 (2.29 mL, 18 mmol) was added to a solution of compound **14** (487 mg, 0.9 mmol) in methanol (5 mL) at 0 °C under N_2 atmosphere. The ice bath was removed, and the mixture was warmed up and kept stirring at 55 °C for 6 h. After cooling down to room temperature, CH_2Cl_2 (10 mL) was added to the solution, the reaction flask was immersed in an ice bath, and the mixture was neutralised with saturated $NaHCO_{3(aq)}$. The crude target material was extracted with CH_2Cl_2 (2 × 10 mL), and the combined organic layer was dried over $MgSO_4$, filtered and concentrated *in vacuo*. The residue was purified by flash column chromatography on silica gel (ethyl acetate/hexanes = 1/3) to afford the α-methyl glycoside **18** (251 mg, 62%). $[\alpha]_D^{23}$ +25.6 (*c* 7.4 in $CHCl_3$); IR ($CHCl_3$) ν 3483, 2924, 2101, 1052 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.87–7.80 (3H, m, Ar-H), 7.75 (1H, s, Ar-H), 7.53–7.46 (2H, m, Ar-H), 7.46–7.38 (6H, m, Ar-H), 5.00 (1H, d, *J* 11.3, C*H*$_2$Ph), 4.90 (1H, d, *J* 10.7,

C*H*$_2$Ph), 4.87 (1H, d, *J* 10.7, C*H*$_2$Ph), 4.82 (1H, d, *J* 11.3, CH$_2$Ph), 4.78 (1H, d, *J* 3.5, 1-H), 4.01 (1H, dd, *J* 8.6, 10.2, 3-H), 3.89–3.64 (4H, m, 4-H, 5-H, 6-H$_a$, 6-H$_b$), 3.37–3.45 (1H, m, 2-H), 3.41 (1H, s, CH$_3$); ^{13}C NMR (100 MHz, CDCl$_3$) δ 137.7 (C), 135.2 (C), 133.1 (C), 132.9 (C), 128.4 (CH), 128.2 (CH), 128.0 (CH), 127.8 (CH), 127.6 (CH), 126.5 (CH), 126.1 (CH), 126.0 (CH), 125.6 (CH), 98.6 (CH), 80.2 (CH), 77.9 (CH), 75.4 (CH$_2$), 75.0 (CH$_2$), 71.2 (CH), 63.6 (CH), 61.4 (CH$_2$), 55.1 (CH$_3$); HRMS (ESI, [M+Na]$^+$) *m/z* calc. for C$_{25}$H$_{27}$N$_3$O$_5$Na 472.1848, found 472.1841.



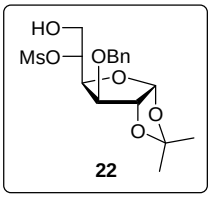
17

Methyl 2-azido-6-*O*-benzoyl-3-*O*-benzyl-2-deoxy-4-*O*-(2-naphthylmethyl)-α-D-glucopyranoside (17). Compound **18** (56 mg, 0.12 mmol) was dissolved in CH$_2$Cl$_2$ (1 mL) under N$_2$ atmosphere, and the reaction flask was cooled down in an ice bath. Et$_3$N (84 μL, 0.62 mmol), benzoyl chloride (29 μL, 0.25 mmol) and DMAP (1.5 mg, 1.2 μmol) were sequentially added to the solution, the ice bath was removed, and the mixture was kept stirring for 2 h. The resulting solution was concentrated *in vacuo* to provide a residue, which was purified by flash column chromatography on silica gel (ethyl acetate/hexanes = 1/4) to yield the 6-*O*-benzoylated **17** (68 mg, 94%). [α]$_D^{19}$ +128.0 (*c* 4.1 in CHCl$_3$); IR (CHCl$_3$) ν 2913, 2107, 1722, 1275, 1051 cm^{-1}; ^{1}H NMR (400 MHz, CDCl$_3$) δ 7.94 (2H, ddd, *J* 8.4, 1.4, 0.8, Bz-H), 7.79–7.69 (3H, m, Ar-H), 7.68 (1H, s, Ar-H), 7.56–7.48 (1H, m, Ar-H), 7.46–7.28 (10H, m, Ar-H), 5.04 (1H, d, *J* 11.1, C*H*$_2$Ar), 4.94, 4.90 (2H, ABq, *J* 10.5, C*H*$_2$Ar), 4.81 (1H, d, *J* 11.1, C*H*$_2$Ar), 4.82 (1H, d, *J* 3.5, 1-H), 4.62 (1H, dd, *J* 12.0, 1.9, 6-H$_a$), 4.50 (1H, dd, *J* 12.0, 4.1, 6-H$_b$), 4.08 (1H, dd, *J* 10.1, 9.1, 3-H), 4.04–3.97 (1H, m, 5-H), 3.73 (1H, t, *J* = 9.1, 4-H), 3.49 (1H, dd, *J* 10.1, 3.5, 2-H), 3.44 (1H, s, CH$_3$); ^{13}C NMR (100 MHz, CDCl$_3$) δ 166.1 (C), 137.6 (C), 134.8 (C), 133.1 (CH), 133.0 (C), 129.7 (C), 129.5 (CH), 128.5 (CH), 128.4 (CH), 128.2 (CH), 128.0 (CH), 127.9 (CH), 127.6 (CH), 126.8 (CH), 126.1 (CH), 126.0 (CH), 125.7 (CH), 98.5 (CH), 80.7 (CH), 78.0 (CH), 75.7 (CH$_2$), 75.2 (CH$_2$), 69.2 (CH), 63.7 (CH), 63.0 (CH$_2$), 55.3 (CH$_3$); HRMS (ESI, [M+Na]$^+$) *m/z* calc. for C$_{32}$H$_{31}$N$_3$O$_6$Na 576.2111, found 576.2119.



21

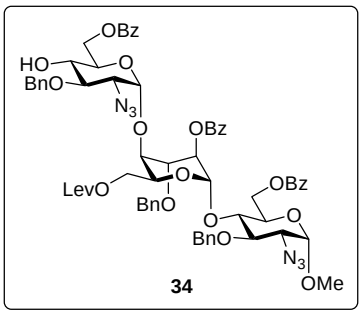
6-*O*-Acetyl-3-*O*-benzyl-1,2-*O*-isopropylidene-5-*O*-methanesulfonyl-α-D-glucofuranose (21). Acetic anhydride (0.1 mL, 1.1 mmol) was slowly added to a solution of the diol **20** (310 mg, 1 mmol) and pyridine (0.82 mL, 10 mmol) in CH_2Cl_2 (3.1 mL) at 0 °C under N_2 atmosphere. After stirring for 2 h, mesyl chloride (0.12 mL, 1.5 mmol) was added to the solution, and the mixture was continuously stirred at room temperature for 16 h. Water (5 mL) was added to quench the reaction, and the crude target material was extracted with ethyl acetate (3 × 15 mL). The combined organic layer was sequentially washed with 1 N $HCl_{(aq)}$, saturated $NaHCO_{3(aq)}$ and finally with brine. The organic portion was dried over $MgSO_4$, filtered and concentrated *in vacuo*. Purification of this residue *via* flash column chromatography (ethyl acetate/hexanes = 1/2) afforded the product **21** (270 mg, 63%) as a white solid. mp 124–125 °C (from EtOH); $[\alpha]_D^{22}$ –7.4 (*c* 0.7 in $CHCl_3$); IR ($CHCl_3$) ν 2988, 2934, 1746, 1358, 1228, 1177, 1076 cm–1; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39–7.23 (5H, m, Ph-H), 5.85 (1H, d, *J* 3.6, 1-H), 5.21 (1H, ddd, *J* 8.0, 6.6, 2.0, 5-H), 4.65 (4H, m, 2-H, 6-H_a, CH_2Ph), 4.33 (1H, dd, *J* 8.0, 3.1, 4-H), 4.20 (1H, dd, *J* 12.7, 6.6, 6-H_b), 4.07 (1H, d, *J* 3.1, 3-H), 2.98 (3H, s, CH_3), 2.06 (3H, s, CH_3), 1.46 (3H, s, CH_3), 1.28 (3H, s, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.3 (C), 137.1 (C), 128.5 (CH), 128.0 (CH), 112.3 (C), 105.3 (CH), 81.5 (CH), 81.1 (CH), 78.3 (CH), 75.4 (CH), 72.3 (CH_2), 63.5 (CH_2), 38.9 (CH_3), 26.9 (CH_3), 26.3 (CH_3), 20.1 (CH_3); HRMS (ESI, $[M+Na]^+$) *m/z* calc. for $C_{19}H_{26}O_9SNa$ 453.1195, found 453.1196.



22

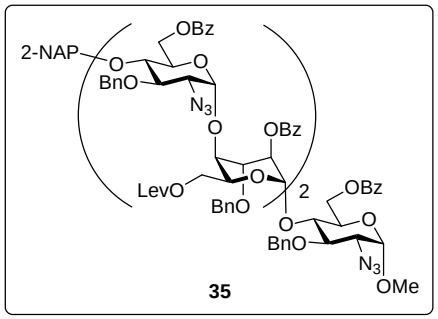
3-*O*-Benzyl-1,2-*O*-isopropylidene-5-*O*-methanesulfonyl-α-D-glucofuranose (22). Sodium methoxide (32 mg, 0.6 mmol) was added to a solution of compound **8** (145 mg, 0.3 mmol) in methanol (2.9 mL) at 0 °C under N_2 atmosphere. After stirring for 16 h, DOWEX 50WX4-200 ion-exchange resin was added to neutralise the reaction, the mixture was filtered, and the filtrate was concentrated *in vacuo*. The residue was purified by flash column

chromatography on silica gel (ethyl acetate/hexanes = 1/1) to furnish the 6-alcohol **22** (88 mg, 77%). $[\alpha]_D^{22}$ –25.6 (*c* 0.9 in $CHCl_3$); IR ($CHCl_3$) ν 3511, 2988, 2936, 1352, 1174, 1076 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.37–7.25 (5H, m, Ph-H), 5.86 (1H, d, *J* 3.7, 1-H), 5.06 (1H, ddd, *J* 8.0, 5.4, 2.6, 5-H), 4.62, 4.59 (2H, ABq, *J* 11.2, CH_2Ph), 4.58 (1H, d, *J* 3.7, 2-H), 4.33 (1H, dd, *J* 8.0, 3.1, 4-H), 4.07 (1H, d, *J* 3.1, 3-H), 4.08–4.0 (1H, m, 6-H_a), 3.85 (1H, dd, *J* 12.9, 5.4, 6-H_b), 3.02 (3H, s, CH_3SO_2), 1.47 (3H, s, CH_3), 1.29 (3H, s, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.0 (C), 128.4 (CH), 128.0 (CH), 112.2 (C), 105.1 (CH), 81.4 (CH), 81.1 (CH), 78.5 (CH), 78.3 (CH), 72.2 (CH_2), 62.5 (CH_2), 38.7 (CH_3), 26.6 (CH_3), 26.1 (CH_3); HRMS (ESI, $[M+Na]^+$) *m/z* calc. for $C_{17}H_{24}O_8SNa$ 411.1090, found 411.1096.



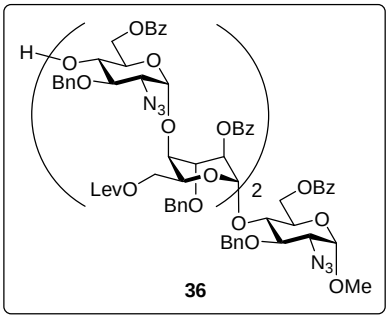
34

Methyl [2-azido-6-*O*-benzoyl-3-*O*-benzyl-2-deoxy-α-D-glucopyranosyl]-(1→4)-(2-*O*-benzoyl-3-*O*-benzyl-6-*O*-levulinyl-α-L-idopyranosyl)-(1→4)-2-azido-6-*O*-benzoyl-3-*O*-benzyl-2-deoxy-α-D-glucopyranoside (34). $[\alpha]_D^{25}$ +49.0 (*c* 0.96 in $CHCl_3$); IR ($CHCl_3$) ν 3442, 2922, 2101, 1716, 1645, 1447, 1267, 1056, 748, 709 cm^{-1}; Partial characterisation data was reported previously.[1]



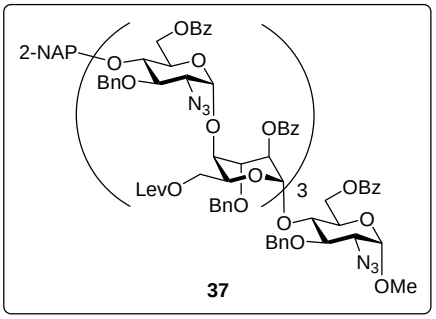
35

Compound 35. $[\alpha]_D^{28}$ +35.4 (*c* 0.96 in $CHCl_3$); IR ($CHCl_3$) ν 2922, 2101, 1716, 1261, 1107, 1023, 747, 709 cm^{-1}; HRMS (ESI, $[M+Na]^+$) *m/z* calc. for $C_{122}H_{121}N_9O_{32}Na$ 2246.8015, found 2246.8022; Partial characterisation data was reported previously.[1]



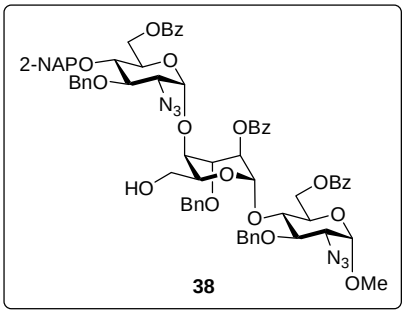
36

Compound 36. $[\alpha]_D^{27}$ +42.0 (*c* 0.32 in $CHCl_3$); IR ($CHCl_3$) ν 3461, 2922, 2101, 1716, 1267, 1100, 1023, 747, 702 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.04–7.97 (10H, m, Bz-H), 7.55–7.31 (18H, m, Bz-H), 7.30–7.16 (22H, m, Ar-H), 5.28 (1H, d, *J* 3.6, 1′-H), 5.22 (1H, d, *J* 3.2, 1‴-H), 5.18–5.16 (2H, m, 2-H, 2‴-H), 4.96 (1H, d, *J* 10.4, C*H*$_2$Ph), 4.79 (1H, d, *J* 11.6, C*H*$_2$Ph), 4.77–4.69 (9H, m, 1-H, 1″-H, 1⁗-H, 6⁗-H_a, 6⁗-H_b, 4 × C*H*$_2$Ph), 4.65–4.61 (2H, m, 6″-H_a, C*H*$_2$Ph), 4.58 (1H, d, *J* 10.8, C*H*$_2$Ph), 4.49 (1H, dd, *J* 11.0, 2.8, 6″-H_b), 4.44–4.39 (3H, m, 6-H_a, 5′-H, 5‴-H), 4.38–4.32 (3H, m, 6-H_b, 6‴-H_a, C*H*$_2$Ph), 4.26 (1H, dd, *J* 11.6, 7.6, 6′-H_a), 4.17 (1H, d, *J* 10.4, C*H*$_2$Ph), 4.13–4.02 (4H, m, 3′-H, 3‴-H, 6′-H_b, 6‴-H_b), 3.96–3.85 (5H, m, 3-H, 4-H, 4″-H, 5-H, 5″-H), 3.80 (1H, dt, *J* 9.6, 2.8, 5⁗-H), 3.75 (1H, t, *J* 4.0, 4‴-H), 3.66 (1H, dd, *J* 10.0, 8.4, 3″-H), 3.58 (1H, dd, *J* 9.6, 9.2, 3⁗-H), 3.47 (1H, dd, *J* 9.6, 4.8, 4⁗-H), 3.40–3.38 (1H, m, 2-H), 3.39 (3H, s, OCH_3), 3.27 (1H, dd, *J* 10.0, 3.6, 2″-H), 3.18 (1H, dd, *J* 10.0, 3.6, 2⁗-H), 3.13 (1H, d, *J* 4.8, 4⁗-OH), 2.63–2.55 (4H, m, 4 × OCOC*H*$_2$), 2.45–2.29 (4H, m, 4 × C*H*$_2$COCH3), 2.08 (3H, s, CH_3), 2.07 (3H, s, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 206.8 (C), 206.3 (C), 172.20 (C), 172.16 (C), 167.2 (C), 166.0 (C), 165.9 (C), 165.44 (C), 165.39 (C), 137.9 (C), 137.8 (C), 137.7 (C), 137.2 (C), 133.4 (CH), 133.3 (CH), 133.2 (CH), 133.1 (CH), 129.77 (CH), 129.68 (CH), 129.65 (CH), 129.42 (C), 129.38 (C), 128.54 (CH), 128.50 (CH), 128.44 (CH), 128.36 (CH), 128.34 (CH), 128.2 (CH), 128.1 (CH), 128.0 (CH), 127.9 (CH), 127.7 (CH), 127.5 (CH), 98.4 (CH), 98.2 (CH), 98.1 (CH), 98.0 (CH), 97.8 (CH), 79.7 (CH), 79.0 (CH), 78.7 (CH), 76.1 (CH), 75.3 (CH_2), 75.2 (CH_2), 75.1 (CH), 75.0 (CH_2), 74.2 (CH), 74.03 (CH), 73.91 (CH), 73.4 (CH), 73.3 (CH_2), 73.2 (CH_2), 71.4 (CH), 70.7 (CH), 70.4 (CH), 70.3 (CH), 69.1 (CH), 67.6 (CH), 67.2 (CH), 63.8 (CH), 63.08 (CH), 63.02 (CH_2), 62.9 (CH_2), 62.6 (CH_2), 62.2 (CH_2), 55.3 (CH_3), 37.8 (CH_2), 29.75 (CH_3), 29.70 (CH_3), 27.73 (CH_2), 27.67 (CH_2); HRMS (ESI, $[M+Na]^+$) *m/z* calc. for $C_{111}H_{113}N_9O_{32}Na$ 2106.7389, found 2106.7390.



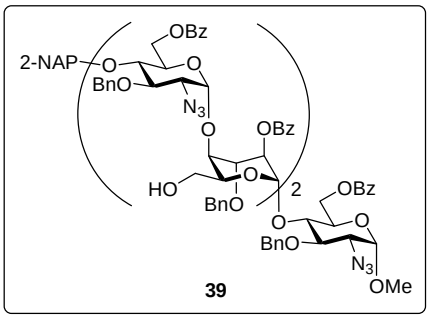
37

Compound 37. $[\alpha]_D^{27}$ +13.1 (*c* 0.16 in $CHCl_3$); IR ($CHCl_3$) ν 2915, 2108, 1723, 1267, 1107, 1017, 748, 709 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 8.04–7.98 (12H, m, Bz-H), 7.87 (2H, d, *J* 7.4, Bz-H), 7.76–7.73 (2H, m, Ar-H), 7.69 (1H, dd, *J* 6.1, 3.6, Ar-H), 7.60 (1H, s, Ar-H), 7.52–7.45 (4H, m, Ar-H), 7.43–7.39 (5H, m, Ar-H), 7.37–7.17 (48H, m, Ar-H), 5.28 (1H, d, *J* 3.6, 1′-H), 5.22 (1H, d, *J* 3.4, 1‴-H), 5.21 (1H, d, *J* 2.8, 1⁗-H), 5.17 (1H, t, *J* 3.6, 2′-H), 5.15 (1H, t, *J* 3.4, 2‴-H), 5.12 (1H, t, *J* 2.8, 2⁗-H), 4.99 (1H, d, *J* 10.3, C*H*$_2$Ar), 4.89 (1H, d, *J* 10.9, C*H*$_2$Ar), 4.80 (1H, d, *J* 11.5, C*H*$_2$Ar), 4.78–4.68 (12H, m, contains 2 anomeric protons), 4.66–4.59 (5H, m, contains 2 anomeric protons), 4.51–4.35 (10H, m), 4.33–4.23 (2H, m), 4.17 (2H, t, *J* 10.4), 4.11–3.96 (7H, m), 3.94–3.83 (8H, m), 3.75 (1H, t, *J* 3.9), 3.69–3.58 (5H, m), 3.41–3.38 (1H, m, 2-H), 3.39 (3H, s, OCH_3), 3.28–3.23 (3H, m, 2″-H, 2⁗-H, 2⁗′-H), 2.61–2.57 (2H, m), 2.56–2.52 (2H, m), 2.51–2.46 (2H, m), 2.45–2.40 (2H, m), 2.37–2.32 (4H, m), 2.07 (3H, s, CH_3), 2.03 (3H, s, CH_3), 1.99 (3H, s, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 206.14 (C), 206.06 (C), 172.1 (C), 172.0 (C), 171.9 (C), 165.9 (C), 165.7 (C), 165.3 (C), 137.8 (C), 137.63 (C), 137.57 (C), 137.3 (C), 137.1 (C), 134.5 (C), 133.2 (CH), 133.1 (CH), 133.0 (CH), 132.9 (CH), 129.6 (CH), 129.5 (CH), 128.6 (C), 128.5 (CH), 128.4 (C), 128.3 (C), 128.2 (CH), 128.10 (CH), 128.07 (CH), 127.9 (CH), 127.8 (CH), 127.7 (CH), 127.6 (CH), 127.5 (CH), 127.4 (CH), 126.9 (CH), 126.01 (CH), 125.94 (CH), 125.8 (CH), 98.4 (CH), 98.3 (CH), 97.9 (CH), 97.8 (CH), 97.7 (CH), 97.6 (CH), 80.5 (CH), 78.8 (CH), 78.7 (CH), 78.6 (CH), 77.2 (CH), 76.9 (CH), 75.14 (CH_2), 75.08 (CH_2), 74.9 (CH_2), 74.7 (CH), 74.6 (CH), 74.4 (CH), 73.8 (CH), 73.4 (CH), 73.2 (CH_2), 73.0 (CH_2), 72.95 (CH), 72.9 (CH_2), 70.2 (CH), 70.0 (CH), 69.7 (CH), 69.4 (CH), 68.9 (CH), 67.4 (CH), 67.0 (CH), 66.6 (CH), 63.6 (CH), 63.80 (CH), 63.75 (CH), 62.9 (CH_2), 62.6 (CH_2), 62.5 (CH_2), 62.4 (CH_2), 62.7 (CH_2), 62.5 (CH_2), 62.5 (CH), 62.3 (CH), 62.1 (CH), 55.2 (CH_3), 37.8 (CH_2), 37.61 (CH_2), 37.56 (CH_2), 37.5 (CH_2), 29.6 (CH_3), 29.5 (CH_3), 27.6 (CH_2), 27.52 (CH_2), 27.48 (CH_2); HRMS (MALDI, $[M+H]^+$) *m/z* calc. for $C_{167}H_{167}N_{12}O_{45}$ 3060.1148, found 3060.1272.



38

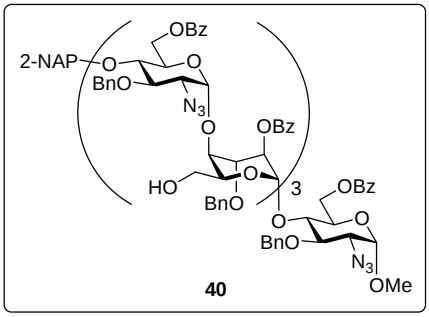
Methyl [2-azido-6-*O*-benzoyl-3-*O*-benzyl-2-deoxy-4-*O*-(2-naphthylmethyl)-α-D-glucopyranosyl]-(1→4)-(2-*O*-benzoyl-3-*O*-benzyl-α-L-idopyranosyl)-(1→4)-2-azido-6-*O*-benzoyl-3-*O*-benzyl-2-deoxy-α-D-glucopyranoside (38). $[\alpha]_D^{25}$ +55.6 (*c* 0.53 in $CHCl_3$); IR ($CHCl_3$) ν 3439, 2917, 2108, 1720, 1631, 1452, 1271, 772, 711 cm^{-1}; HRMS (ESI, $[M+Na]^+$) *m/z* calc. for $C_{72}H_{70}N_6O_{17}Na$ 1313.4695, found 1313.4697; Partial characterisation data was reported previously.[1]



39

Compound 39. $[\alpha]_D^{26}$ +41.1 (*c* 0.34 in $CHCl_3$); IR ($CHCl_3$) ν 3436, 2922, 2101, 1716, 1646, 1267, 1106, 1023, 748, 709 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 8.04 (2H, dd, *J* 7.8, 1.5, Bz-H), 8.02 (2H, dd, *J* 7.8, 1.6, Bz-H), 7.99 (2H, dd, *J* 8.2, 1.1, Bz-H), 7.96 (2H, dd, *J* 8.3, 1.2, Bz-H), 7.88 (2H, dd, *J* 8.2, 1.1, Bz-H), 7.75–7.73 (2H, m, Ar-H), 7.69–7.66 (1H, m, Ar-H), 7.59 (1H, s, Ar-H), 7.51–7.47 (3H, m, Ar-H), 7.43–7.41 (2H, m, Ar-H), 7.38–7.24 (33H, m, Ar-H), 7.22–7.17 (5H, m, Ar-H), 5.21 (1H, d, *J* 2.4, 1′-H), 5.19 (1H, t, *J* 2.4, 2′-H), 5.14–5.10 (2H, m, 1‴-H, 2‴-H), 4.89 (1H, d, *J* 10.8, C*H*$_2$Ar), 4.86–4.77 (4H, m, 4 × C*H*$_2$Ar), 4.75 (1H, d, *J* 3.8, 1″-H), 4.70 (1H, d, *J* 3.1, 1⁗-H), 4.73–4.66 (5H, m, 6‴-H_a, 4 × C*H*$_2$Ar), 4.58 (1H, d, *J* 11.1, 6-H_a), 4.49–4.40 (5H, m, 6″-H_a, 6″-H_b, 6‴-H_a, 2 × C*H*$_2$Ar), 4.34 (1H, dd, *J* 12.8, 2.5, 6-H_b), 4.29 (1H, d, *J* 10.5, C*H*$_2$Ar), 4.25–4.22 (1H, m, 5‴-H), 4.23 (1H, d, *J* 10.2, C*H*$_2$Ar), 4.13–4.08 (1H, m, 5′-H), 4.03–3.99 (4H, m, 5-H, 3′-H, 5″-H, 3‴-H), 3.95–3.89 (4H, m, 3-H, 4-H, 4″-H, 4⁗-H), 3.71 (1H, dd, *J* 10.1, 9.1, 3⁗-H), 3.65–3.62 (2H, m, 4′-H, 5‴-H), 3.61 (1H, dd, *J* 10.1, 9.3, 3″-H), 3.58–3.51 (2H, m, 6‴-H_a, 4‴-H), 3.50–3.45 (1H, m, 6‴-H_b), 3.49 (1H, dd, *J* 10.1, 3.6, 2-H), 3.43 (3H, s, OCH_3), 3.35–3.27 (1H, m, 6′-H_a), 3.33 (1H, dd, *J*

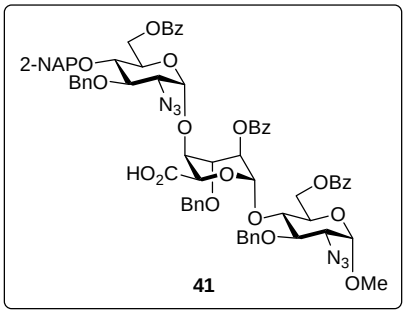
10.1, 3.7, 2″-H), 3.31 (1H, dd, *J* 10.1, 3.7, 2‴-H), 3.25–3.20 (1H, m, 6′-H_b); ^{13}C NMR (125 MHz, $CDCl_3$) δ 166.1 (C), 165.98 (C), 165.95 (C), 165.8 (C), 165.7 (C), 137.7 (C), 137.5 (C), 137.44 (C), 137.38 (C), 137.35 (C), 134.7 (C), 133.3 (CH), 133.2 (CH), 133.10 (CH), 133.07 (C), 133.0 (C), 129.8 (CH), 129.7 (CH), 129.6 (C), 129.59 (C), 129.5 (CH), 129.4 (C), 128.7 (CH), 128.5 (CH), 128.44 (CH), 128.42 (CH), 128.35 (CH), 128.11 (CH), 128.06 (CH), 128.0 (CH), 127.95 (CH), 127.8 (CH), 127.7 (CH), 127.0 (CH), 126.14 (CH), 126.05 (CH), 125.9 (CH), 98.4 (CH), 97.93 (CH), 97.86 (CH), 97.7 (CH), 97.0 (CH), 80.7 (CH), 79.1 (CH), 77.5 (CH), 75.4 (CH_2), 75.3 (CH_2), 75.2 (CH_2), 75.1 (CH_2), 74.5 (CH), 73.8 (CH), 73.6 (CH), 73.2 (CH), 73.1 (CH), 72.9 (CH_2), 72.8 (CH_2), 72.7 (CH), 70.2 (CH), 69.99 (CH), 69.5 (CH), 69.4 (CH), 69.2 (CH), 68.0 (CH), 67.7 (CH), 64.01 (CH), 63.94 (CH), 63.8 (CH), 63.0 (CH_2), 62.9 (CH_2), 62.8 (CH_2), 61.20 (CH_2), 61.17 (CH_2), 55.4 (CH_3); HRMS (MALDI, $[M+Na]^+$) *m/z* calc. for $C_{112}H_{109}N_9O_{28}Na$ 2050.7278, found 2050.7244.



40

Compound 40. $[\alpha]_D^{29}$ +36.3 (*c* 1.09 in $CHCl_3$); IR ($CHCl_3$) ν 3546, 2922, 2101, 1716, 1446, 1267, 1107, 1017, 748, 709 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.05–7.95 (12H, m, Bz-H), 7.88 (2H, d, *J* 7.6, Bz-H), 7.75–7.73 (2H, m, Ar-H), 7.69–7.67 (1H, m, Ar-H), 7.59 (1H, s, Ar-H), 7.52–7.46 (4H, m, Ar-H), 7.42–7.41 (2H, m, Ar-H), 7.38–7.24 (45H, m, Ar-H), 7.19–7.15 (8H, m, Ar-H), 5.21 (1H, s, 1′-H), 5.19 (1H, s, 2′-H), 5.16 (1H, s, 1‴-H), 5.14 (1H, s, 2‴-H), 5.11 (2H, s, 1⁗-H, 2⁗-H), 4.89 (1H, d, *J* 11.0, C*H*$_2$Ar), 4.85 (1H, d, *J* 11.5, C*H*$_2$Ar), 4.83 (1H, d, *J* 11.0, C*H*$_2$Ar), 4.82–4.79 (2H, m, contains 1 anomeric proton), 4.77 (1H, d, *J* 10.8, C*H*$_2$Ar), 4.75 (1H, d, *J* 3.5, 1″-H), 4.73–4.68 (8H, m, contains 2 anomeric protons), 4.58 (2H, t, *J* 12.7), 4.48–4.39 (6H, m), 4.36–4.23 (5H, m), 4.19–4.11 (4H, m), 4.04–4.00 (5H, m), 3.96–3.85 (6H, m), 3.70 (1H, t, *J* = 9.4), 3.66–3.53 (8H, m), 3.49 (1H, dd, *J* 9.9, 3.4), 3.48–3.44 (2H, m), 3.43 (3H, s, OCH_3), 3.36–3.29 (4H, m), 3.22–3.19 (2H, m); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.9 (C), 165.8 (C), 165.7 (C), 165.6 (C), 137.6 (C), 137.4 (C), 137.33 (C), 137.25 (C), 137.2 (C), 134.6 (C), 133.2 (CH), 133.1 (CH), 133.0 (C), 132.9 (CH), 129.7 (CH), 129.6 (CH), 129.5 (C), 129.4 (CH), 128.6 (CH), 128.3 (CH), 128.2 (CH),

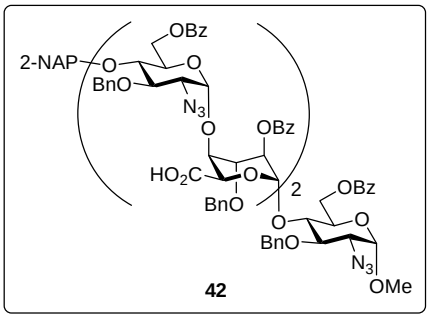
128.0 (CH), 127.9 (CH), 127.8 (CH), 127.7 (CH), 127.6 (CH), 126.1 (CH), 126.0 (CH), 125.8 (CH), 98.3 (CH), 97.83 (CH), 97.76 (CH), 97.7 (CH), 97.6 (CH), 96.9 (CH), 96.8 (CH), 80.6 (CH), 79.0 (CH), 77.4 (CH), 75.3 (CH_2), 75.2 (CH_2), 75.1 (CH_2), 75.0 (CH_2), 74.9 (CH_2), 74.3 (CH), 74.1 (CH), 74.0 (CH), 73.8 (CH), 73.6 (CH), 73.4 (CH), 73.0 (CH_2), 72.9 (CH), 72.8 (CH_2), 72.7 (CH_2), 72.6 (CH_2), 72.5 (CH), 70.1 (CH), 70.0 (CH), 69.9 (CH), 69.2 (CH), 69.1 (CH), 69.0 (CH), 67.8 (CH), 67.5 (CH), 67.4 (CH), 63.9 (CH), 63.7 (CH), 62.9 (CH_2), 62.8 (CH_2), 62.7 (CH_2), 62.6 (CH_2), 61.1 (CH_2), 55.3 (CH_3); HRMS (MALDI, $[M+Na]^+$) *m/z* calc. for $C_{152}H_{148}N_{12}O_{39}Na$ 2787.9864, found 2787.9851.



41

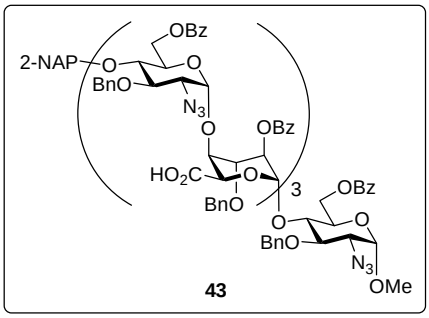
Methyl [2-azido-6-*O*-benzoyl-3-*O*-benzyl-2-deoxy-4-*O*-(2-naphthylmethyl)-α-D-glucopyranosyl]-(1→4)-(2-*O*-benzoyl-3-*O*-benzyl-α-L-idopyranosyluronic acid)-(1→4)-2-azido-6-*O*-benzoyl-3-*O*-benzyl-2-deoxy-α-D-glucopyranoside (41). $[\alpha]_D^{23}$ +60.5 (*c* 0.45 in $CHCl_3$); IR ($CHCl_3$) ν 3430, 2923, 2108, 1722, 1642, 1452, 1273, 1112, 1052, 1027, 754, 712 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.02 (2H, dd, *J* 7.1, 1.1, Bz-H), 7.98 (2H, dd, *J* 7.1, 1.1, Bz-H), 7.85 (2H, dd, *J* 7.4, 0.8, Bz-H), 7.82–7.76 (2H, m, Ar-H), 7.75–7.69 (1H, m, Ar-H), 7.64 (1H, s, Ar-H), 7.52–7.46 (4H, m, Ar-H), 7.42–7.38 (8H, m, Ar-H), 7.36–7.26 (13H, m, Ar-H), 7.20–7.18 (2H, m, Ar H), 5.51 (1H, d, *J* 2.0, 1′-H), 5.13 (1H, t, *J* 2.0, 2′-H), 5.00 (1H, d, *J* 10.3, C*H*$_2$Ar), 4.97 (1H, d, *J* 2.2, 5′-H), 4.92 (1H, d, *J* 11.0, C*H*$_2$Ar), 4.88 (1H, d, *J* 11.6, C*H*$_2$Ar), 4.80 (1H, d, *J* 11.6, C*H*$_2$Ar), 4.77 (1H, d, *J* 11.0, C*H*$_2$Ar), 4.72–4.78 (4H, m, 1-H, 1″-H, 6″-H_a, C*H*$_2$Ar), 4.66 (1H, d, *J* 11.5, 6-H_a), 4.48 (1H, dd, *J* 11.5, 3.4, 6-H_b), 4.37 (1H, dd, *J* 12.3, 2.2, 6″-H_b), 4.35 (1H, d, *J* 10.6, C*H*$_2$Ar), 4.13 (1H, d, *J* 10.5, C*H*$_2$Ar), 4.17–4.07 (3H, m, 3′-H, 4′-H, 5″-H), 4.00–3.89 (3H, m, 3-H, 4-H, 5-H), 3.67–3.58 (2H, m, 3″-H, 4″-H), 3.42 (1H, d, *J* 3.7, 2-H), 3.40 (3H, s, OCH_3), 3.21 (1H, dd, *J* 9.1, 3.8, 2″-H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.3 (C), 166.3 (C), 166.0 (C), 165.4 (C), 137.6 (C), 137.2 (C), 135.0 (C), 133.2 (CH), 133.14 (C), 133.07 (CH), 133.05 (C), 129.9 (CH), 129.7 (CH), 129.5 (CH), 129.4 (C), 128.6 (CH), 128.4 (CH), 128.33 (CH), 128.26 (CH), 128.2 (CH), 128.0 (CH), 127.9 (CH), 127.73 (CH), 127.69 (CH), 127.0 (CH), 126.2 (CH), 126.1 (CH), 98.9 (CH),

98.6 (CH), 98.5 (CH), 80.3 (CH), 79.0 (CH), 77.6 (CH), 76.1 (CH), 75.1 (2 × CH_2), 74.9 (CH_2), 74.7 (CH), 73.2 (CH), 72.7 (CH_2), 70.2 (CH), 69.0 (2 × CH), 68.5 (CH), 63.8 (2 × CH), 63.0 (CH_2), 62.6 (CH_2), 55.3 (CH_3); HRMS (ESI, $[M+Na]^+$) *m/z* calc. for $C_{72}H_{68}N_6O_{18}Na$ 1327.4488, found 1327.4493; Partial characterisation data was reported previously.[1]



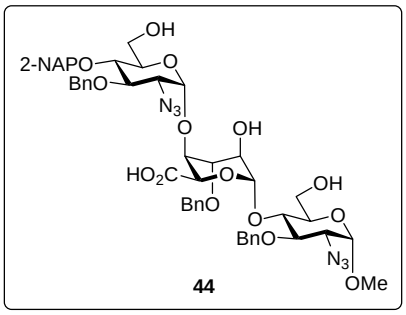
42

Compound 42. $[\alpha]_D^{21}$ +29.7 (*c* 5 in $CHCl_3$); IR ($CHCl_3$) ν 3449, 2925, 2109, 1722, 1274, 753, 712 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.08–7.93 (10H, m, Bz-H), 7.85 (2H, d, *J* 7.5, Ar-H), 7.82–7.74 (2H, m, Ar-H), 7.74–7.67 (2H, m, Ar-H), 7.63 (1H, s, Ar-H), 7.53 (36H, m, Ar-H), 7.18–7.12 (4H, m, Ar-H), 5.523 (1H, s, 1′-H), 5.516 (1H, s, 1‴-H), 5.16 (1H, t, *J* 3.7, 2′-H), 5.13–5.08 (1H, m, 2‴-H), 4.96 (1H, d, *J* 10.6, C*H*$_2$Ar), 4.94–4.90 (2H, m, 5′-H, 5‴-H), 4.88 (1H, d, *J* 10.6, C*H*$_2$Ar), 4.86–4.80 (2H, m, 2 × C*H*$_2$Ar), 4.86–4.65 (10H, m, 1′-H, 1″-H, 1⁗-H, 6-H_a, 6″-H_a, 6⁗-H_a, 4 × C*H*$_2$Ar), 4.58 (1H, d, *J* 10.8, C*H*$_2$Ar), 4.47 (1H, dd, *J* 12.0, 4.0, 6″-H_b), 4.39 (2H, t, *J* 11.9, 6-H_b, 6⁗-H_b), 4.22 (1H, d, *J* 10.8, C*H*$_2$Ar), 4.15 (1H, d, *J* 11.2, C*H*$_2$Ar), 4.13–4.02 (8H, m, 4-H, 5-H, 3′-H, 4′-H, 3‴-H, 4‴-H, 5⁗-H, C*H*$_2$Ar), 4.02–3.83 (3H, m, 3-H, 4″-H, 5″-H), 3.75–3.67 (1H, m, 3″-H), 3.67–3.57 (2H, m, 3⁗-H, 4⁗-H), 3.47–3.41 (1H, m, 2-H), 3.40 (3H, s, OCH_3), 3.25–3.15 (2H, m, 2"-H, 2⁗-H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.3 (C), 169.8 (C), 166.4 (C), 166.0 (C), 165.5 (C), 165.4 (C), 137.6 (C), 137.4 (C), 137.1 (C), 137.0 (C), 135.0 (C), 133.4 (CH), 133.1 (CH), 133.0 (CH), 129.84 (CH), 129.78 (CH), 129.7 (CH), 129.6 (CH), 129.5 (C), 129.3 (C), 128.6 (CH), 128.5 (CH), 128.4 (CH), 128.3 (CH), 128.1 (CH), 128.0 (CH), 127.8 (CH), 127.7 (CH), 127.0 (CH), 126.2 (CH), 126.0 (CH), 98.6 (CH), 98.4 (CH), 98.3 (CH), 97.6 (CH), 97.5 (CH), 80.3 (CH), 78.9 (CH), 78.5 (CH), 77.5 (CH), 77.2 (CH), 76.4 (CH), 75.4 (CH), 75.2 (CH_2), 75.1 (CH_2), 75.9 (CH_2), 74.8 (CH), 74.6 (CH_2), 73.7 (CH), 73.0 (CH), 72.8 (CH_2), 72.7 (CH_2), 70.1 (CH), 69.7 (CH), 69.6 (CH), 69.1 (2 × CH), 68.8 (CH), 68.3 (CH), 63.9 (CH), 63.74 (CH), 63.67 (CH), 63.0 (CH_2), 62.6 (2 × CH_2), 55.3 (CH_3); HRMS (ESI, $[M+Na]^+$) *m/z* calc. for $C_{112}H_{105}N_9O_{30}Na$ 2078.6865, found 2078.6863.



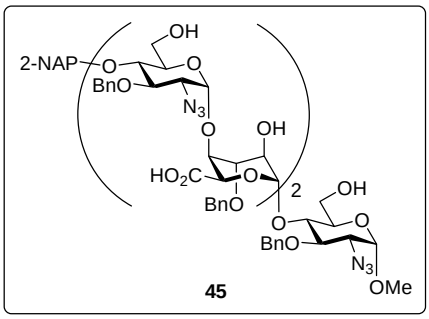
43

Compound 43. $[\alpha]_D^{20}$ +31.6 (*c* 1.0 in $CHCl_3$); IR ($CHCl_3$) ν 3469, 2922, 2109, 1720, 1270, 1069, 751, 712 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 8.09–7.90 (10H, m, Bz-H), 7.88–7.74 (4H, m, Bz-H), 7.74–7.56 (3H, m, Ar-H), 7.56–6.95 (58H, m, Ar-H), 5.63–4.98 (3H, m, 1′-H, 1‴-H, 1⁗-H), 5.26–5.05 (6H, m, 2′-H, 2‴-H, 2⁗-H, 5′-H, 5‴-H, 5⁗-H), 5.04–4.62 (20H, m, contains 4 anomeric protons), 4.61–4.52 (3H, m), 4.52–4.43 (2H, m), 4.43–4.31 (3H, m), 4.24–3.82 (15H, m), 3.77–3.51 (3H, m), 3.48–3.33 (1H, m), 3.39 (3H, s, OCH_3), 3.30–3.11 (3H, m); HRMS (ESI, $[M+Na]^+$) *m/z* calc. for $C_{152}H_{142}N_{12}O_{42}Na$ 2829.9242, found 2829.9148.



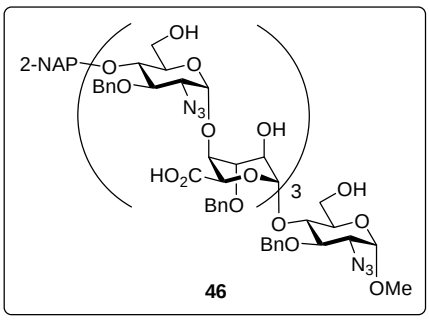
44

Methyl [2-azido-3-*O*-benzyl-2-deoxy-4-*O*-(2-naphthylmethyl)-α-D-glucopyranosyl]-(1→4)-(3-*O*-benzyl-α-L-idopyranosyluronic acid)-(1→4)-2-azido-3-*O*-benzyl-2-deoxy-α-D-glucopyranoside (44). $[\alpha]_D^{22}$ +13.7 (*c* 1.3 in MeOH); IR ($CHCl_3$) ν 3440, 2925, 2111, 1727, 1028, 753, 698 cm^{-1}; HRMS (ESI, $[M+Na]^+$) *m/z* calc. for $C_{51}H_{56}N_6O_{15}Na$ 1015.3701, found 1015.3706; Partial characterisation data was reported previously.[1]



45

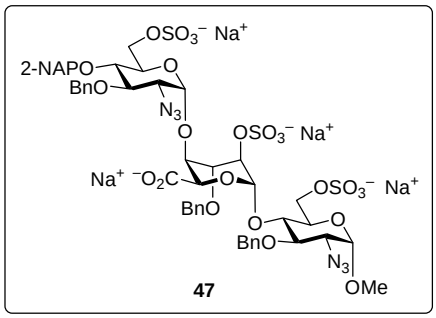
Compound 45. $[\alpha]_D^{21}$ +9.3 (*c* 1.3 in $CHCl_3$); IR ($CHCl_3$) ν 3430, 2927, 2112, 1633, 1028 cm^{-1}; ^1H NMR (500 MHz, CD_3OD) δ 7.82–7.62 (4H, m, Ar-H), 7.48–7.13 (28H, m, Ar-H), 5.68 (1H, s, 1′-H), 5.36 (1H, s, 1‴-H), 5.08–4.95 (3H, m, contains 2 anomeric protons), 4.93–4.65 (8H, m, contains 1 anomeric proton), 4.59–4.44 (3H, m), 4.39 (1H, s), 4.30–4.18 (4H, m), 4.02–3.70 (14H, m), 3.58 (6H, m), 3.43–3.37 (1H, m), 3.36 (3H, s, OCH_3), 3.23–3.14 (1H, m); ^{13}C NMR (125 MHz, CD_3OD) δ 177.9 (C), 176.2 (C), 139.8 (C), 139.6 (C), 139.51 (C), 139.46 (C), 137.2 (C), 137.0 (C), 134.8 (C), 134.6 (C), 129.7 (CH), 129.5 (CH), 129.3 (CH), 129.2 (CH), 128.99 (CH), 128.96 (CH), 128.9 (CH), 128.7 (CH), 127.9 (CH), 127.4 (CH), 127.2 (CH), 102.4 (CH), 100.2 (CH), 97.7 (CH), 97.3 (CH), 97.0 (CH), 82.3 (CH), 80.0 (CH), 79.7 (CH), 77.7 (CH), 77.4 (CH), 77.0 (CH), 76.4 (2 × CH_2), 76.3 (CH_2), 75.1 (CH), 74.1 (CH), 74.8 (CH_2), 74.7 (CH_2), 73.7 (CH_2), 73.5 (CH), 73.2 (CH), 72.9 (CH_2), 72.1 (CH), 71.8 (CH), 71.6 (CH), 70.4 (CH), 70.2 (CH), 68.9 (CH), 68.8 (CH), 68.6 (CH), 65.6 (CH), 64.8 (CH), 63.1 (CH_2), 62.1 (CH_2), 62.0 (CH_2), 55.6 (CH_3); HRMS (ESI, $[M+Na]^+$) *m/z* calc. for $C_{77}H_{85}N_9O_{25}Na$ 1558.5554, found 1558.5552.



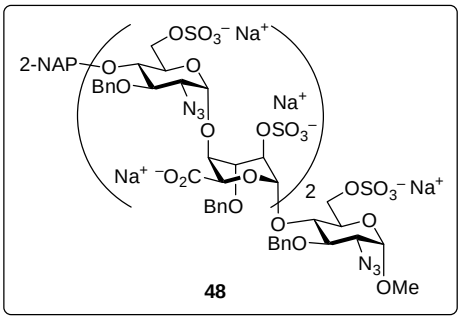
46

Compound 46. $[\alpha]_D^{21}$ –13.3 (*c* 4.0 in MeOH); IR ($CHCl_3$) ν 3436, 2925, 2111, 1606, 1027, 770, 698 cm^{-1}; ^1H NMR (600 MHz, CD_3OD) δ 7.84–7.73 (3H, m, Ar-H), 7.70 (1H, s, Ar-H), 7.48–7.13 (38H, m, Ar-H), 5.55–5.40 (2H, br s, anomeric protons), 5.27 (1H, br s, anomeric proton), 5.11–5.00 (3H, m, contains 1 anomeric proton), 5.00–4.79 (8H, m), 4.79–4.58 (13H, m, contains 3 anomeric protons), 4.51 (1H, d, *J* 12.5, C*H*$_2$Ar), 4.31–4.01 (7H, m), 3.99–3.70 (20H, m), 3.68–3.53 (5H, m), 3.53–3.45 (1H, m), 3.35 (3H, s, OCH_3), 3.28–3.22

(1H, m); HRMS (ESI, $[M+Na]^+$) *m/z* calc. for $C_{103}H_{114}N_{12}O_{35}Na$ 2101.7407, found 2101.7444.

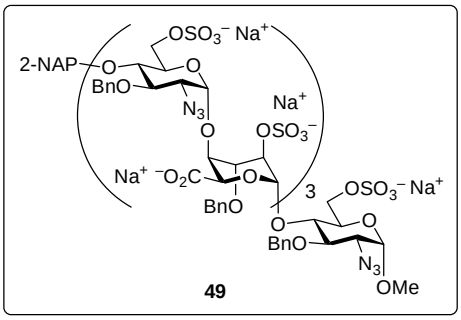


Methyl [2-azido-3-*O*-benzyl-2-deoxy-4-*O*-(2-naphthylmethyl)-6-*O*-sodium sulfonato-α-D-glucopyranosyl]-(1→4)-(sodium 3-*O*-benzyl-2-*O*-sodium sulfonato-α-L-idopyranosyluronate)-(1→4)-2-azido-3-*O*-benzyl-2-deoxy-6-*O*-sodium sulfonato-α-D-glucopyranoside (47). $[\alpha]_D^{21}$ +1.1 (*c* 0.9 in MeOH); ^{1}H NMR (600 MHz, CD_3OD) δ 7.82–7.79 (1H, m, Ar-H), 7.79–7.72 (3H, m, Ar-H), 7.49–7.40 (5H, m, Ar-H), 7.37–7.33 (2H, m), 7.33–7.15 (11H, m, Ar-H), 5.72 (1H, s, 1′-H), 5.13 (1H, d, *J* 2.9, 1-H), 5.02 (1H, d, *J* 10.4, C*H*$_2$Ph), 4.98–4.89 (3H, m, 5′-H, 2 × C*H*$_2$Ph), 4.87 (1H, d, *J* 10.6, C*H*$_2$Ph), 4.83 (1H, d, *J* 11.4, C*H*$_2$Ph), 4.81 (1H, d, *J* 10.8, C*H*$_2$Ph), 4.76 (1H, d, *J* 2.8, 1″-H), 4.69 (1H, d, *J* 10.8, C*H*$_2$Ph), 4.60 (1H, s, 2′-H), 4.53 (1H, dd, *J* 11.9, 2.9, 6-H$_a$), 4.43 (1H, dd, *J* 11.0, 2.2, 6″-H$_a$), 4.35 (1H, d, *J* 11.0, 6″-H$_b$), 4.22 (4H, m, 3′-H, 4′-H, 6-H$_b$, C*H*$_2$Ph), 4.12–4.02 (2H, m, 5-H, 4″-H), 3.96–3.87 (2H, m, 3-H, 5″-H), 3.79–3.70 (2H, m, 4-H, 3″-H), 3.69–3.63 (1H, m, 2-H), 3.42 (3H, s, CH_3), 3.17 (1H, dd, *J* 10.7, 2.5, 2″-H); ^{13}C NMR (150 MHz, CD_3OD) δ 176.0 (C), 140.1 (C), 139.5 (C), 139.3 (C), 137.4 (C), 134.9 (C), 134.6 (C), 129.9 (CH), 129.7 (CH), 129.54 (CH), 129.50 (CH), 129.3 (CH), 129.14 (CH), 129.07 (CH), 128.94 (CH), 128.87 (CH), 128.8 (CH), 128.6 (CH), 128.4 (CH), 127.9 (CH), 127.1 (CH), 127.0 (CH), 100.1 (CH), 98.9 (CH), 96.6 (CH), 82.5 (CH), 79.9 (CH), 79.3 (CH), 76.7 (CH_2), 76.5 (CH_2), 76.2 (CH_2), 73.8 (CH), 73.5 (CH_2), 72.8 (CH), 72.7 (CH), 72.3 (CH), 71.6 (CH), 71.4 (CH), 70.7 (CH), 68.3 (CH_2), 67.5 (CH_2), 65.8 (CH), 64.9 (CH), 55.9 (CH_3); HRMS (ESI, $[M-3H+2Na]^-$) *m/z* calcd for $C_{51}H_{53}N_6O_{24}S_3Na_2$ 1275.2074, found 1275.2087.



48

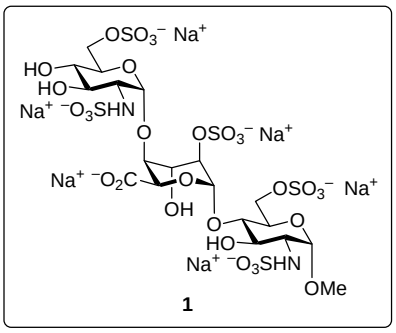
Compound 48. $[\alpha]_D^{21}$ +10.3 (*c* 1.0 in MeOD); ^{1}H NMR (500 MHz, D_2O) δ 7.92–7.84 (3H, m, Ar-H), 7.81 (1H, s, Ar-H), 7.56–7.48 (3H, m, Ar-H), 7.47–7.41 (4H, m, Ar-H), 7.40–7.35 (21H, m, Ar-H), 5.28 (1H, s, 1′-H), 5.23 (1H, s, 1‴-H), 5.02 (1H, d, *J* 3.6, 1-H), 4.94 (1H, d, *J* 3.2, 1″-H), 4.93–4.87 (1H, m), 4.86–4.79 (4H, m), 4.84 (1H, d, *J* 3.6, 1⁗-H), 4.78–4.67 (3H, m), 4.65 (1H, d, *J* 11.8, C*H*$_2$Ar), 4.55 (1H, d, *J* 11.3, C*H*$_2$Ar), 4.51–4.44 (3H, m), 4.40 (1H, d, *J* 9.7), 4.37–4.22 (5H, m), 4.22–4.11 (4H, m), 4.10–4.08 (2H, m), 3.98–384 (7H, m), 3.76 (1H, dd, *J* 10.2, 9.2, 3-H), 3.70 (1H, t, *J* 9.2, 4-H), 3.56 (1H, dd, *J* 10.2, 3.6, 2-H), 3.48–3.42 (2H, m, 2″-H, 2⁗-H), 3.45 (3H, s, OCH_3), 3.32–3.20 (1H, m); ^{13}C NMR (125 MHz, D_2O) δ 174.41 (C), 174.36 (C), 137.2 (C), 136.9 (C), 136.8 (C), 136.5 (C), 134.4 (C), 132.83 (C), 132.81 (C), 128.91 (CH), 128.85 (CH), 128.68 (CH), 128.66 (CH), 128.63 (CH), 128.57 (CH), 128.5 (CH), 128.4 (CH), 128.33 (CH), 128.28 (CH), 128.2 (CH), 128.1 (CH), 127.9 (CH), 127.63 (CH), 127.57 (CH), 126.6 (CH), 126.43 (CH), 126.39 (CH), 98.1 (CH), 97.6 (CH), 97.5 (CH), 93.92 (CH), 93.86 (CH), 79.7 (CH), 78.4 (CH), 78.2 (CH), 76.8 (CH), 75.5 (CH_2), 75.3 (CH_2), 75.2 (CH_2), 74.2 (CH), 72.5 (CH), 72.2 (CH_2), 72.1 (CH_2), 71.4 (CH), 71.3 (CH), 70.5 (CH), 69.9 (CH), 69.4 (CH), 69.2 (CH), 69.13 (CH), 69.06 (CH), 68.2 (CH), 67.6 (CH), 66.5 (CH_2), 66.3 (CH_2), 65.9 (CH_2), 63.14 (CH), 63.07 (CH), 62.94 (CH), 54.8 (CH_3); HRMS (ESI, $[M+Na]^+$) *m/z* calc. for $C_{77}H_{80}N_9O_{40}S_5Na$ 2068.2492, found 2068.2500.



49

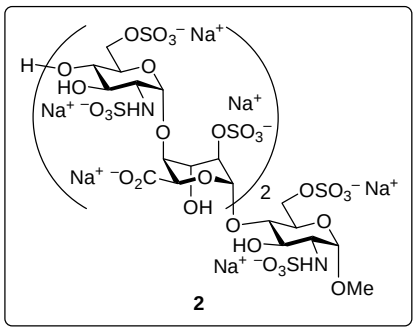
Compound 49. $[\alpha]_D^{22}$ +2.3 (*c* 2.1 in MeOH); ^{1}H NMR (600 MHz, CD_3OD) δ 7.85–7.81 (1H, m, Ar-H), 7.80–7.75 (3H, m, Ar-H), 7.50–7.44 (3H, m, Ar-H), 7.44–7.39 (6H, m,

Ar-H), 7.38–7.34 (2H, m, Ar-H), 7.34–7.23 (18H, m, Ar H), 7.23–7.16 (3H, m, Ar-H), 5.92 (1H, s, anomeric proton), 5.81 (1H, s, anomeric proton), 5.50 (1H, s, anomeric proton), 5.27 (1H, d, *J* 3.5, anomeric proton), 5.19 (1H, s, anomeric proton), 5.14 (1H, d, *J* 11.1, C*H*$_2$Ar), 5.10–4.95 (7H, m, contains 1 anomeric proton), 4.89–4.80 (3H, m), 4.75 (1H, d, *J* 3.5, anomeric proton), 4.73–4.52 (10H, m), 4.48–4.39 (3H, m), 4.38–4.28 (5H, m), 4.28–4.22 (2H, m), 4.22–4.16 (3H, m), 4.16–4.13 (2H, m), 4.12–4.00 (5H, m), 3.96–3.87 (2H, m), 3.84–3.75 (4H, m), 3.67–3.63 (1H, m), 3.61–3.56 (2H, m), 3.56–3.42 (2H, m), 3.37 (3H, s, OCH_3), 3.22–3.10 (1H, m, 2‴-H); HRMS (ESI, $[M–10H+7Na]^{3–}$) *m/z* calc. for $C_{103}H_{104}N_{12}O_{56}S_7Na_7$ 929.7663, found 929.7661.



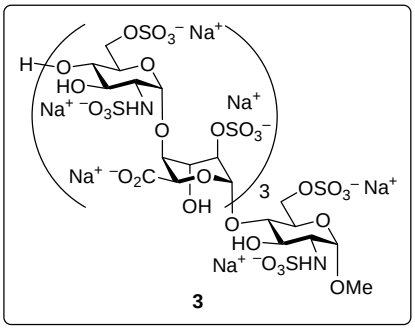
1

Methyl (2-deoxy-2-sodium sulfonatamido-6-*O*-sodium sulfonato-α-D-glucopyranosyl)-(1→4)-(sodium 2-*O*-sodium sulfonato-α-L-idopyranosyluronate)-(1→4)-2-deoxy-2-sodium sulfonatamido-6-*O*-sodium sulfonato-α-D-glucopyranoside (1). ^{1}H NMR (600 MHz, D_2O) δ 5.37 (1H, s, 1-H), 5.17 (1H, s, 1′-H), 4.97 (1H, s, 1″-H), 4.79–4.70 (1H, m, 5′-H), 4.36–4.21 (3H, m, 6-H_a, 2′-H, 6″-H_a), 4.20–4.09 (2H, m, 6-H_b, 6″-H_b), 4.09–4.01 (1H, m, 5-H), 3.95–3.91 (1H, m, 5″-H), 3.77–3.54 (3H, m, 3-H, 4-H, 3″-H), 3.54–3.46 (1H, m, 4″-H), 3.42 (1H, dd, *J* 9.1, 3.0, 3′-H), 3.39–3.32 (4H, m, OCH_3, 4′-H), 3.25–3.15 (2H, m, 2-H, 2″-H); HRMS (ESI, $[M–6H+4Na]^{2–}$) *m/z* calc. for $C_{19}H_{28}N_2O_{30}S_5Na_4$ 507.9460, found 507.9247.



2

Compound 2. ^{1}H NMR (600 MHz, D_2O) δ 5.30–5.18 (2H, m, anomeric protons), 5.08–4.95 (2H, m, anomeric protons), 4.81 (1H, s, anomeric proton), 4.33–4.21 (8H, m), 4.13–3.97 (5H, m), 3.86–3.70 (4H, m), 3.95–3.74 (4H, m), 3.74–3.49 (5H, m), 3.49–3.42 (1H, m), 3.30 (3H, s, CH_3), 3.20–3.08 (3H, m, 2-H, 2‴-H, 2⁗-H); HRMS (ESI, $[M–5H+3Na]^{2–}$) *m/z* calc. for $C_{31}H_{48}N_3O_{49}S_8Na_3$ 785.4407, found 785.4289.

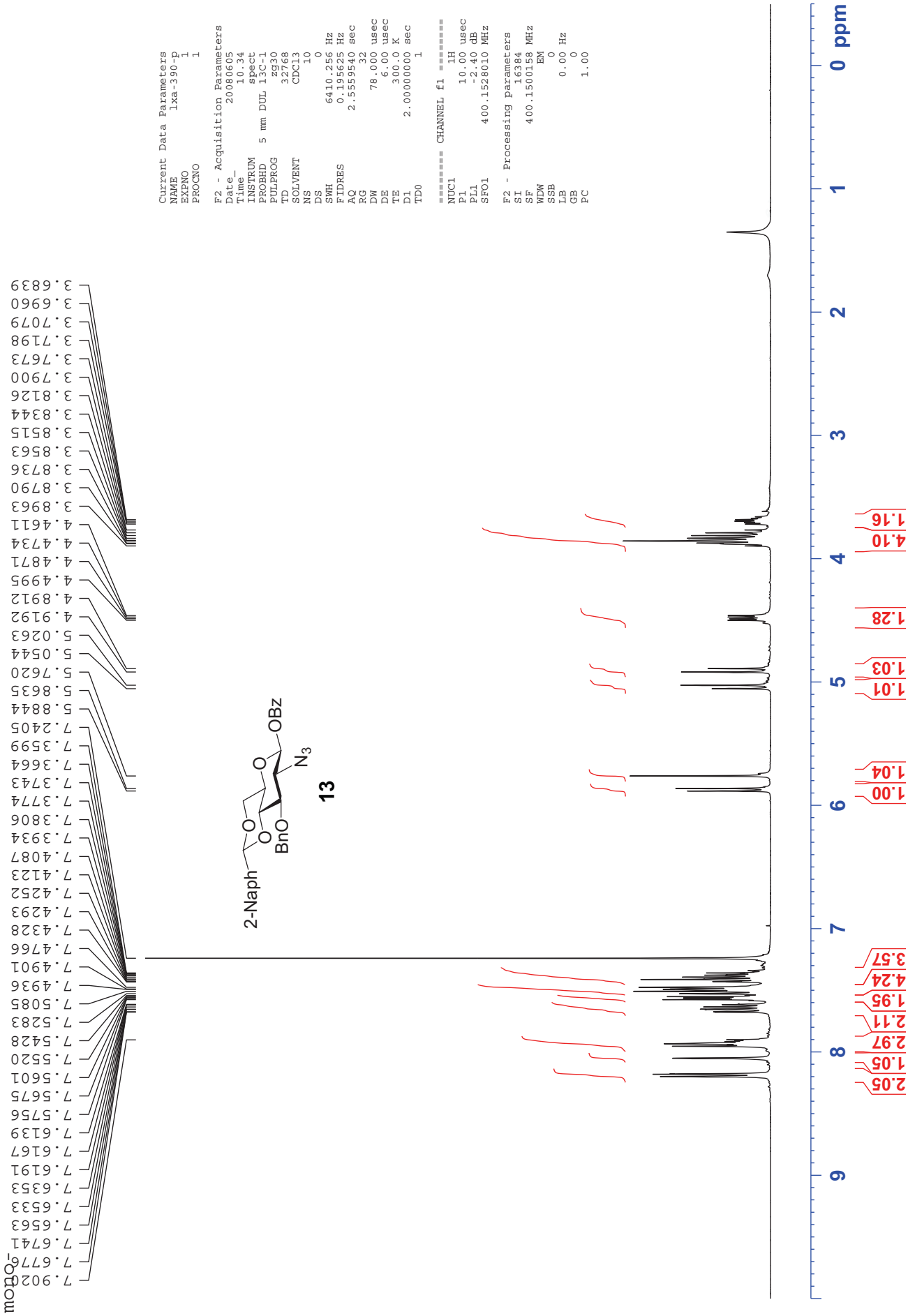


3

Compound (3). ^{1}H NMR (600 MHz, D_2O) δ 5.42–5.21 (3H, m, anomeric protons), 5.20–5.01 (3H, m, anomeric protons), 4.97–4.85 (2H, m, contains 1 anomeric proton), 4.36–3.95 (21H, m), 3.94–3.76 (6H, m), 3.74–3.49 (9H, m), 3.49–3.42 (1H, m), 3.31 (3H, s, CH_3), 3.38–3.34 (1H, m), 3.31–3.17 (3H, m); HRMS (ESI, $[M–7H+5Na]^{2–}$) *m/z* calc. for $C_{43}H_{65}N_4O_{68}S_{11}Na_5$ 1095.9083, found 1095.9166; $[M–5H+2Na]^{3–}$ *m/z* calc. for $C_{43}H_{67}N_4O_{68}S_{11}Na_2$ 708.2877, found 708.2914.

Reference:

1 J.-C. Lee, X.-A. Lu, S. S. Kulkarni, Y.-S. Wen and S.-C. Hung, *J. Am. Chem. Soc.*, 2004, **126**, 476.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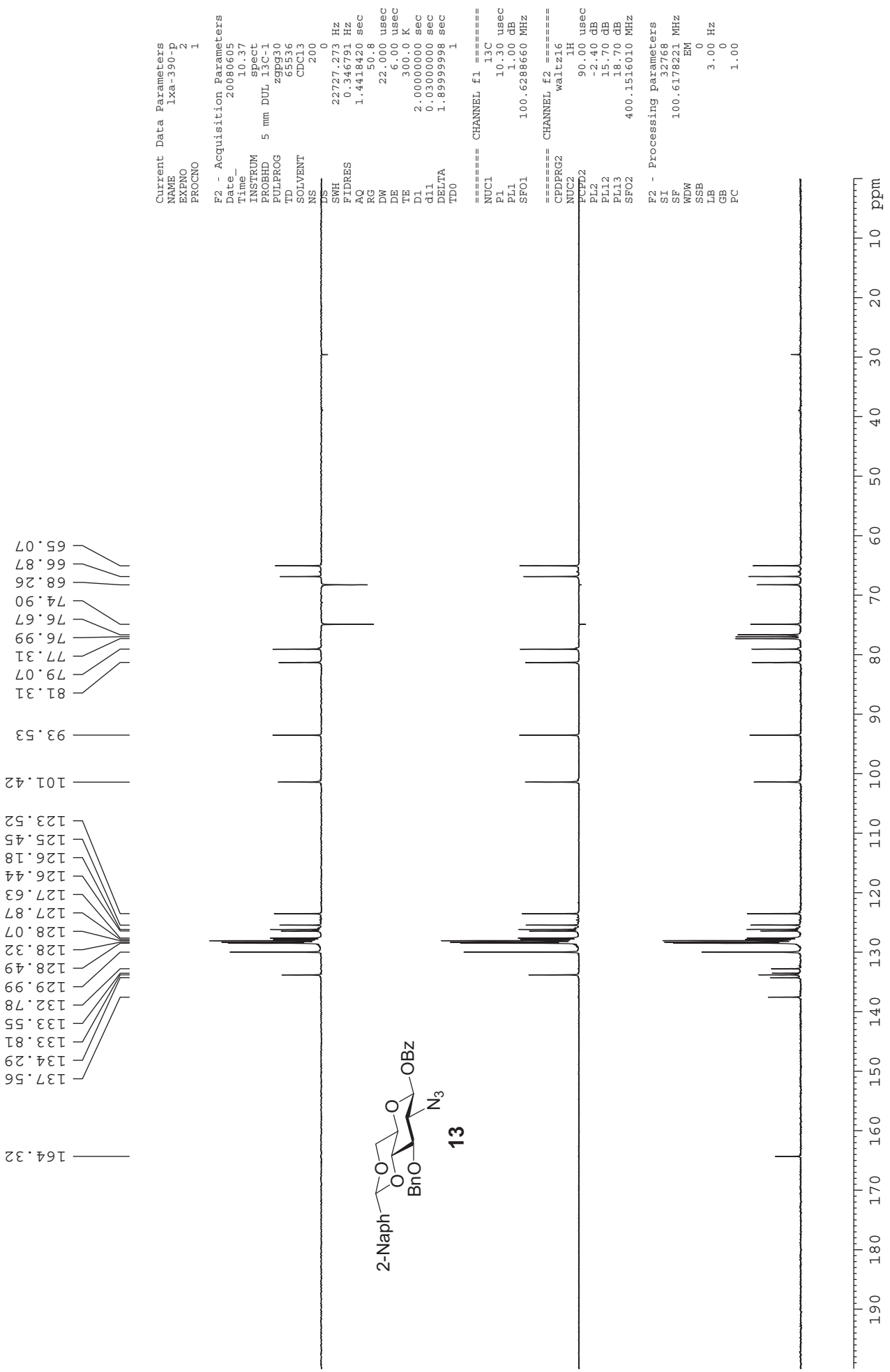
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PL13 18.70 dB
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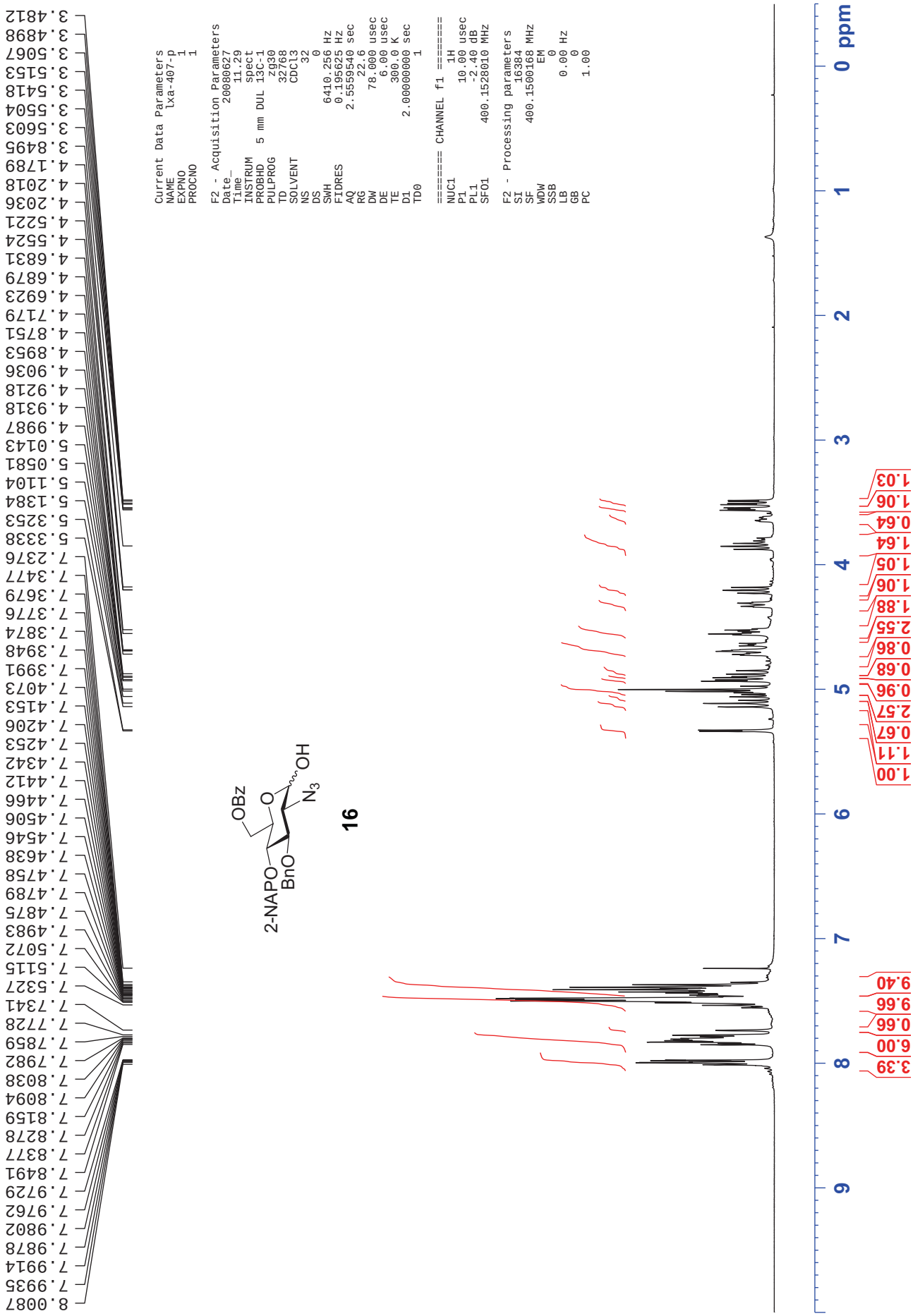
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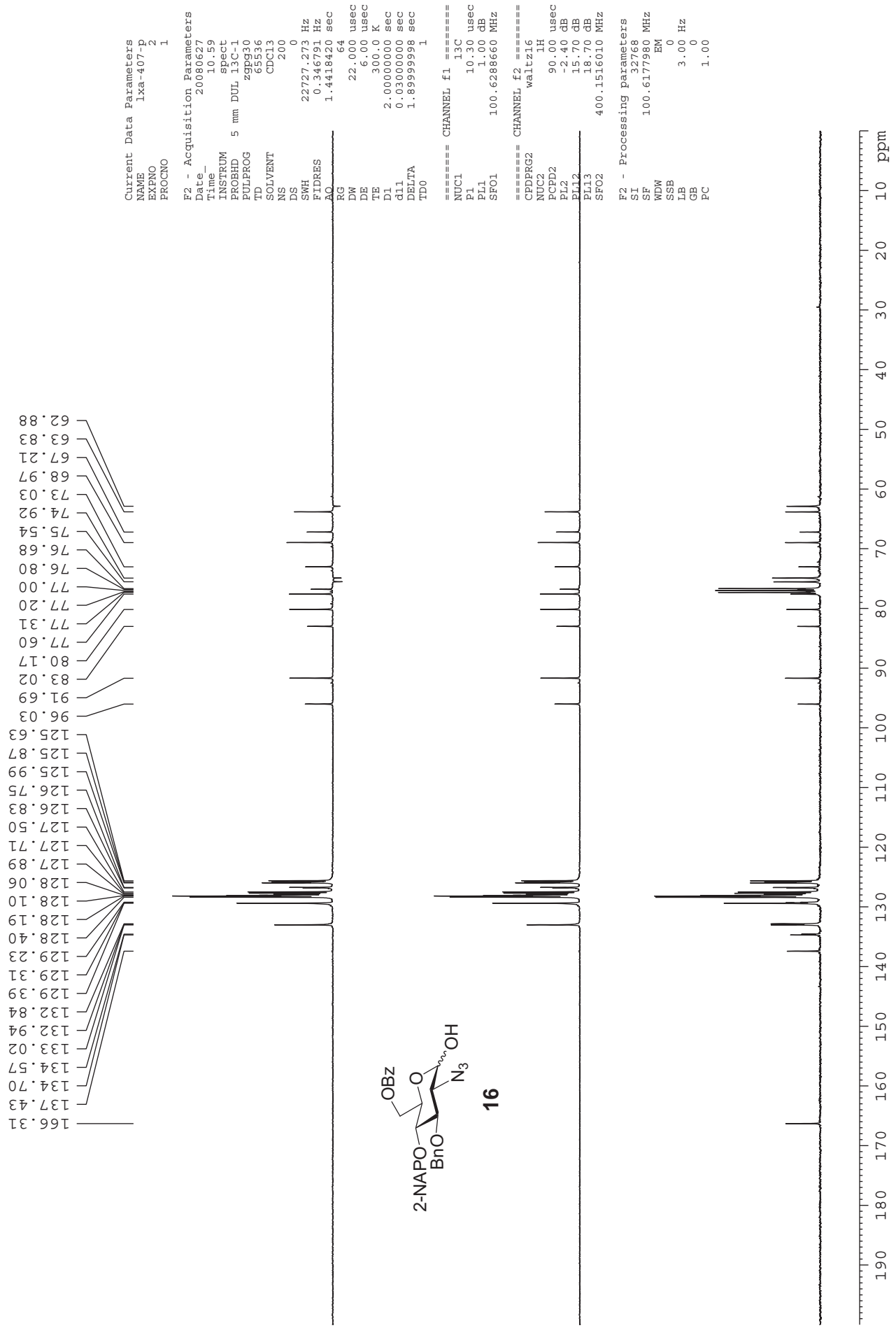

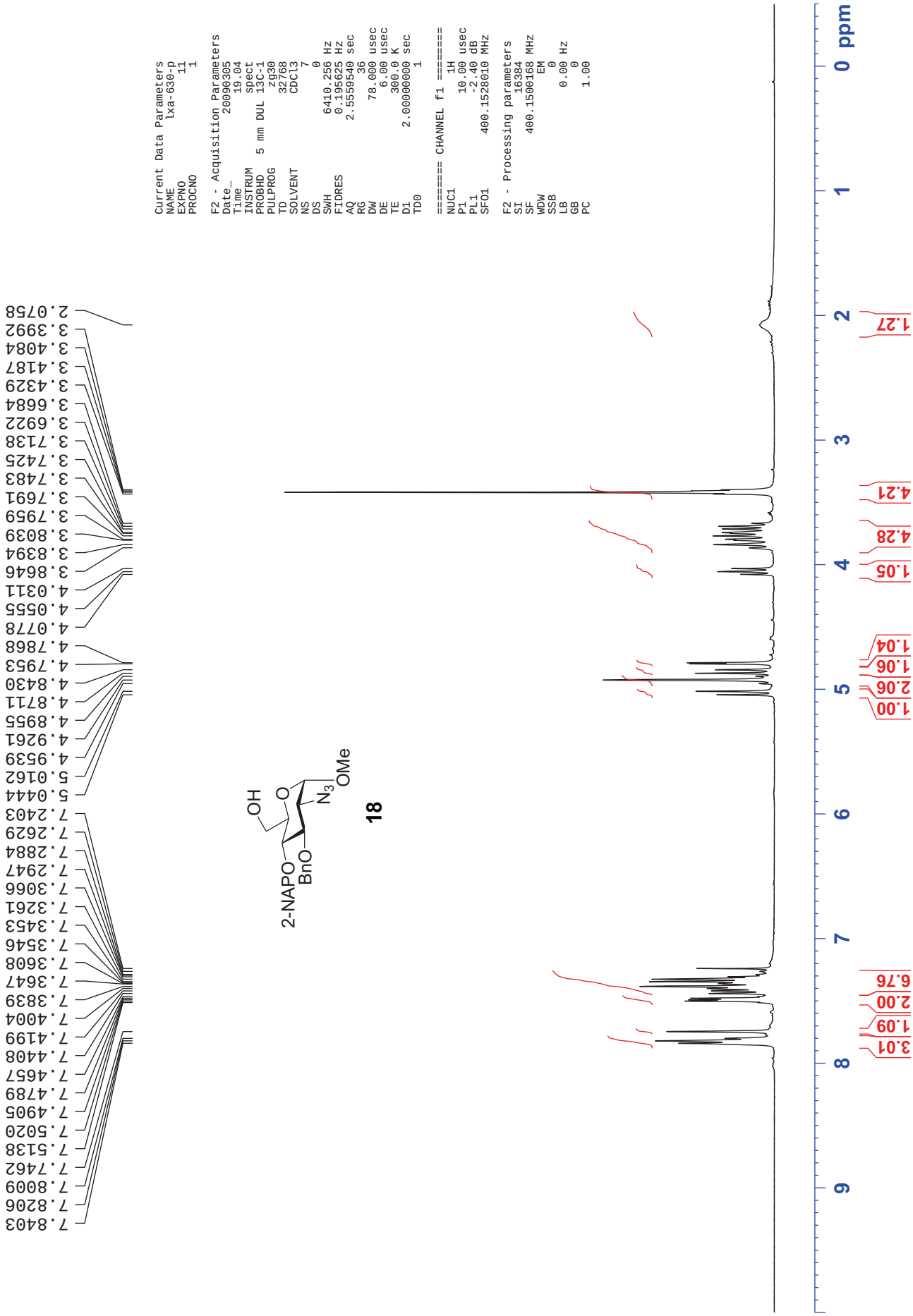
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F2 - Processing parameters
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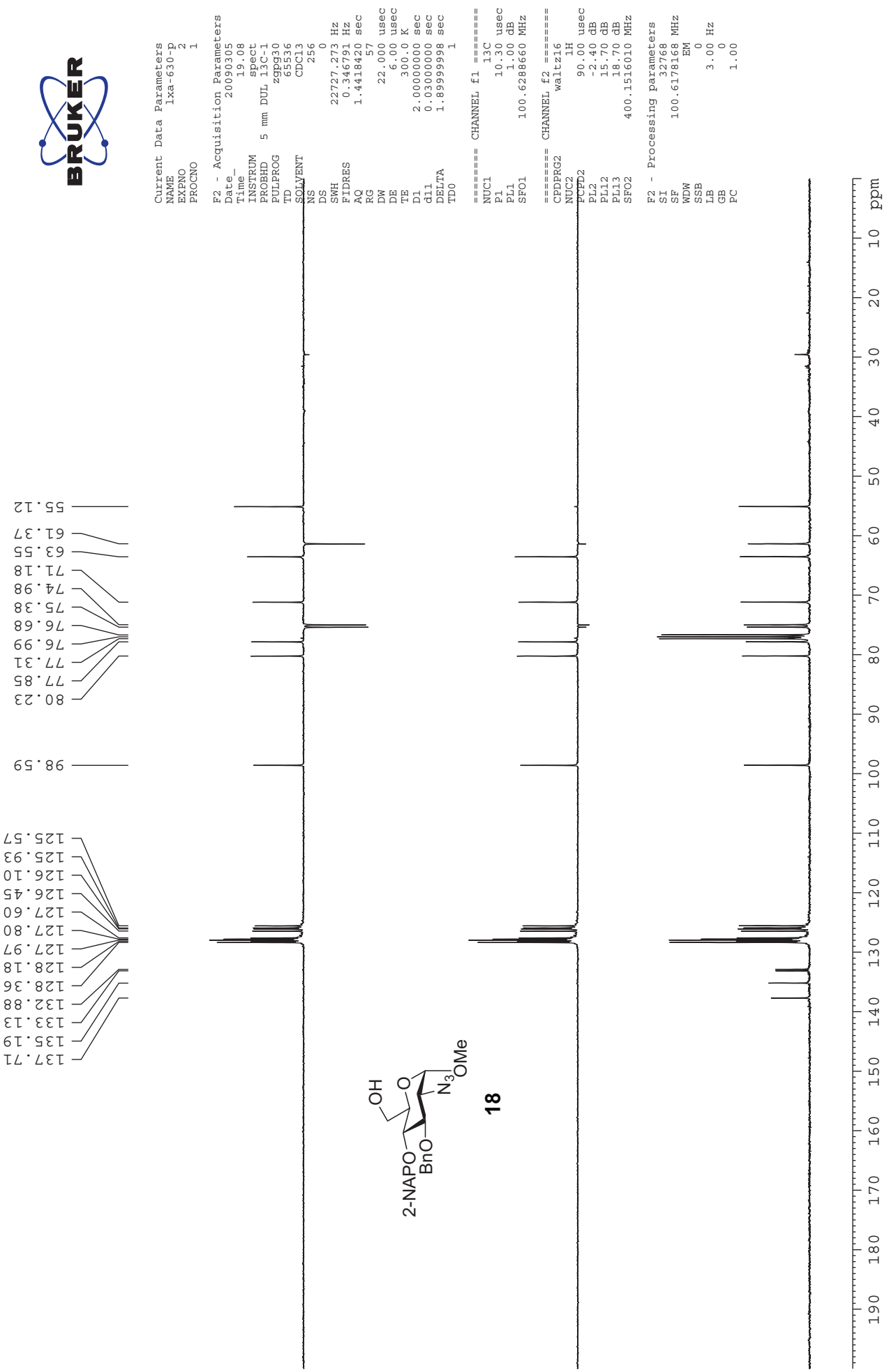
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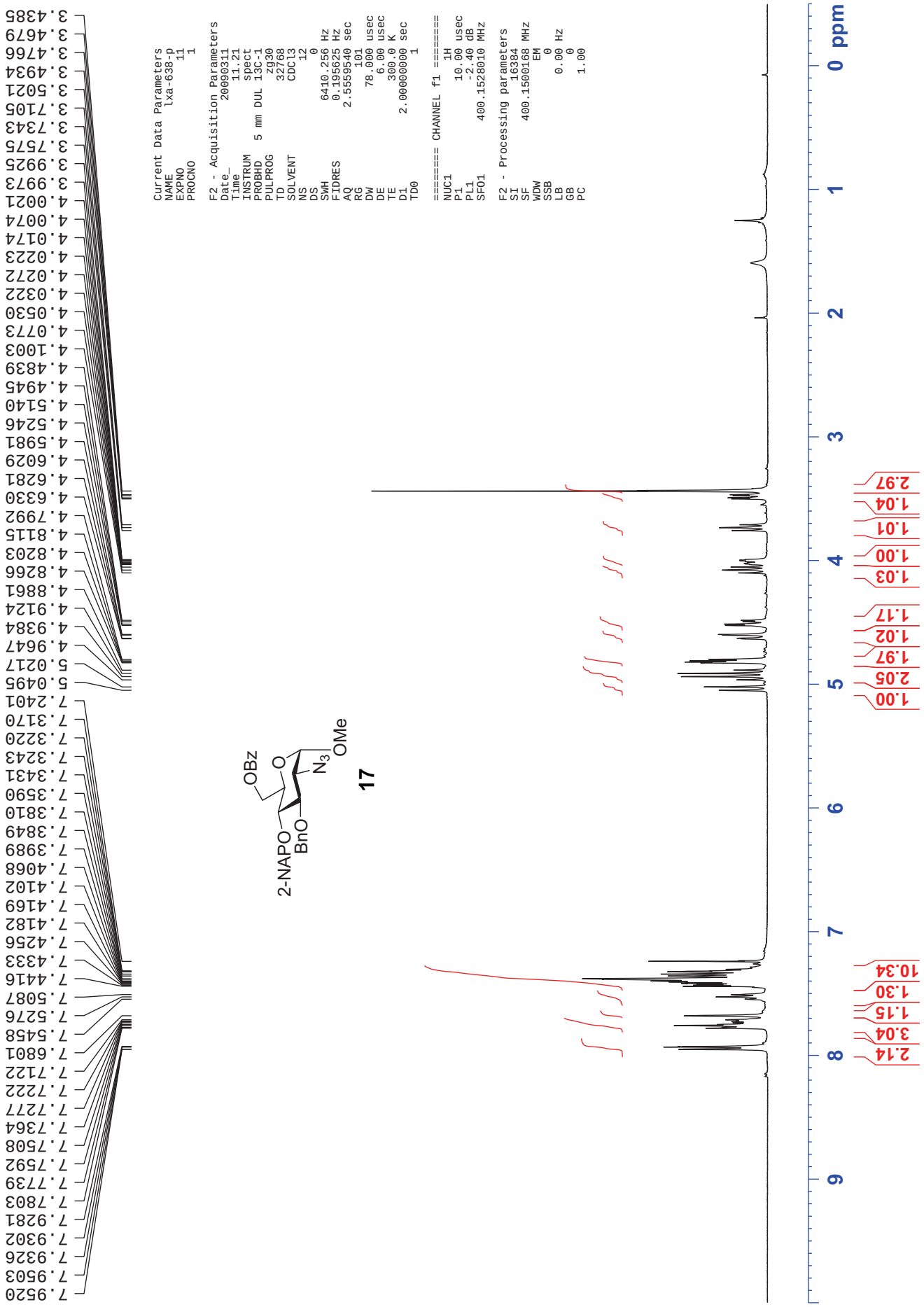
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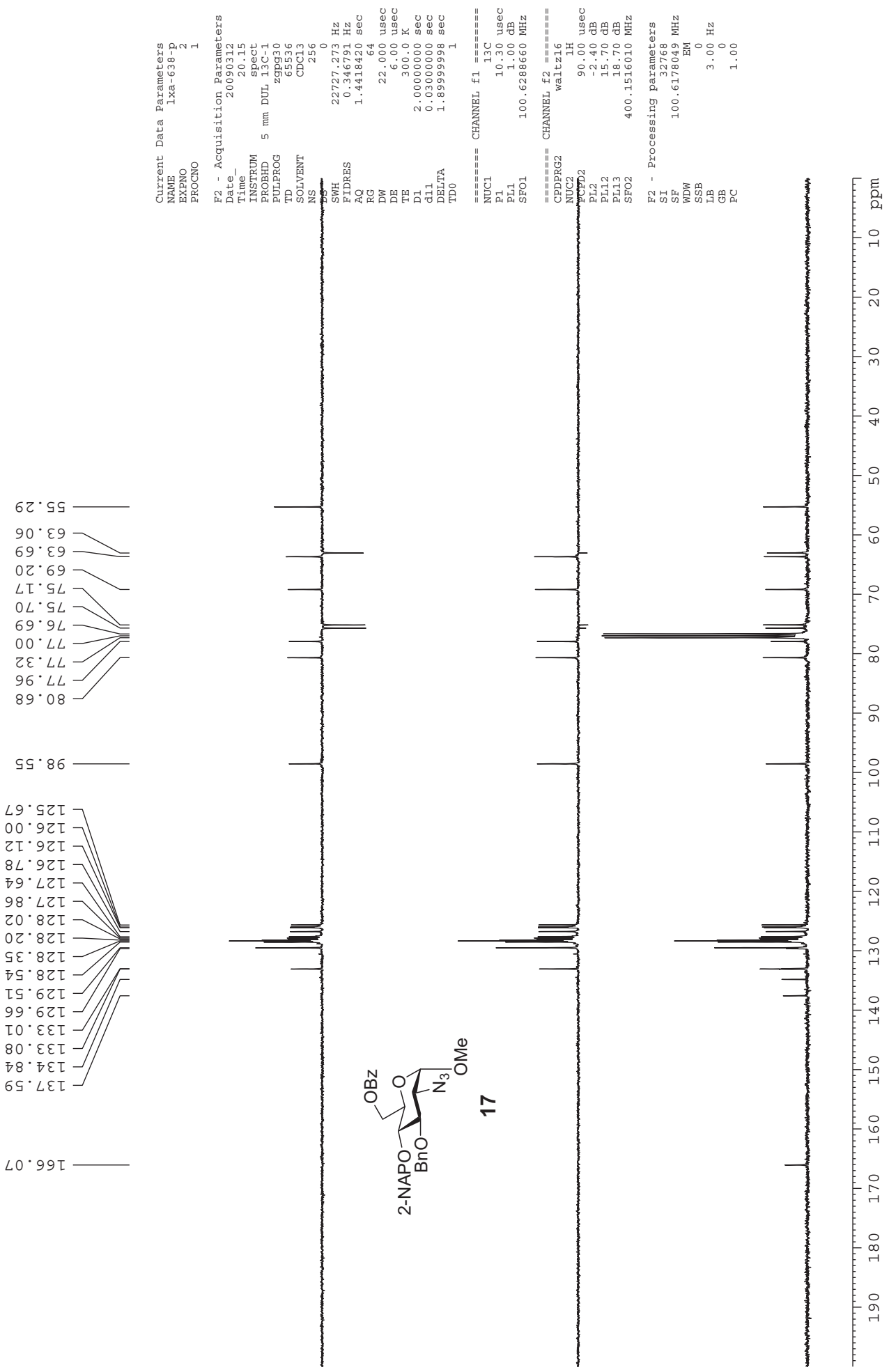
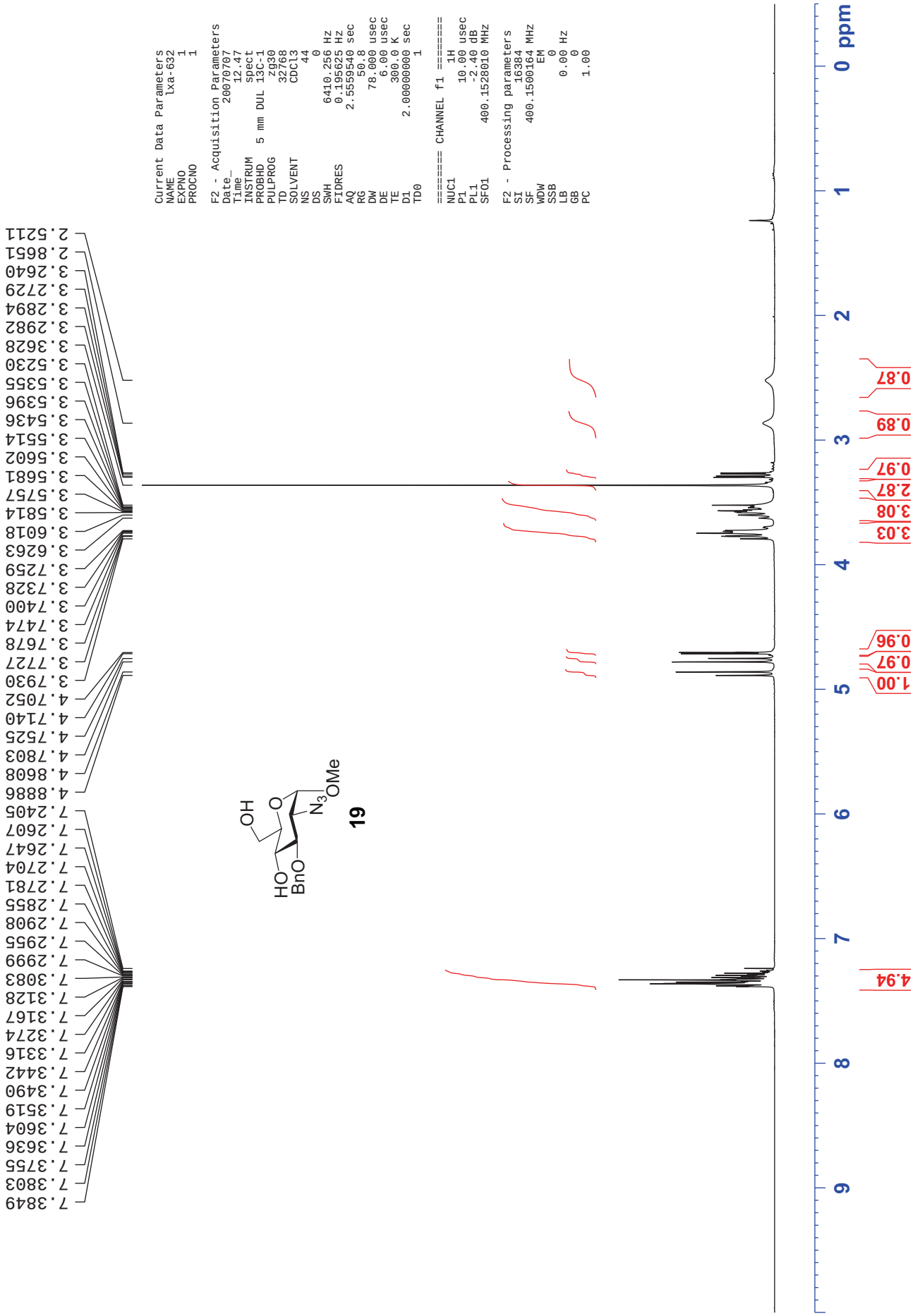
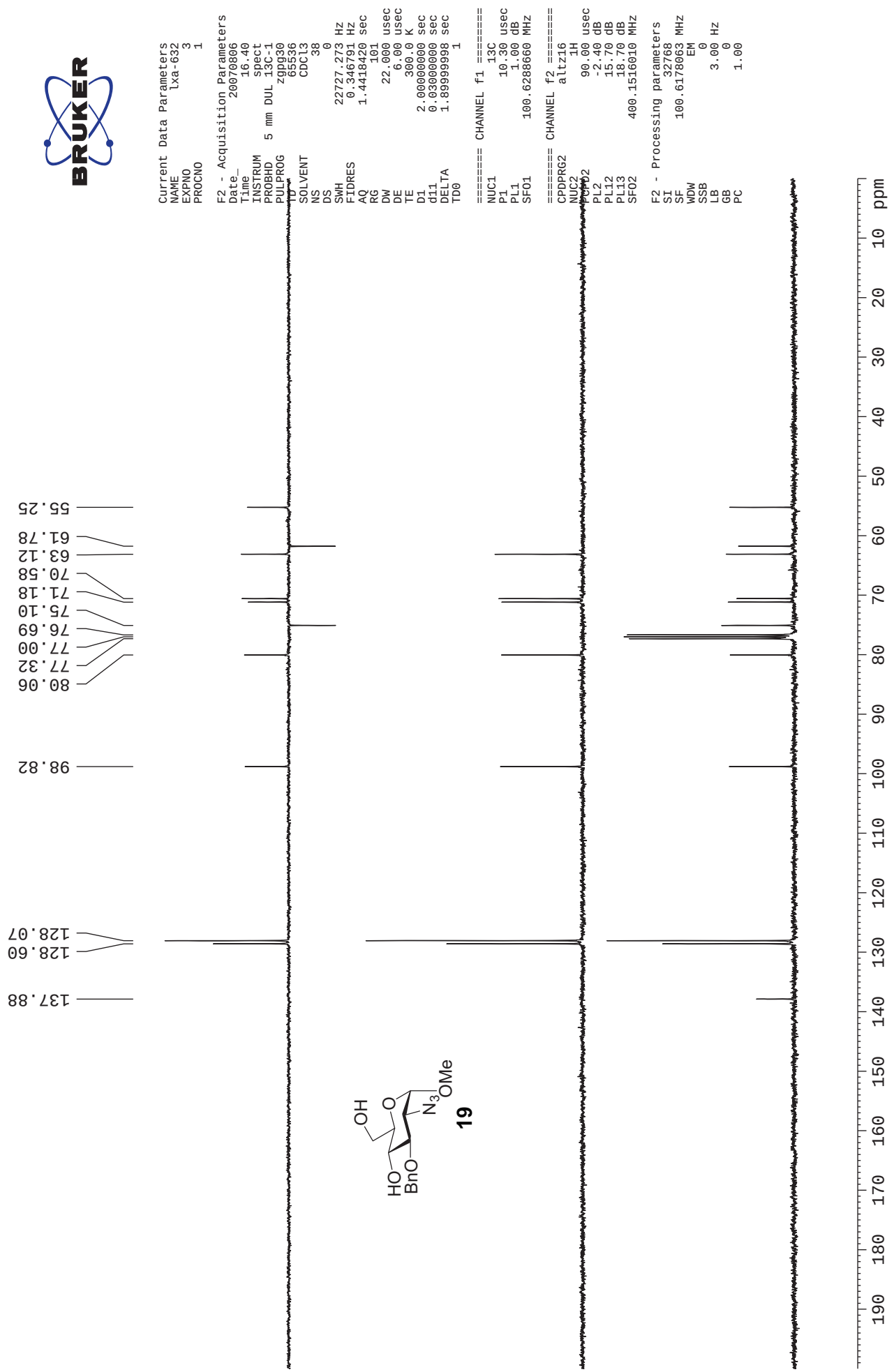
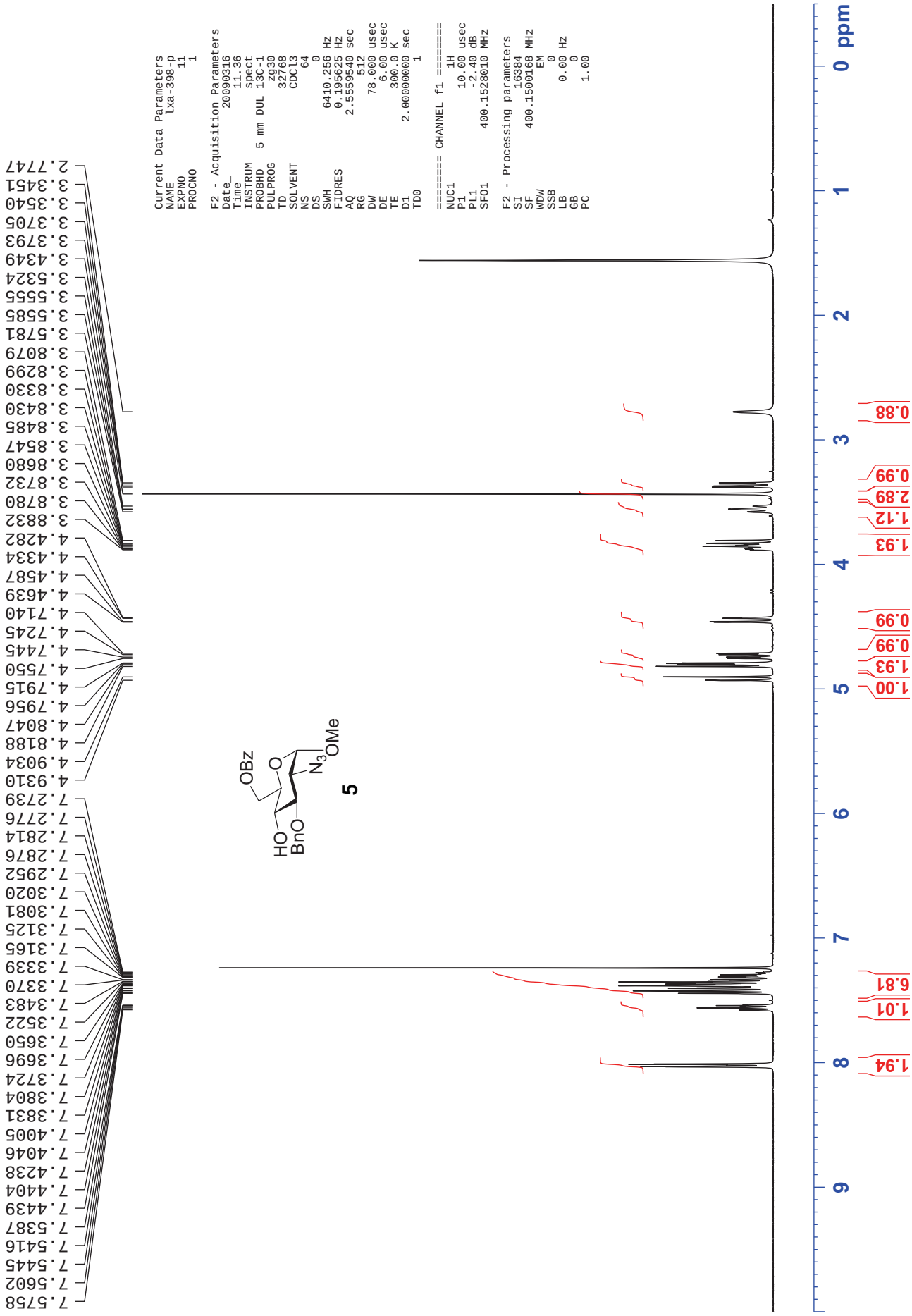
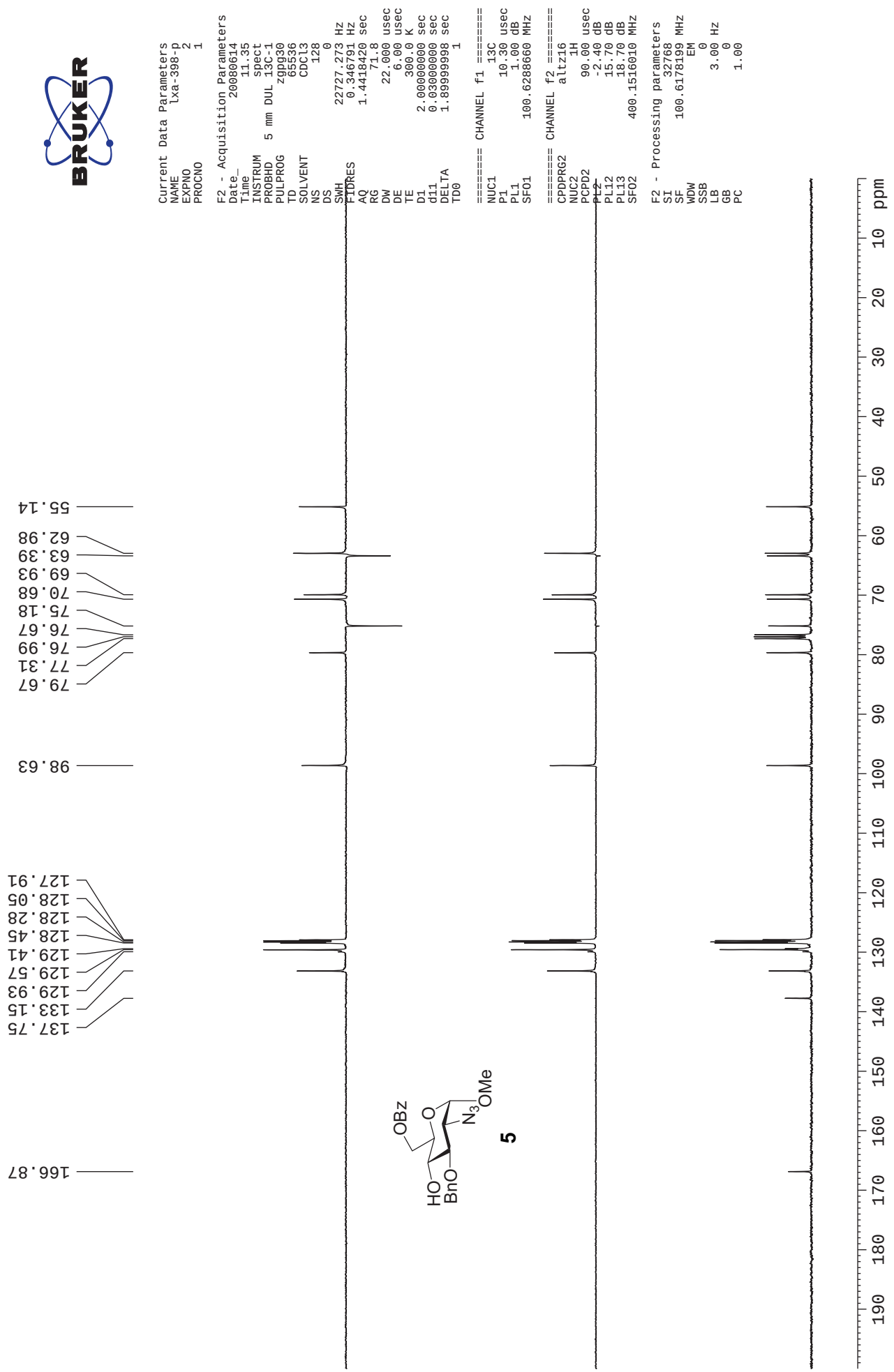
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090311
Time_ 11.21
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 12
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 101
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500168 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
OBz
O
N3
OMe
2-NAPO
BnO
17
0 ppm
1 2 3 4 5 6 7 8 9
2.97
1.04
1.01
1.00
1.03
1.00
1.17
1.02
1.97
2.05
1.00
10.34
1.30
1.15
3.04
2.14
3.4385 3.4679 3.4766 3.4934 3.5021 3.7105 3.7343 3.7575 3.9925 3.9973 4.0021 4.0074 4.0174 4.0223 4.0272 4.0322 4.0530 4.0773 4.1003 4.4839 4.4945 4.5140 4.5246 4.5981 4.6029 4.6281 4.6330 4.7992 4.8115 4.8203 4.8266 4.8861 4.9124 4.9384 4.9647 5.0217 5.0495 7.2401 7.3170 7.3220 7.3243 7.3431 7.3590 7.3810 7.3849 7.3989 7.4068 7.4102 7.4169 7.4182 7.4256 7.4333 7.4416 7.5087 7.5276 7.5458 7.6801 7.7122 7.7222 7.7277 7.7364 7.7508 7.7592 7.7739 7.7803 7.9281 7.9302 7.9326 7.9503 7.9520

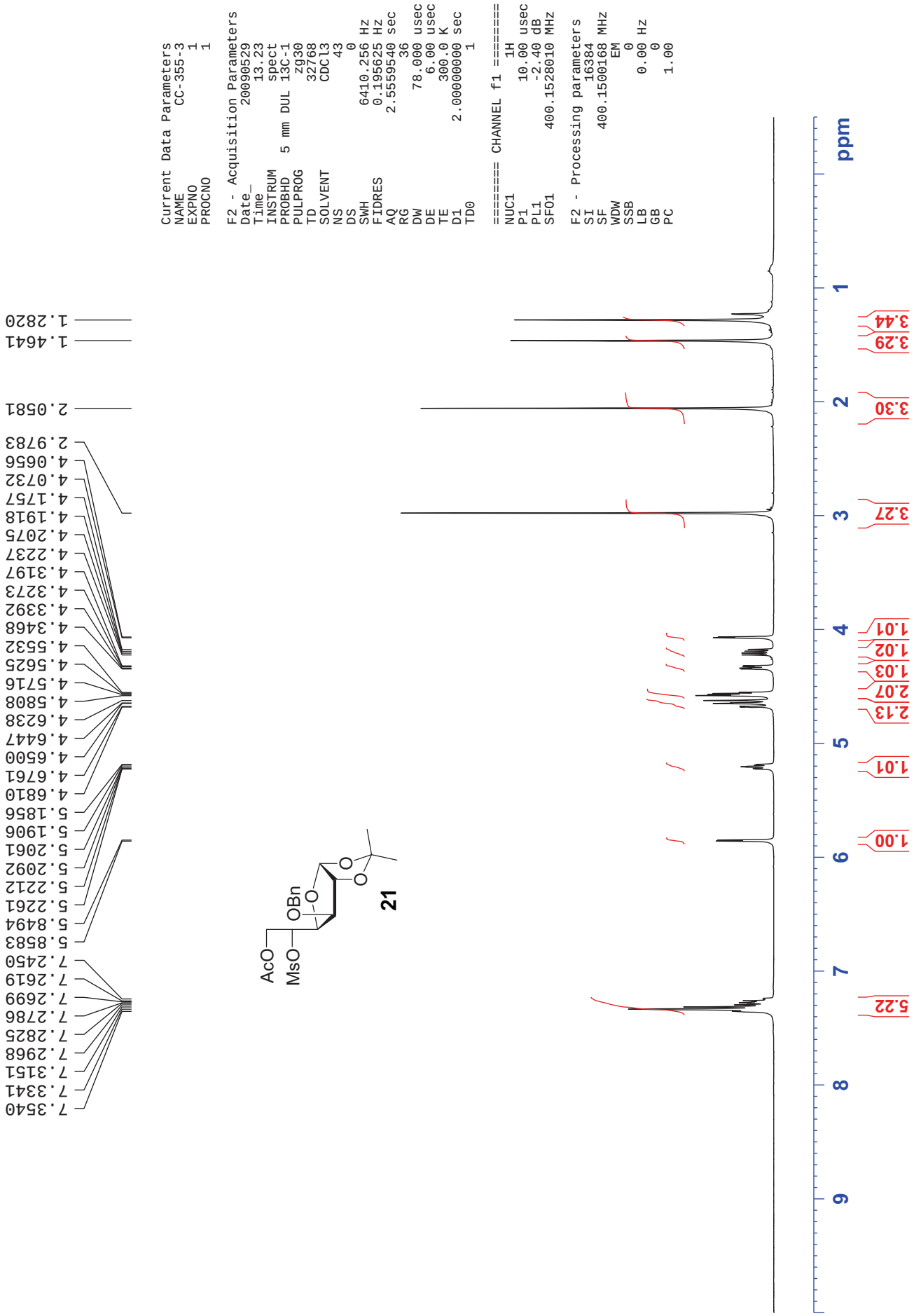

Current Data Parameters
NAME lxa-638-p
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090312
Time_ 20.15
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 64
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.30 usec
PL1 1.00 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.70 dB
PL13 18.70 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178049 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
55.29
63.06
63.69
69.20
75.17
75.70
76.69
77.00
77.32
77.96
80.68
98.55
125.67
126.00
126.12
126.78
127.64
127.86
128.02
128.20
128.35
128.54
129.51
129.66
133.01
133.08
134.84
137.59
166.07
OBz
O
N3
OMe
2-NAPO
BnO
17
ppm

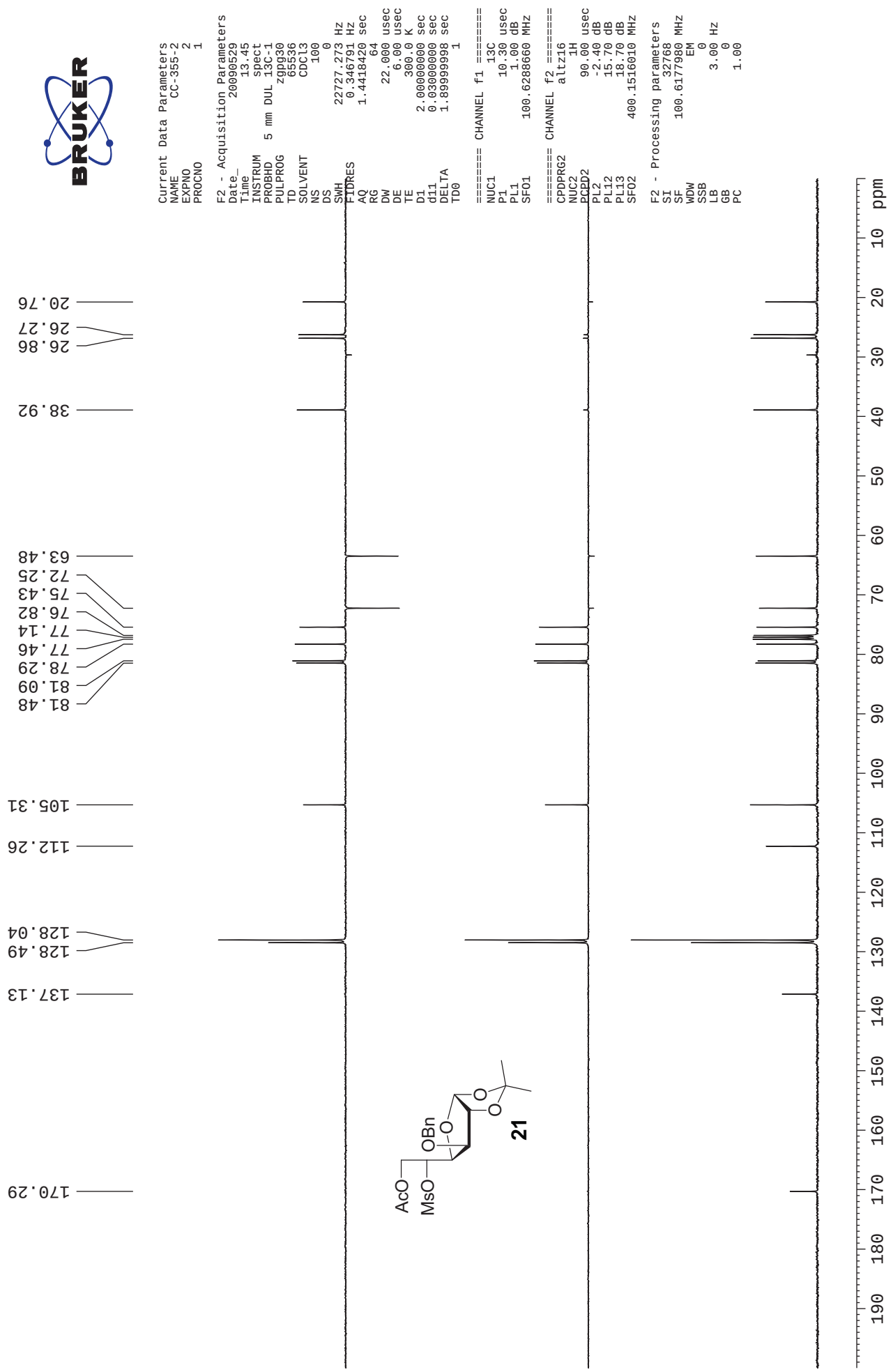

Current Data Parameters
NAME lxa-632
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070707
Time_ 12.47
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 44
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 50.8
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500164 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
OH
O
N3
OMe
HO
BnO
19
ppm
0.87
0.89
0.97
2.87
3.08
3.03
0.96
0.97
1.00
4.94

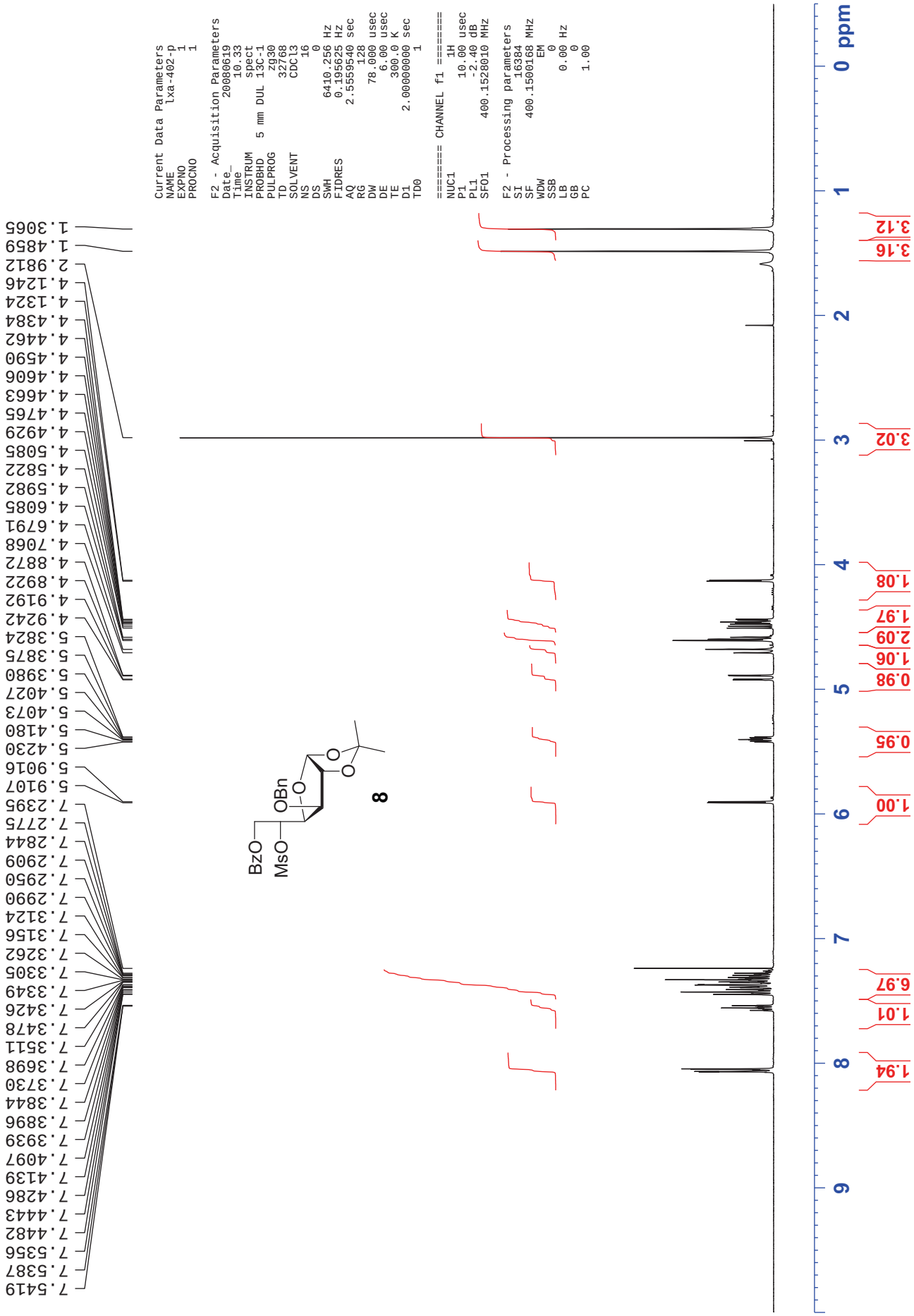

Current Data Parameters
NAME lxa-632
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070806
Time 16.40
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 38
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 101
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.30 usec
PL1 1.00 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 altz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.70 dB
PL13 18.70 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178063 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
55.25
61.78
63.12
70.58
71.18
75.10
76.69
77.00
77.32
80.06
98.82
128.07
128.60
137.88
OH
O
N3
OMe
HO
BnO
19
ppm

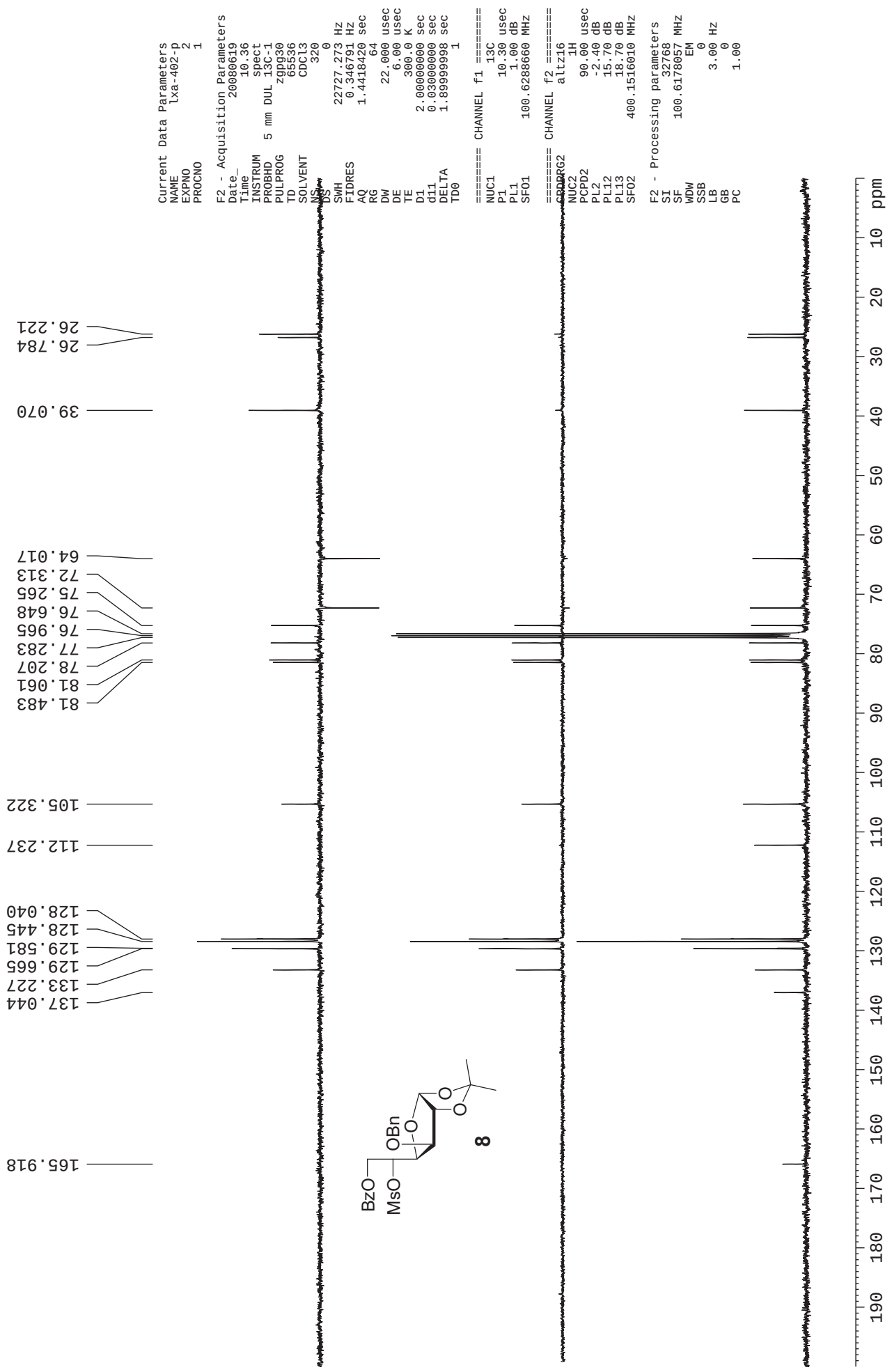

Current Data Parameters
NAME lxa-398-p
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090316
Time 11.36
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 64
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 512
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500168 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
OBz
O
N3
OMe
HO
BnO
5
0.88
0.99
2.89
1.12
1.93
0.99
0.99
1.93
1.00
6.81
1.01
1.94
0 ppm
1
2
3
4
5
6
7
8
9
2.7747
3.3451
3.3540
3.3705
3.3793
3.4349
3.5324
3.5555
3.5585
3.5781
3.8079
3.8299
3.8330
3.8430
3.8485
3.8547
3.8680
3.8732
3.8780
3.8832
4.4282
4.4334
4.4587
4.4639
4.7140
4.7245
4.7445
4.7550
4.7915
4.7956
4.8047
4.8188
4.9034
4.9310
7.2739
7.2776
7.2814
7.2876
7.2952
7.3020
7.3081
7.3125
7.3165
7.3339
7.3370
7.3483
7.3522
7.3650
7.3696
7.3724
7.3804
7.3831
7.4005
7.4046
7.4238
7.4404
7.4439
7.5387
7.5416
7.5445
7.5602
7.5758

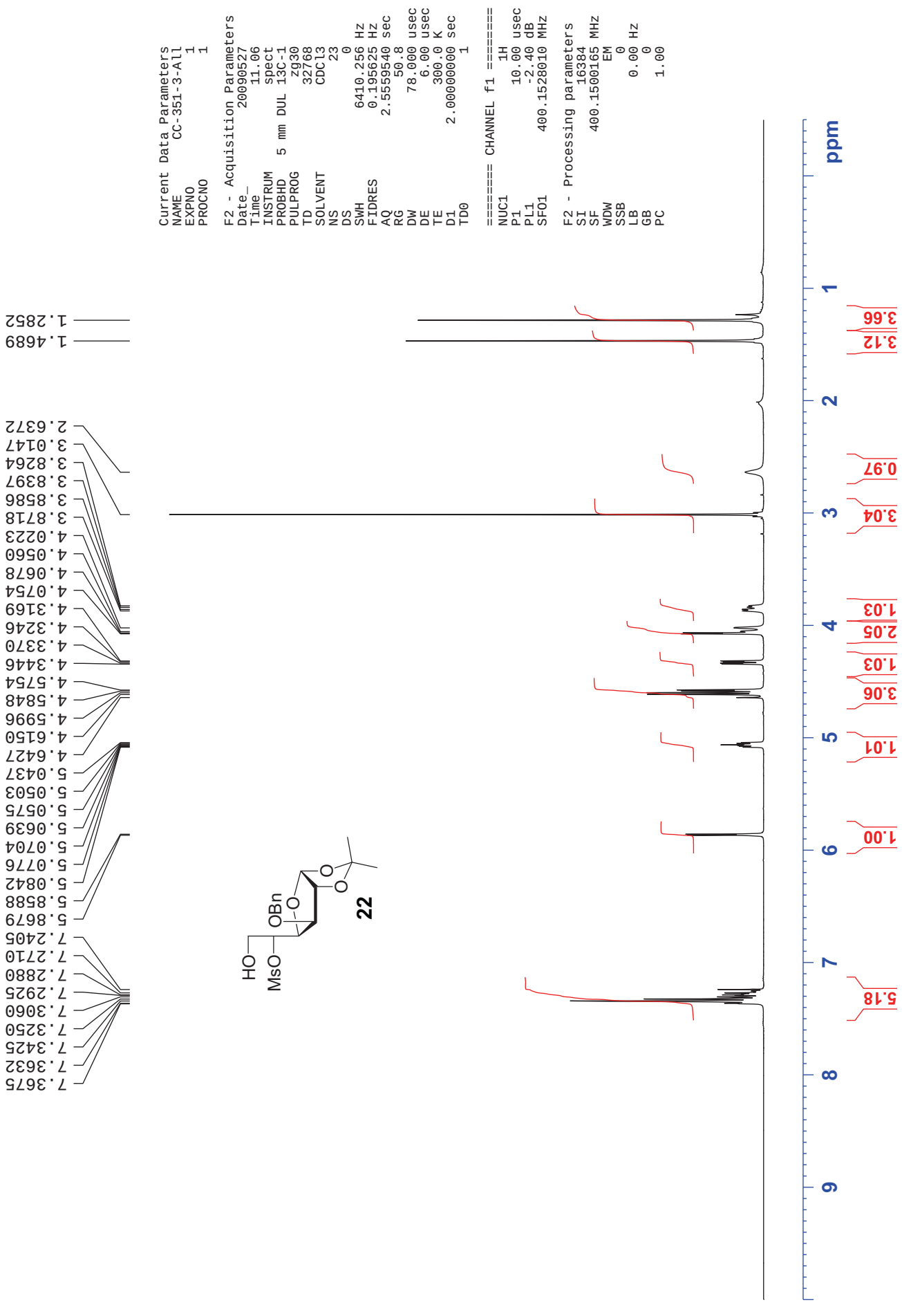

Current Data Parameters
NAME lxa-398-p
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080614
Time 11.35
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 71.8
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.30 usec
PL1 1.00 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 altz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.70 dB
PL13 18.70 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178199 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
55.14
62.98
63.39
69.93
70.68
75.18
76.67
76.99
77.31
79.67
98.63
127.91
128.05
128.28
128.45
129.41
129.57
129.93
133.15
137.75
166.87
OBz
O
N3
OMe
HO
BnO
5
ppm

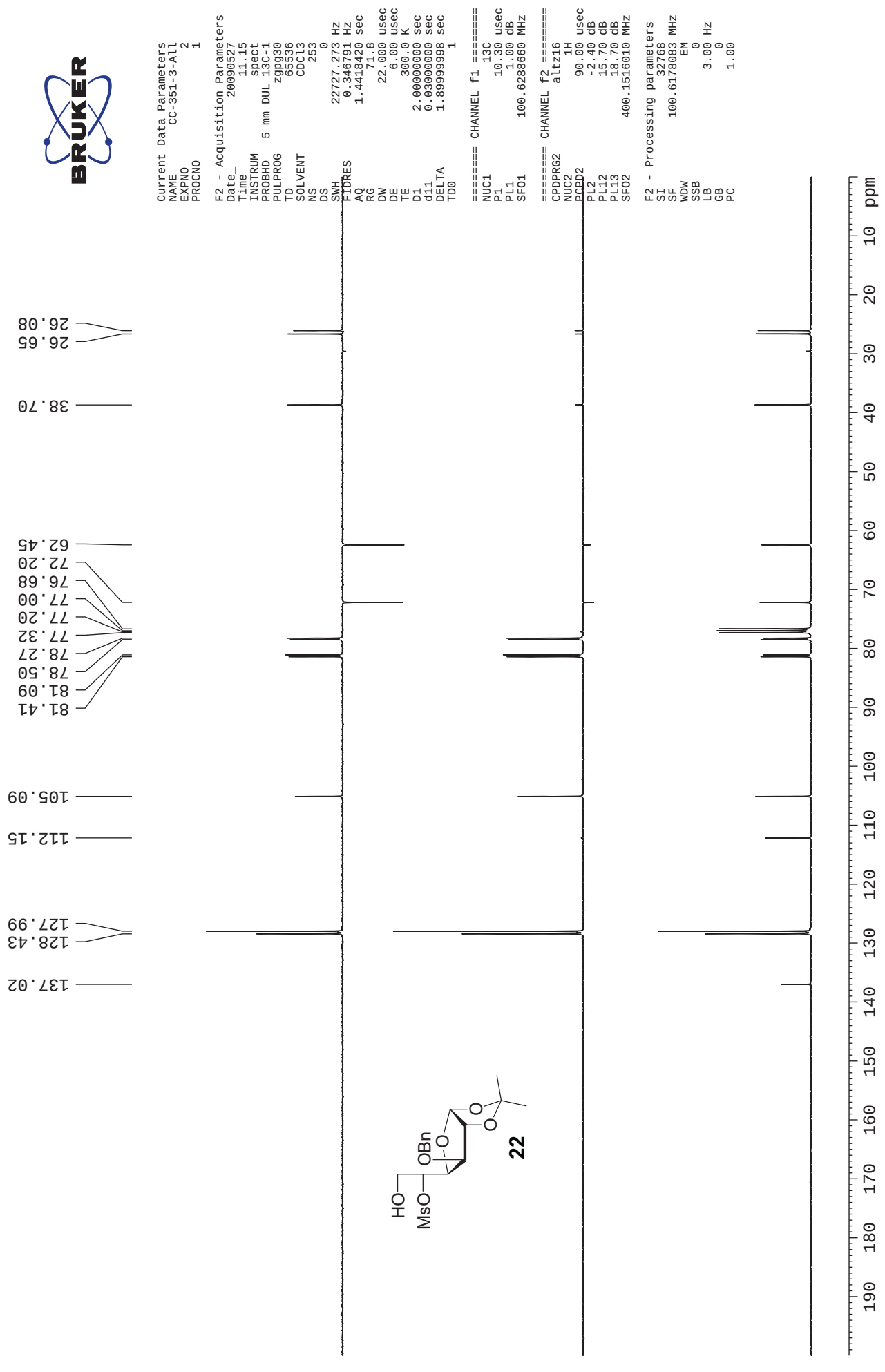

Current Data Parameters
NAME CC-355-3
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090529
Time 13.23
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 43
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 36
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500168 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1.2820
1.4641
2.0581
2.9783
4.0656
4.0732
4.1757
4.1918
4.2075
4.2237
4.3197
4.3273
4.3392
4.3468
4.5532
4.5625
4.5716
4.5808
4.6238
4.6447
4.6500
4.6761
4.6810
5.1856
5.1906
5.2061
5.2092
5.2212
5.2261
5.8494
5.8583
7.2450
7.2619
7.2699
7.2786
7.2825
7.2968
7.3151
7.3341
7.3540
AcO
MsO
OBn
21
ppm
1
2
3
4
5
6
7
8
9
3.44
3.29
3.30
3.27
1.01
1.02
1.03
2.07
2.13
1.01
1.00
5.22

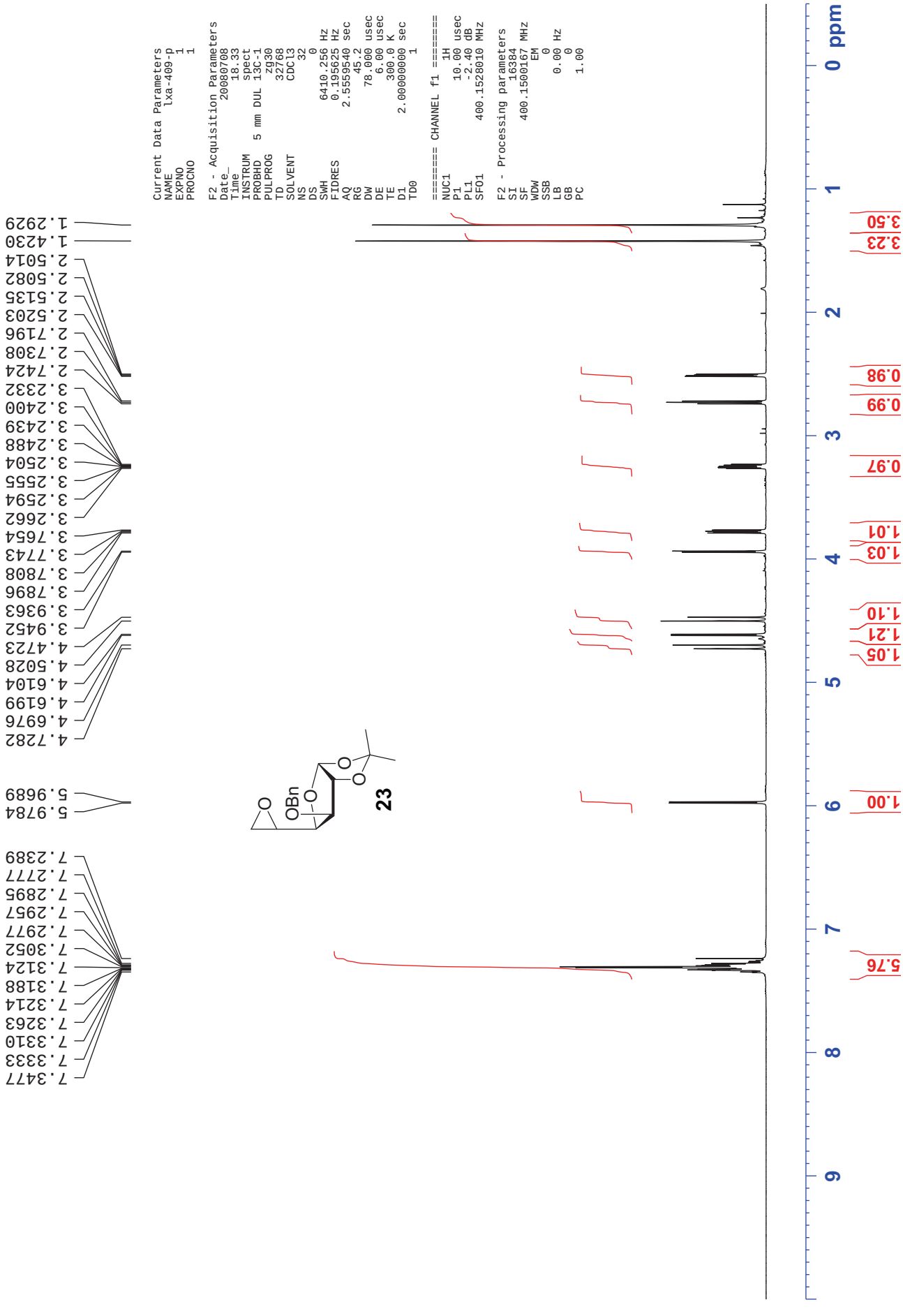


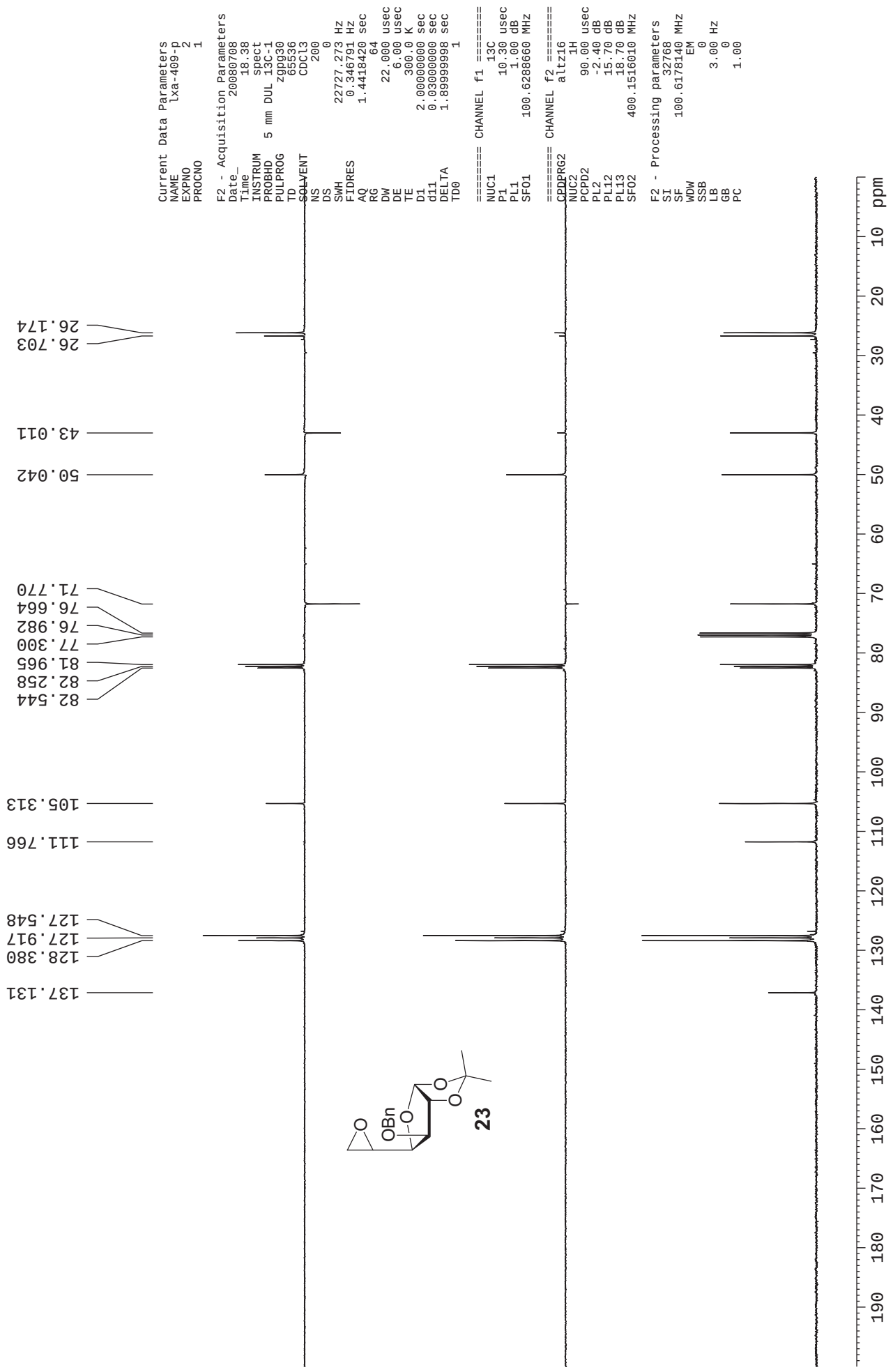

Current Data Parameters
NAME lxa-402-p
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080619
Time 10.33
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 128
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500168 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
BzO
MsO
OBn
8
ppm
3.12
3.16
3.02
1.08
1.97
2.09
1.06
0.98
0.95
1.00
6.97
1.01
1.94

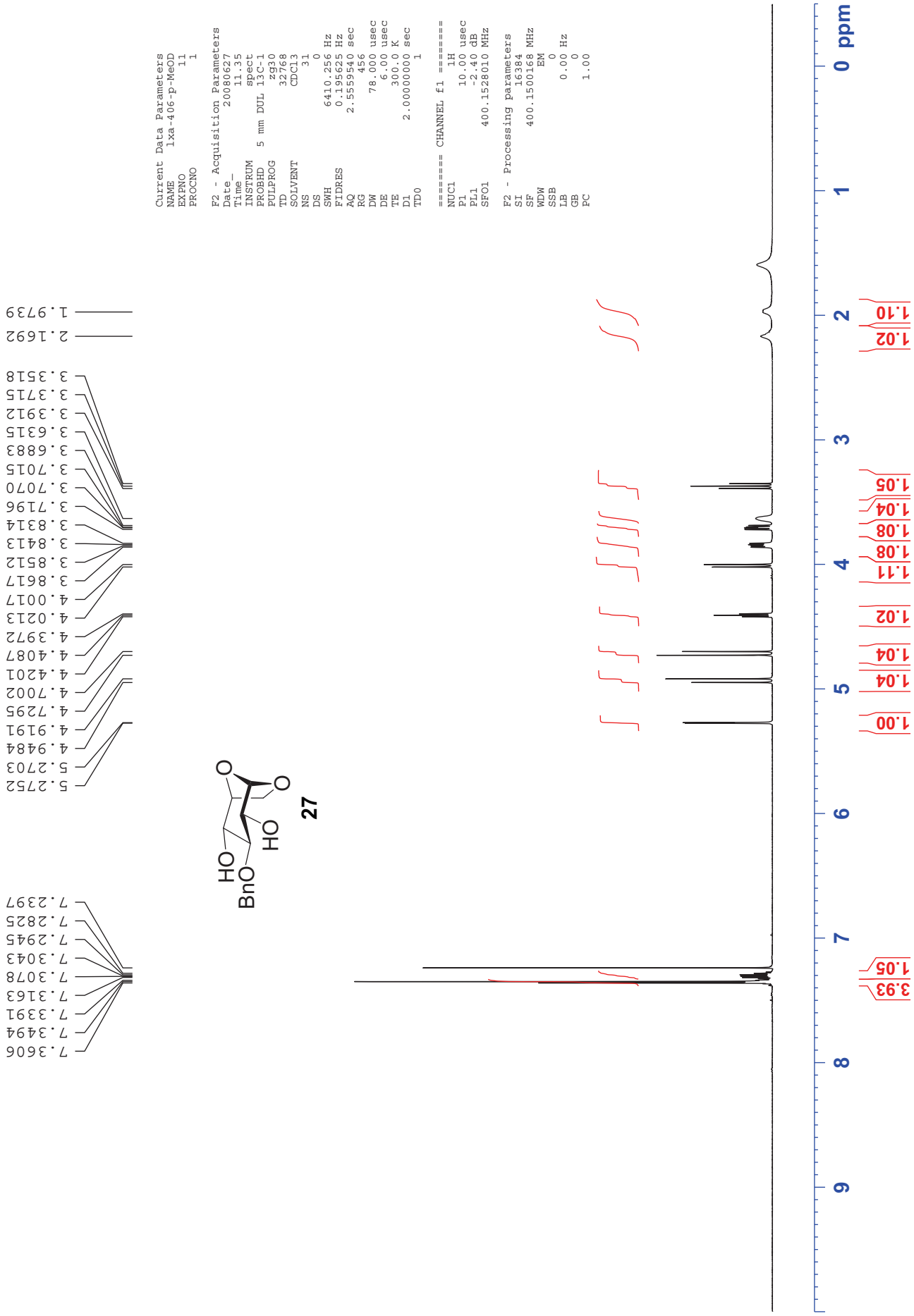

Current Data Parameters
NAME lxa-402-p
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080619
Time 10.36
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 320
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 64
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.30 usec
PL1 1.00 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 altz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.70 dB
PL13 18.70 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178057 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
165.918
137.044
133.227
129.665
129.581
128.445
128.040
112.237
105.322
81.483
81.061
78.207
77.283
76.965
76.648
75.265
72.313
64.017
39.070
26.784
26.221
BzO
MsO
OBn
O
O
O
8
ppm

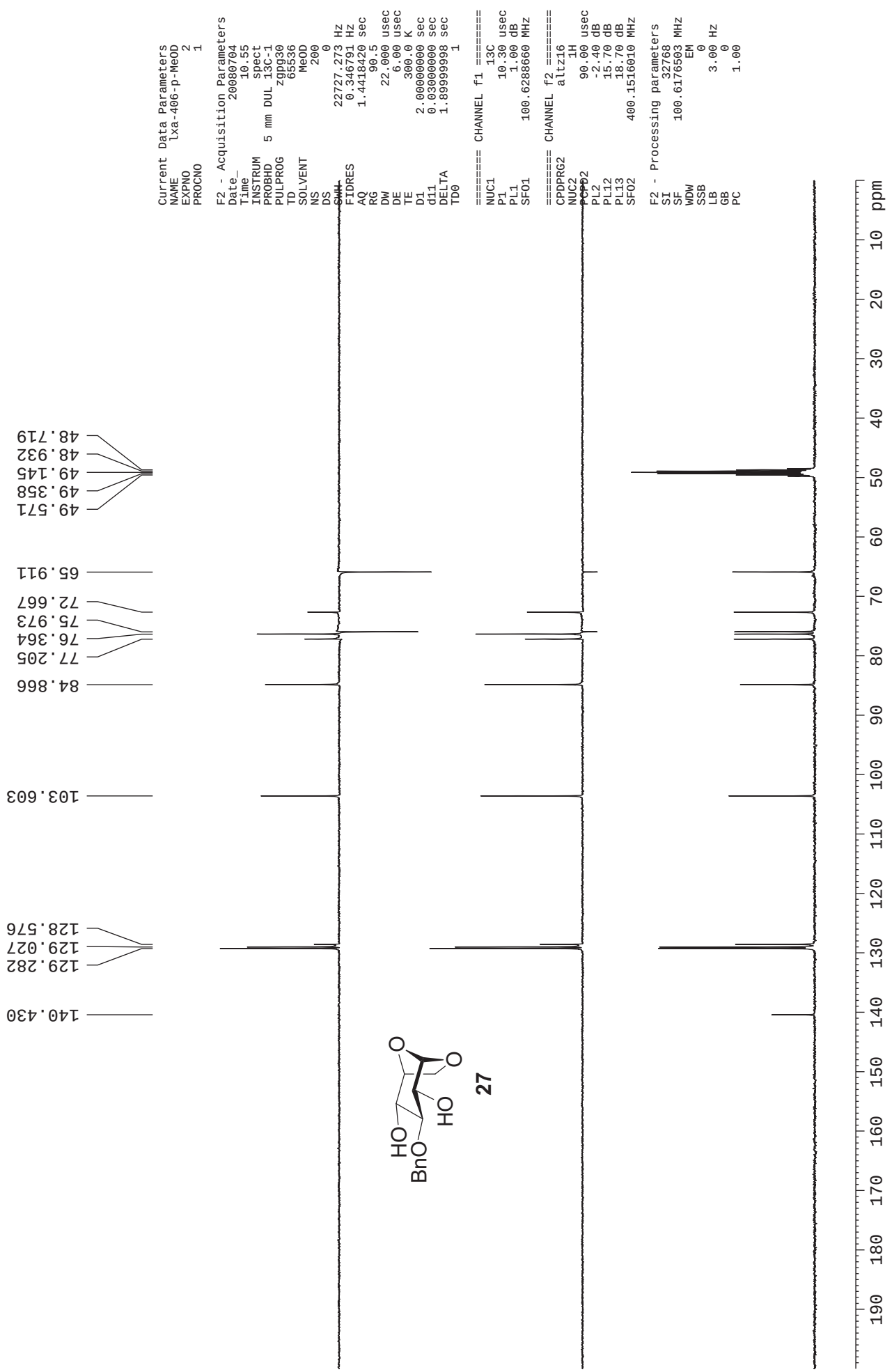


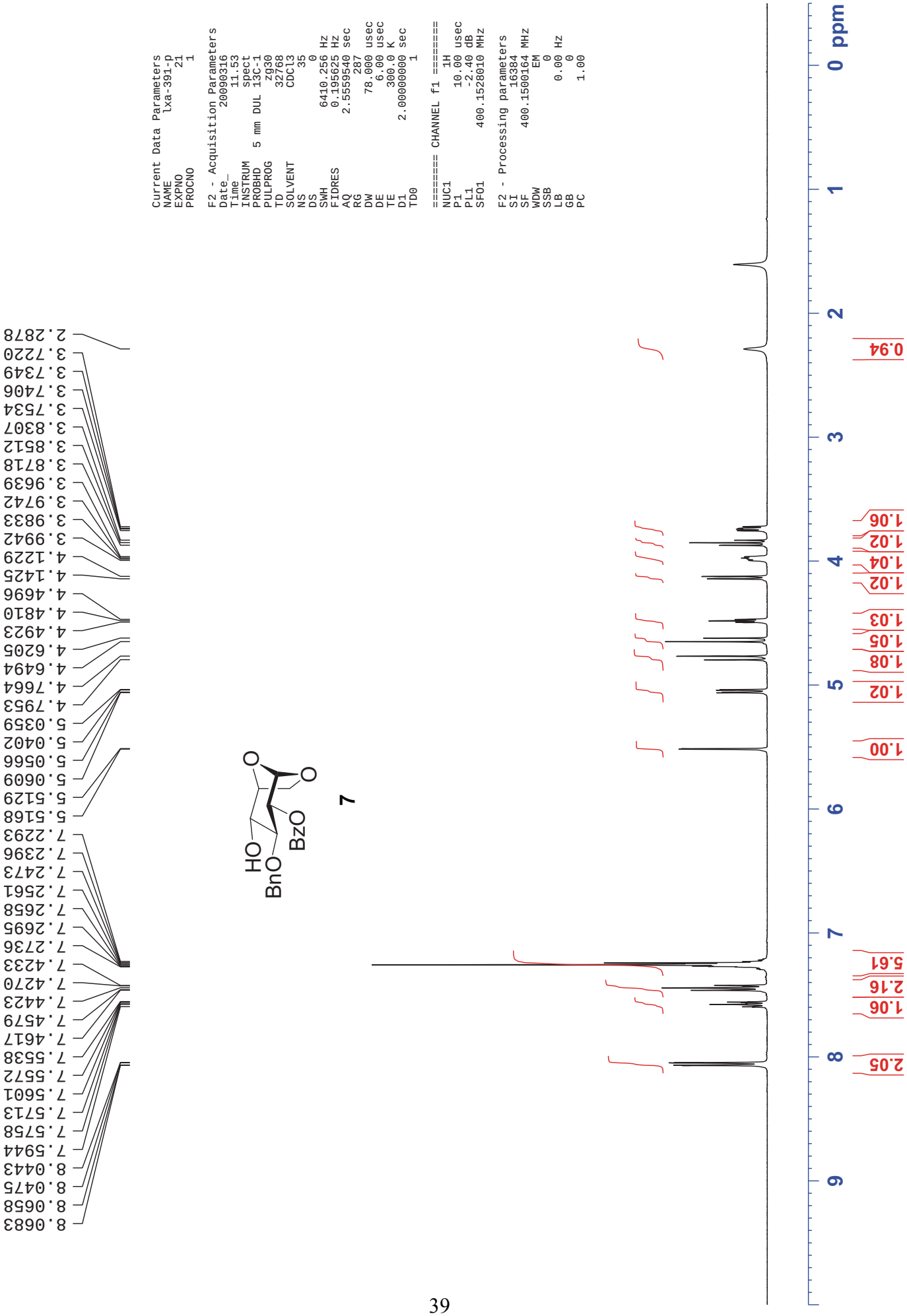


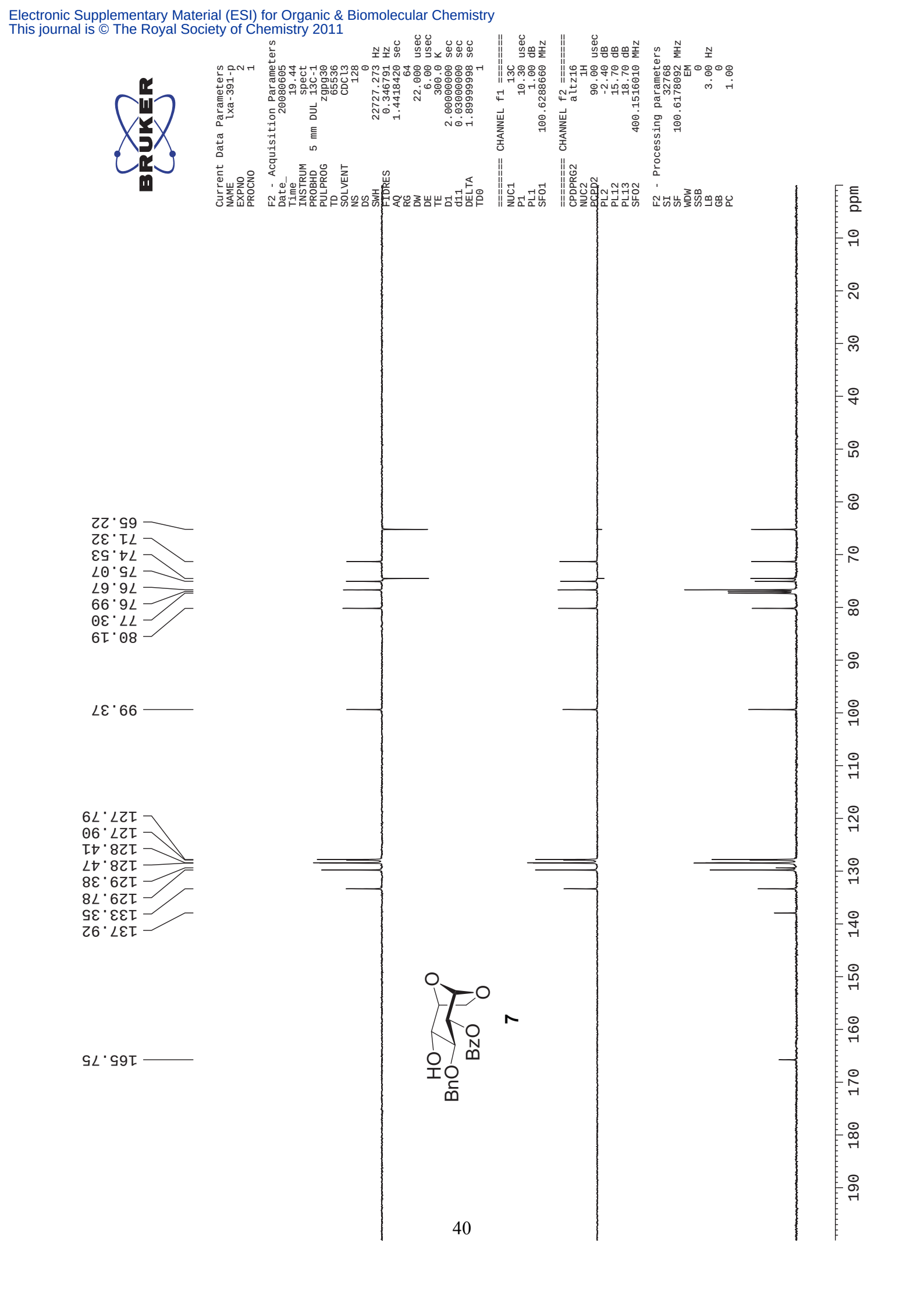

Current Data Parameters
NAME lxa-409-p
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080708
Time_ 18.33
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 45.2
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500167 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
OBn
23
0 ppm
1 2 3 4 5 6 7 8 9
3.50
3.23
0.98
0.99
0.97
1.01
1.03
1.10
1.21
1.05
1.00
5.76
1.2929
1.4230
2.5014
2.5082
2.5135
2.5203
2.7196
2.7308
2.7424
3.2332
3.2400
3.2439
3.2488
3.2504
3.2555
3.2594
3.2662
3.7654
3.7743
3.7808
3.7896
3.9363
3.9452
4.4723
4.5028
4.6104
4.6199
4.6976
4.7282
5.9689
5.9784
7.2389
7.2777
7.2895
7.2957
7.2977
7.3052
7.3124
7.3188
7.3214
7.3263
7.3310
7.3333
7.3477

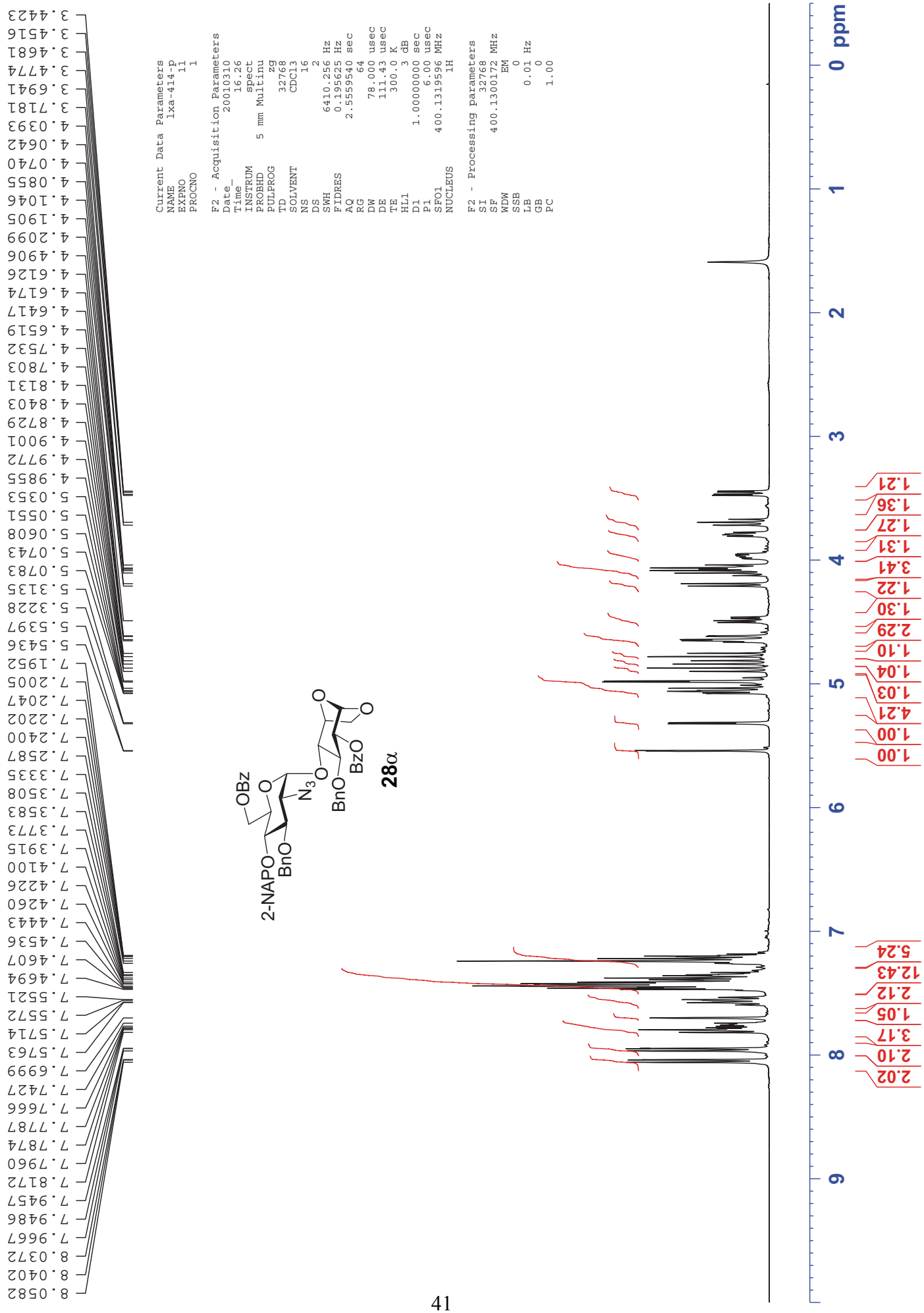

Current Data Parameters
NAME lxa-409-p
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080708
Time 18.38
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 200
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 64
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.30 usec
PL1 1.00 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 altz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.70 dB
PL13 18.70 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178140 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
26.174
26.703
43.011
50.042
71.770
76.664
76.982
77.300
81.965
82.258
82.544
105.313
111.766
127.548
127.917
128.380
137.131
OBn
23
ppm

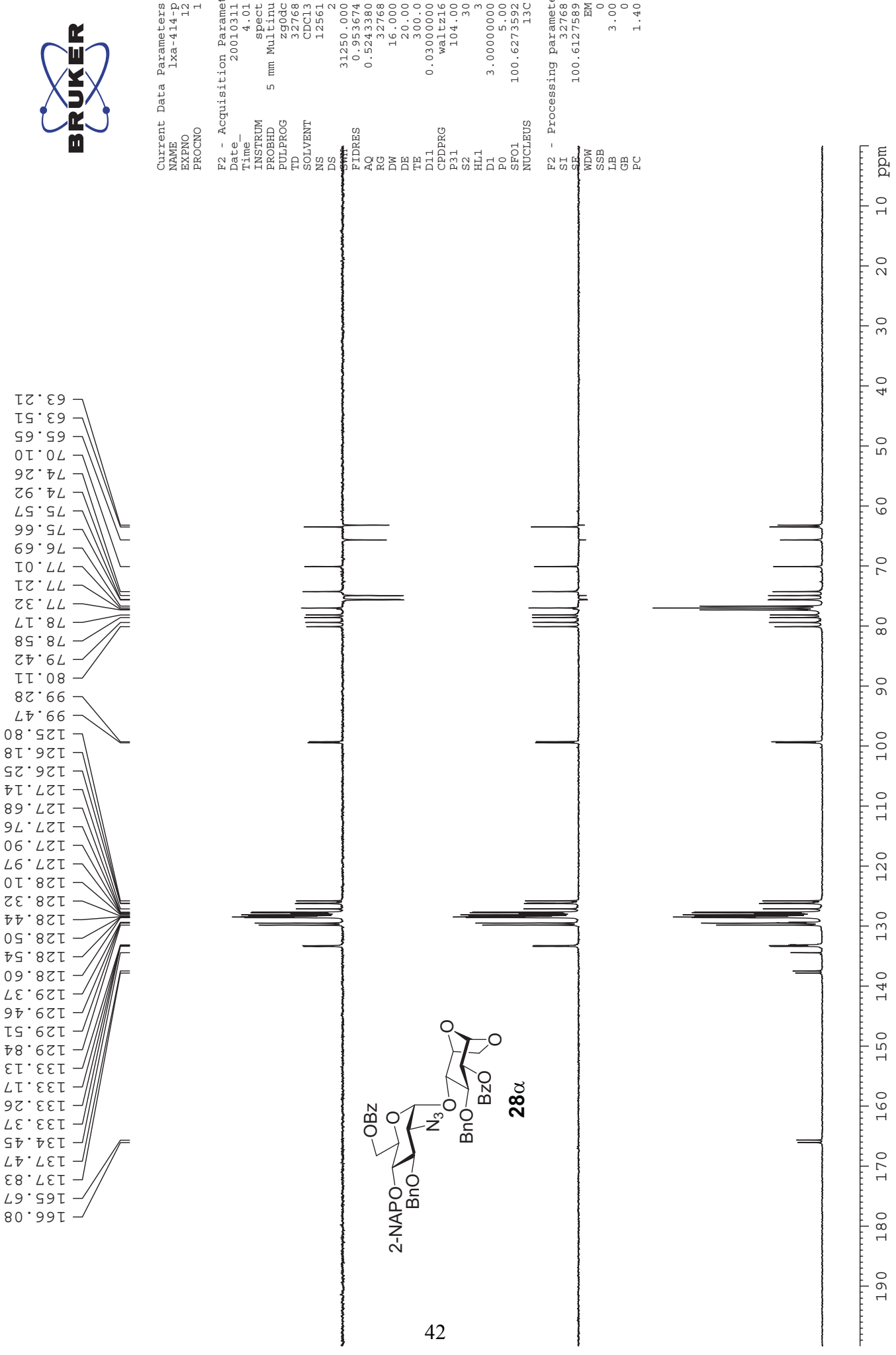

Current Data Parameters
NAME 1xa-406-p-MeOD
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080627
Time_ 11.35
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 31
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 456
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500168 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1.973
2.169
3.351
3.371
3.391
3.631
3.688
3.701
3.707
3.719
3.831
3.841
3.851
3.861
4.001
4.021
4.397
4.408
4.420
4.700
4.729
4.919
4.948
5.270
5.275
7.239
7.282
7.294
7.304
7.307
7.316
7.339
7.349
7.360
BnO
HO
HO
O
O
27
0 ppm
1.10
1.02
1.05
1.04
1.08
1.08
1.11
1.02
1.04
1.04
1.00
3.93
1.05

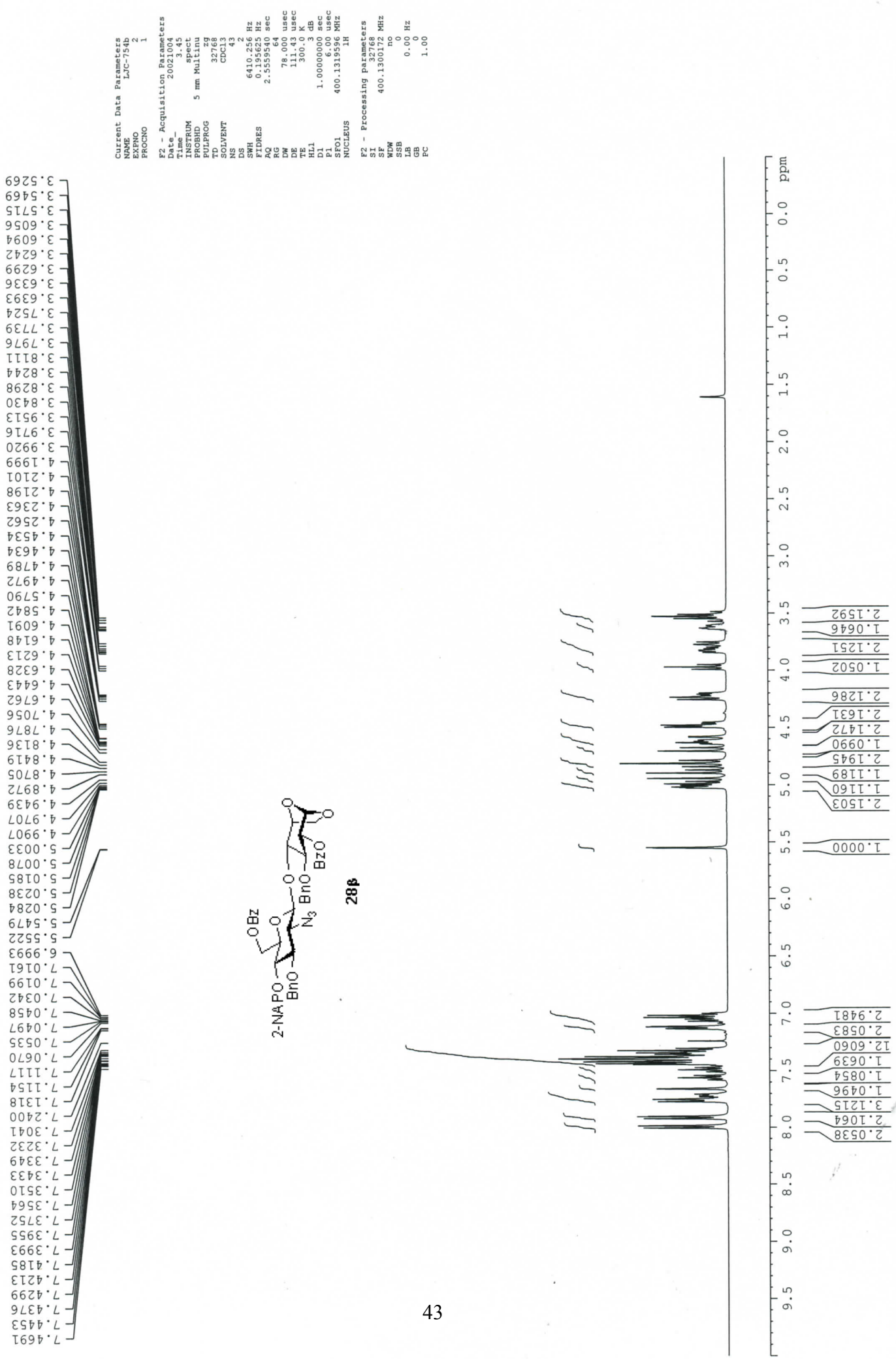

Current Data Parameters
NAME lxa-406-p-MeOD
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080704
Time 10.55
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 200
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 90.5
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.30 usec
PL1 1.00 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 altz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.70 dB
PL13 18.70 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6176503 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
48.719
48.932
49.145
49.358
49.571
65.911
72.667
75.973
76.364
77.205
84.866
103.603
128.576
129.027
129.282
140.430
HO
BnO
HO
O
O
27
ppm


Current Data Parameters
NAME lxa-391-p
EXPNO 21
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090316
Time 11.53
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 35
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 287
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500164 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
HO
BnO
BzO
O
O
7
8.0683
8.0658
8.0475
8.0443
7.5944
7.5758
7.5713
7.5601
7.5572
7.5538
7.4617
7.4579
7.4423
7.4270
7.4233
7.2736
7.2695
7.2658
7.2561
7.2473
7.2396
7.2293
5.5168
5.5129
5.0609
5.0566
5.0402
5.0359
4.7953
4.7664
4.6494
4.6205
4.4923
4.4810
4.4696
4.1425
4.1229
3.9942
3.9833
3.9742
3.9639
3.8718
3.8512
3.8307
3.7534
3.7406
3.7349
3.7220
2.2878
0.94
1.06
1.02
1.04
1.02
1.03
1.05
1.08
1.02
1.00
5.61
2.16
1.06
2.05
0 ppm
1
2
3
4
5
6
7
8
9

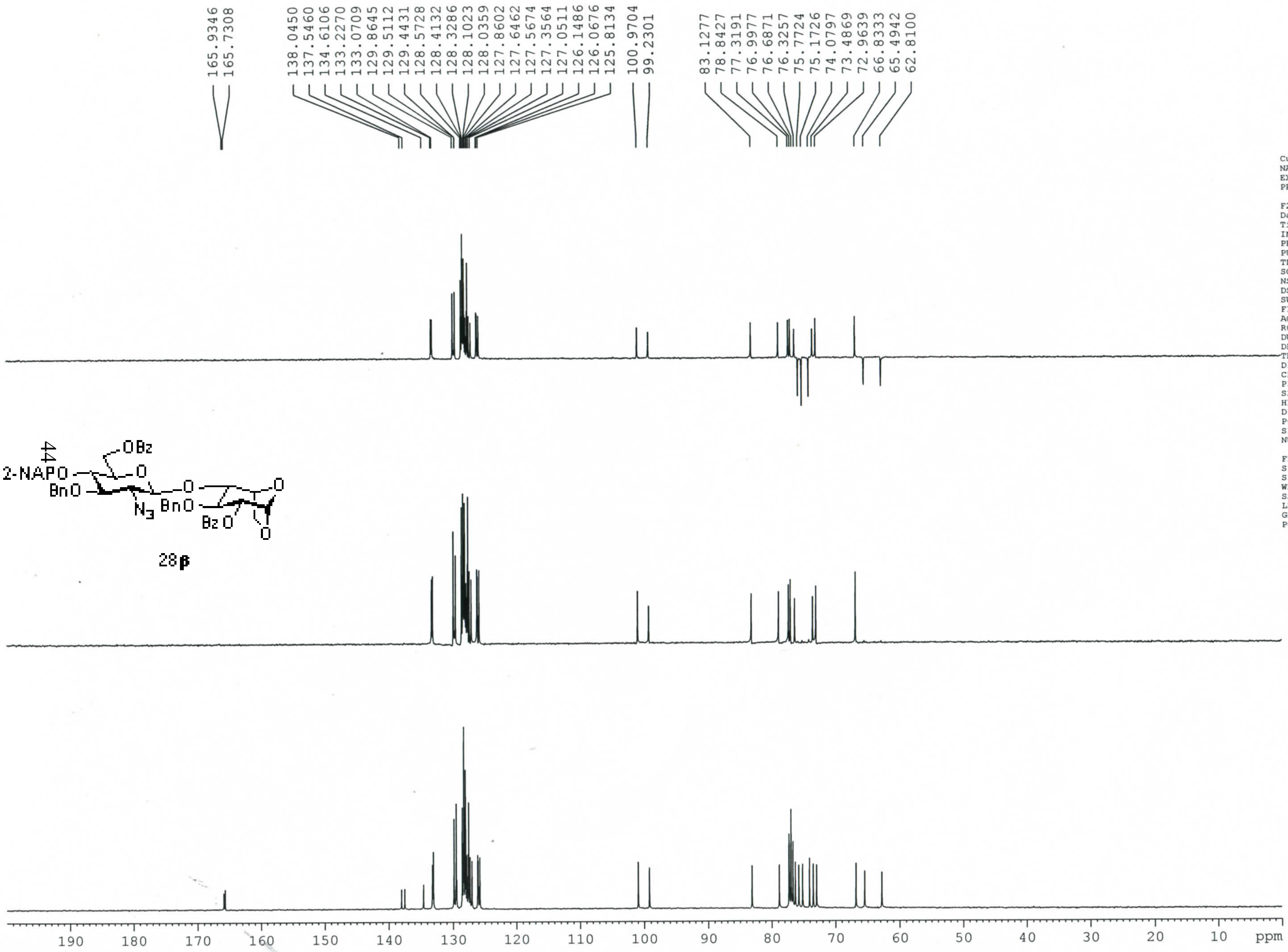



Current Data Parameters
NAME 1xa-414-p
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20010310
Time_ 16.26
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 64
DW 78.000 usec
DE 111.43 usec
TE 300.0 K
HL1 3 dB
D1 1.00000000 sec
P1 6.00 usec
SFO1 400.1319596 MHz
NUCLEUS 1H
F2 - Processing parameters
SI 32768
SF 400.1300172 MHz
WDW EM
SSB 0
LB 0.01 Hz
GB 0
PC 1.00
OBz
O
N3
BnO
BzO
O
O
2-NAPO
BnO
28α
ppm
0
1
2
3
4
5
6
7
8
9
1.00
1.00
4.21
1.03
1.04
1.10
2.29
1.30
1.22
3.41
1.31
1.27
1.36
1.21
2.02
2.10
3.17
1.05
2.12
12.43
5.24




Current Data Parameters
NAME LJC-754b
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20021004
Time 3.45
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 43
DS 2
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559940 sec
RG 64
DW 78.000 usec
DE 111.43 usec
TE 300.3 K
D1 1.00000000 sec
P1 6.00 usec
SFO1 400.1319596 MHz
NUCLEUS 1H
F2 - Processing parameters
SI 32768
SF 400.1300172 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
2-NAPO
BnO
OBz
N3
BnO
BzO
28β
ppm

Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry



Current Data Parameters

NAME	LJC-754b
EXPNO	3
PROCNO	1

F2 - Acquisition Parameters

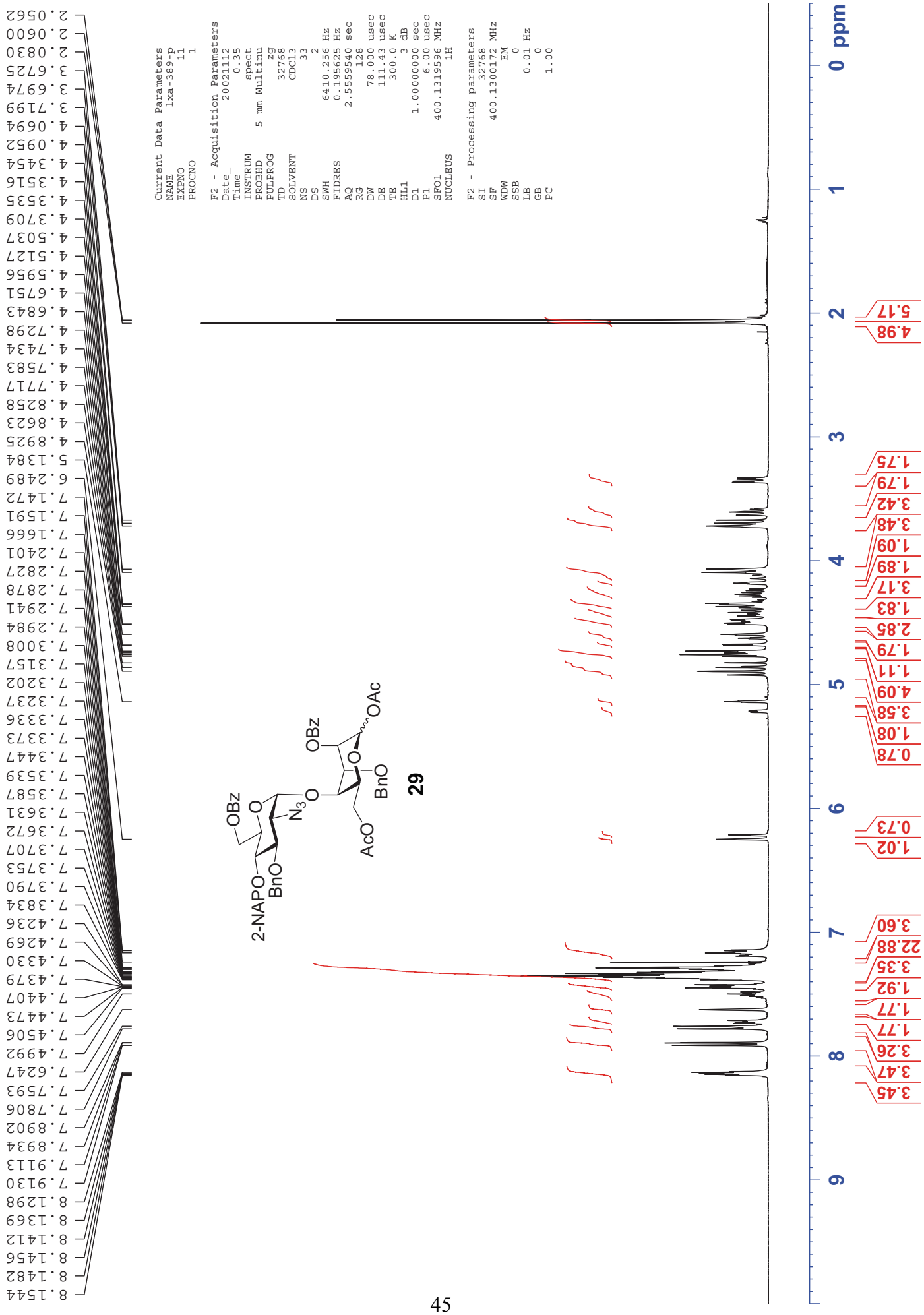
Date_	20021004
Time	3.52
INSTRUM	spect
PROBHD	5 mm Multinu
PULPROG	zg0dc
TD	32768
SOLVENT	CDCl3
NS	3072
DS	2
SWH	31250.000 Hz
FIDRES	0.953674 Hz
AQ	0.5243380 sec
RG	32768
DW	16.000 usec
DE	20.00 usec
TE	300.0 K
D11	0.03000000 sec
CPDPRG	waltz16
P31	100.00 usec
S2	28 dB
HL1	3 dB
D1	3.00000000 sec
P0	8.00 usec
SFO1	100.6273592 MHz
NUCLEUS	13C

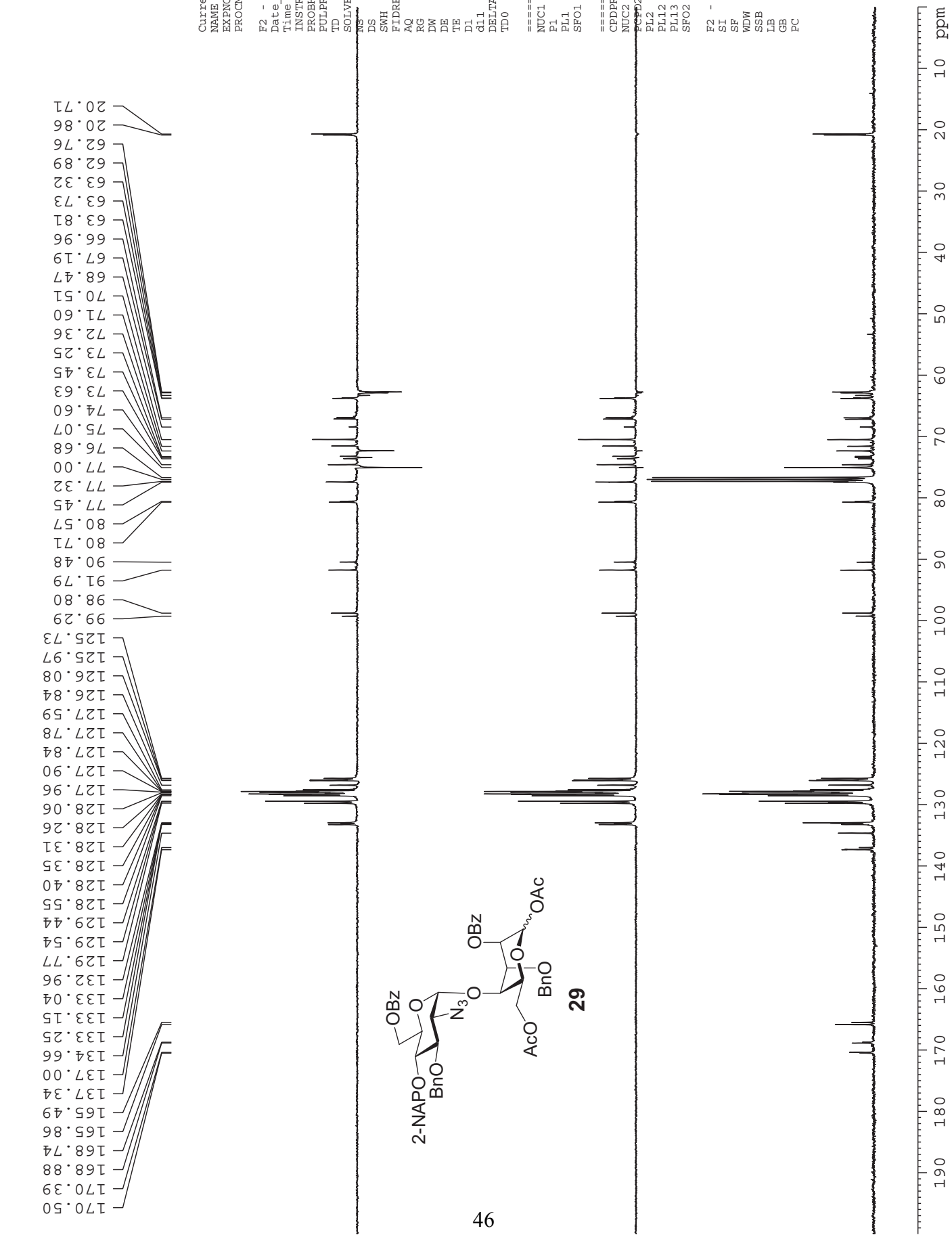
F2 - Processing parameters

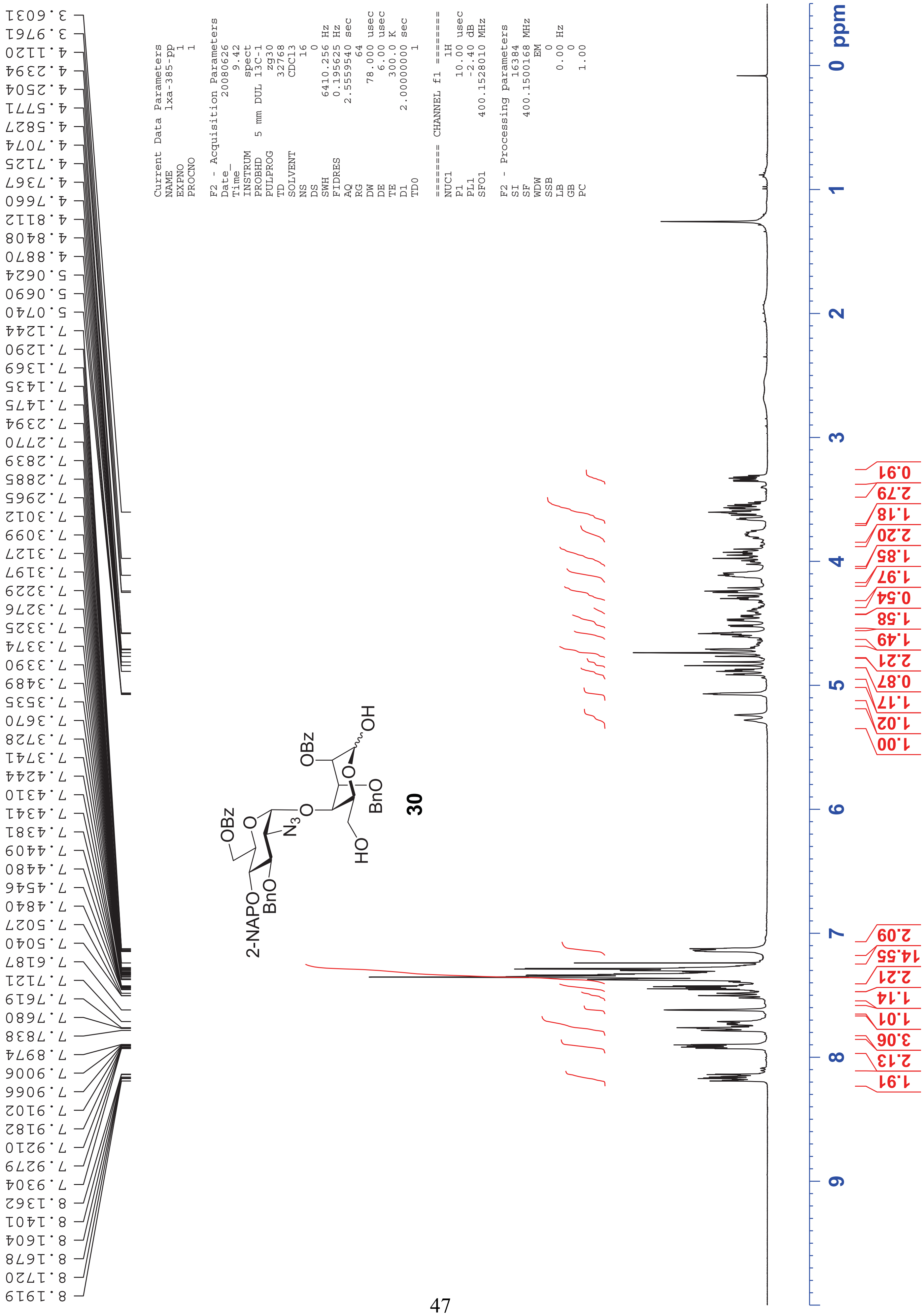
SI	32768
SF	100.6127760 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

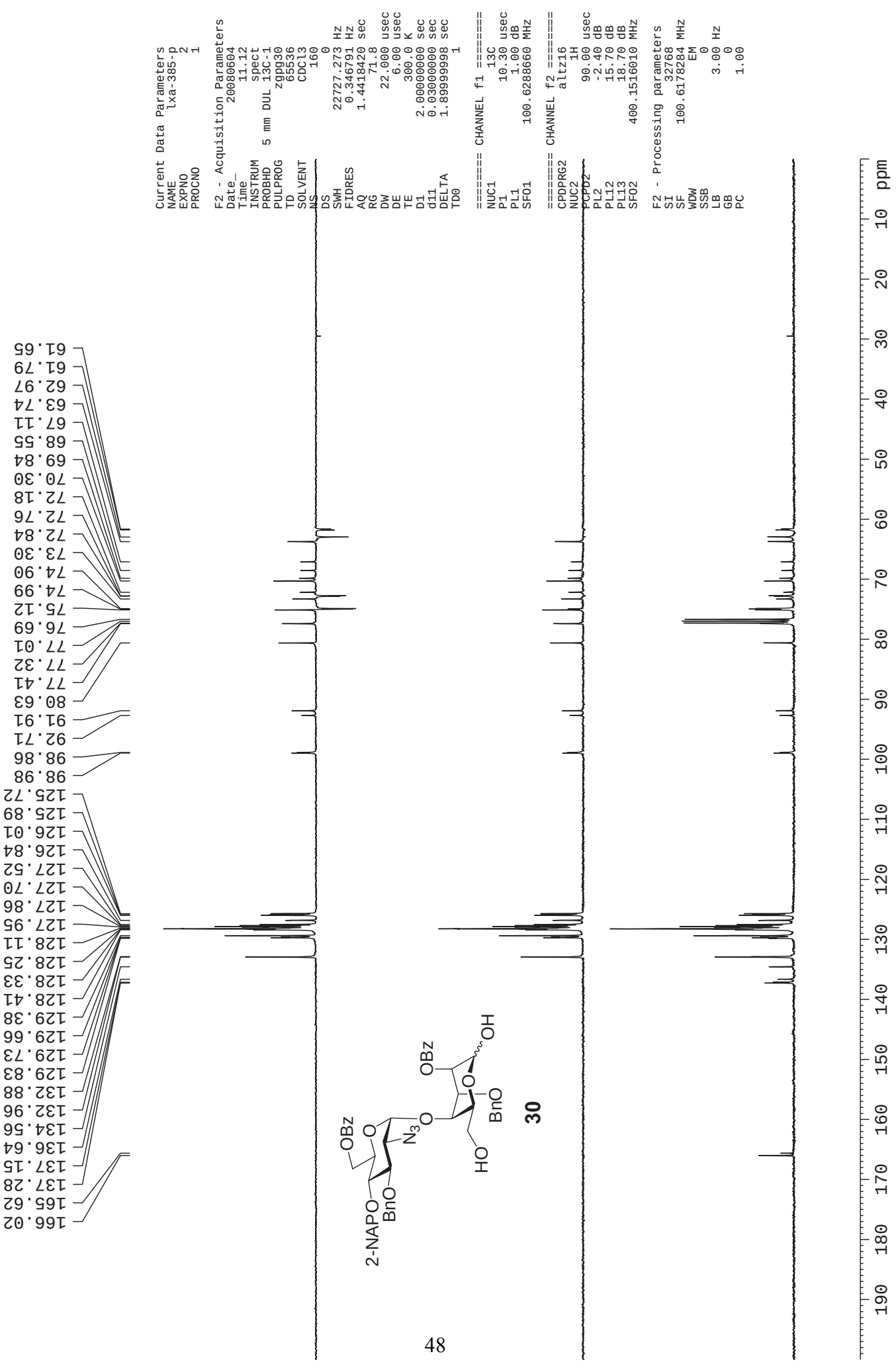
44

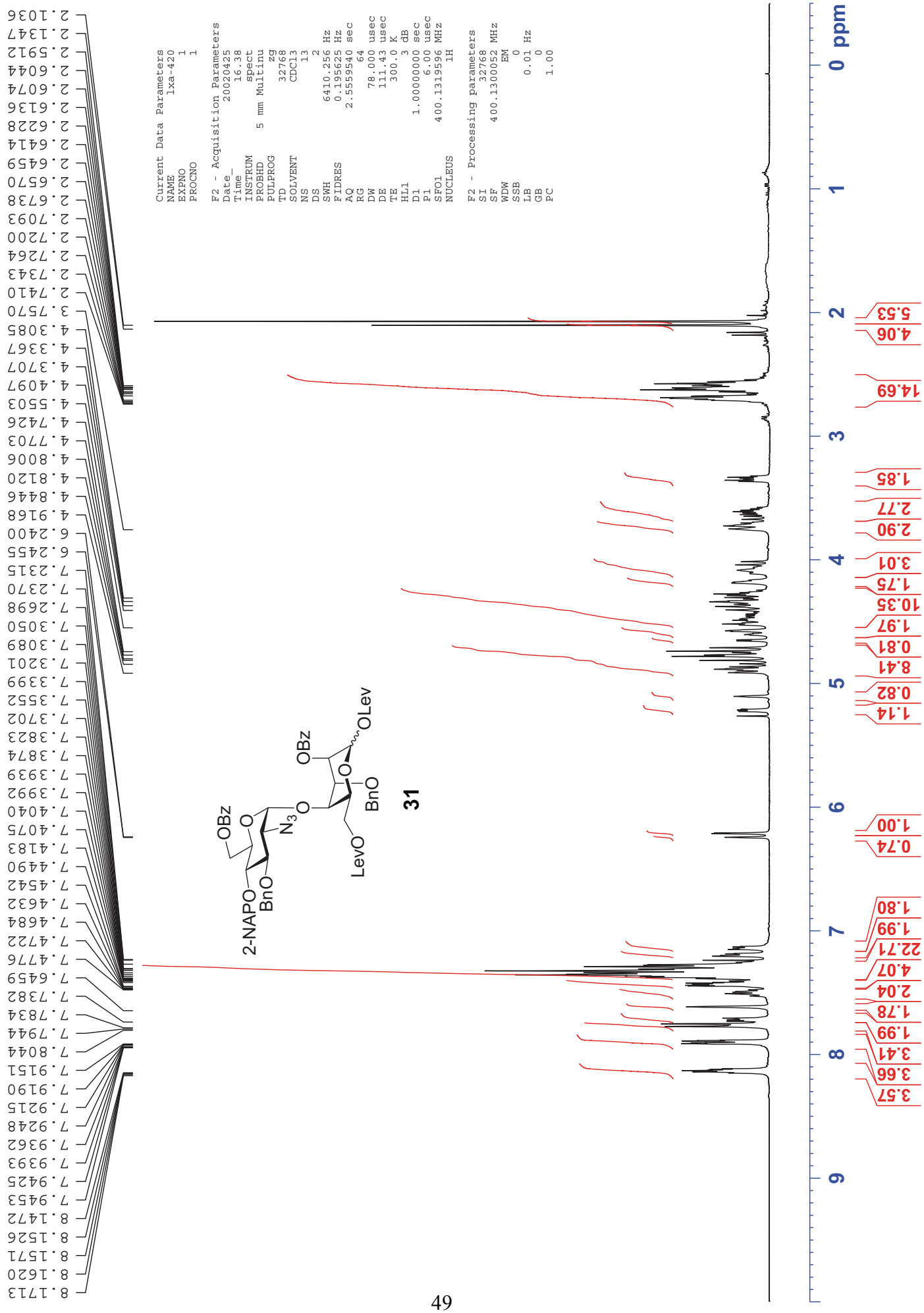
28β

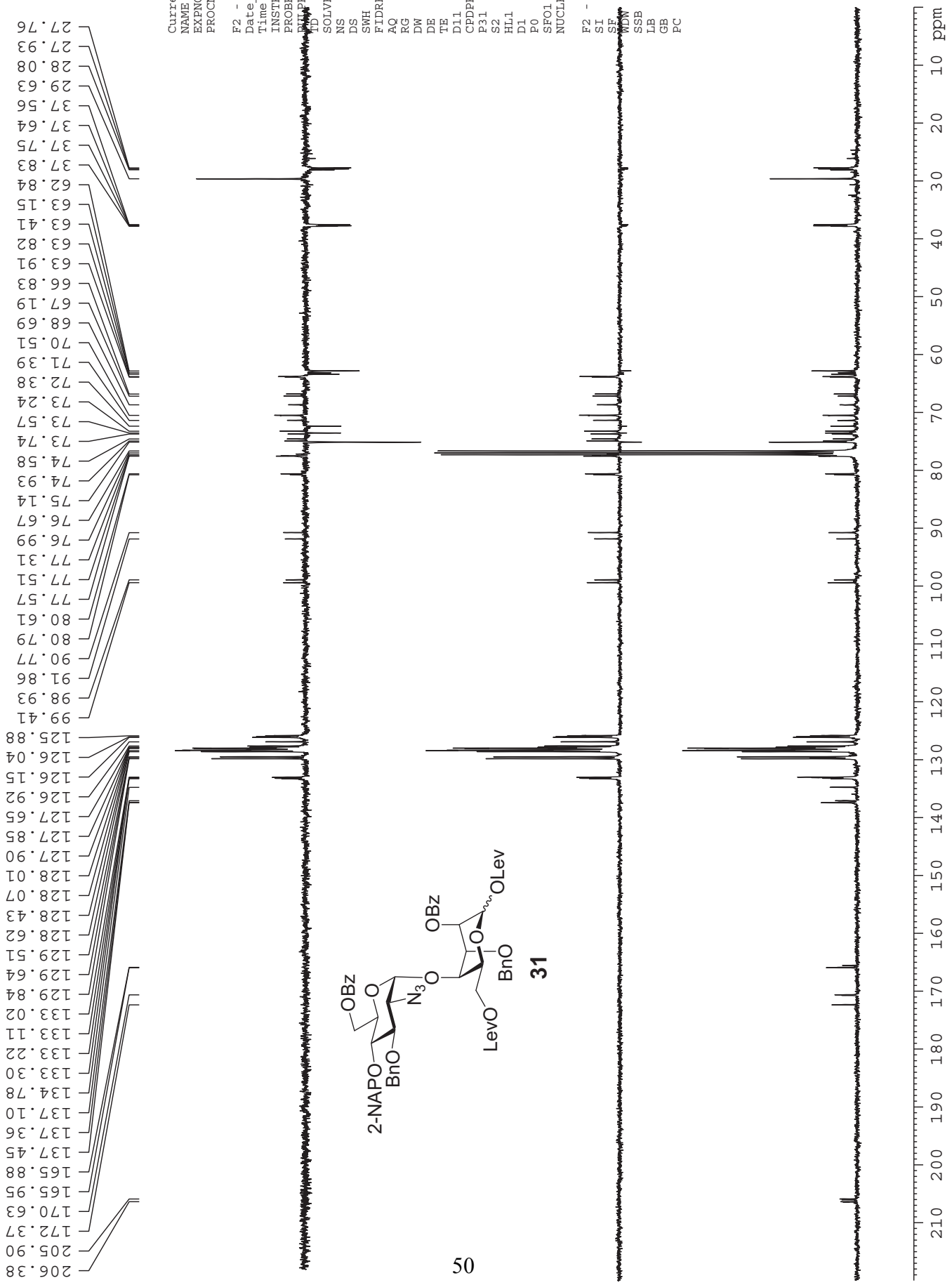

Current Data Parameters
NAME 1xa-389-p
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20021112
Time 0.35
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 128
DW 78.000 usec
DE 111.43 usec
TE 300.0 K
HL1 3 dB
D1 1.00000000 sec
P1 6.00 usec
SFO1 400.1319596 MHz
NUCLEUS 1H
F2 - Processing parameters
SI 32768
SF 400.1300172 MHz
WDW EM
SSB 0
LB 0.01 Hz
GB 0
PC 1.00
OBz
O
N3
O
OBz
OAc
O
BnO
AcO
2-NAPO
BnO
29
ppm
0
1
2
3
4
5
6
7
8
9
5.17
4.98
1.75
1.79
3.42
3.48
1.09
1.89
3.17
1.83
2.85
1.79
1.11
4.09
3.58
1.08
0.78
1.02
0.73
3.60
22.88
3.35
1.92
1.77
1.77
3.26
3.47
3.45


Current Data Parameters
NAME 1xa-389-p
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080618
Time_ 11.05
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 400
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 71.8
DW 22.000 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 10.30 usec
PL1 1.00 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.70 dB
PL13 18.70 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6177980 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
20.71
20.86
62.76
62.89
63.32
63.73
63.81
66.96
67.19
68.47
70.51
71.60
72.36
73.25
73.45
73.63
74.60
75.07
76.68
77.00
77.32
77.45
80.57
80.71
90.48
91.79
98.80
99.29
125.73
125.97
126.08
126.84
127.59
127.78
127.84
127.90
127.96
128.06
128.26
128.31
128.35
128.40
128.55
129.44
129.54
129.77
132.96
133.04
133.15
133.25
134.66
137.00
137.34
165.49
165.86
168.74
168.88
170.39
170.50
OBz
O
N3
O
2-NAPO
BnO
OBz
O
OAc
BnO
AcO
29
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


Current Data Parameters
NAME lxa-385-pp
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080626
Time 9.42
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 64
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500168 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
2-NAPO
BnO
OBz
N3
O
HO
BnO
OBz
OH
30
ppm


Current Data Parameters
NAME lxa-385-p
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080604
Time 11.12
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 160
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 71.8
DW 22.000 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 10.30 usec
PL1 1.00 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 altz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.70 dB
PL13 18.70 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178284 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
166.02
165.62
137.28
137.15
136.64
134.56
132.96
132.88
129.83
129.73
129.66
129.38
128.41
128.33
128.25
128.11
127.95
127.86
127.70
127.52
126.84
126.01
125.89
125.72
98.98
98.86
92.71
91.91
80.63
77.41
77.32
77.01
76.69
75.12
74.99
74.90
73.30
72.84
72.76
72.18
70.30
69.84
68.55
67.11
63.74
62.97
61.79
61.65
OBz
O
2-NAPO
BnO
N3
O
OBz
O
BnO
HO
OH
30
ppm


Current Data Parameters
NAME 1xa-420
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20020425
Time_ 16.38
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 13
DS 2
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 64
DW 78.000 usec
DE 111.43 usec
TE 300.0 K
HL1 3 dB
D1 1.00000000 sec
P1 6.00 usec
SFO1 400.1319596 MHz
NUCLEUS 1H
F2 - Processing parameters
SI 32768
SF 400.1300052 MHz
WDW EM
SSB 0
LB 0.01 Hz
GB 0
PC 1.00
OBz
O
N3
O
OBz
OLev
O
BnO
2-NAPO
BnO
LevO
31
ppm
0
1
2
3
4
5
6
7
8
9
5.53
4.06
14.69
1.85
2.77
2.90
3.01
1.75
10.35
1.97
0.81
8.41
0.82
1.14
1.00
0.74
1.80
1.99
22.71
4.07
2.04
1.78
1.99
3.41
3.66
3.57


Current Data Parameters
NAME lxa-420
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20020428
Time 20.32
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg0dc
TD 32768
SOLVENT CDCl3
NS 4096
DS 2
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 0.5243380 sec
RG 32768
DW 16.000 usec
DE 20.00 usec
TE 300.0 K
D11 0.03000000 sec
CPDPRG waltz16
P31 104.00 usec
S2 30 dB
HL1 3 dB
D1 3.00000000 sec
P0 5.00 usec
SFO1 100.6273592 MHz
NUCLEUS 13C
F2 - Processing parameters
SI 32768
SF 100.6127589 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
99.41
98.93
91.86
90.77
80.79
80.61
77.57
77.51
77.31
76.99
76.67
75.14
74.93
74.58
73.74
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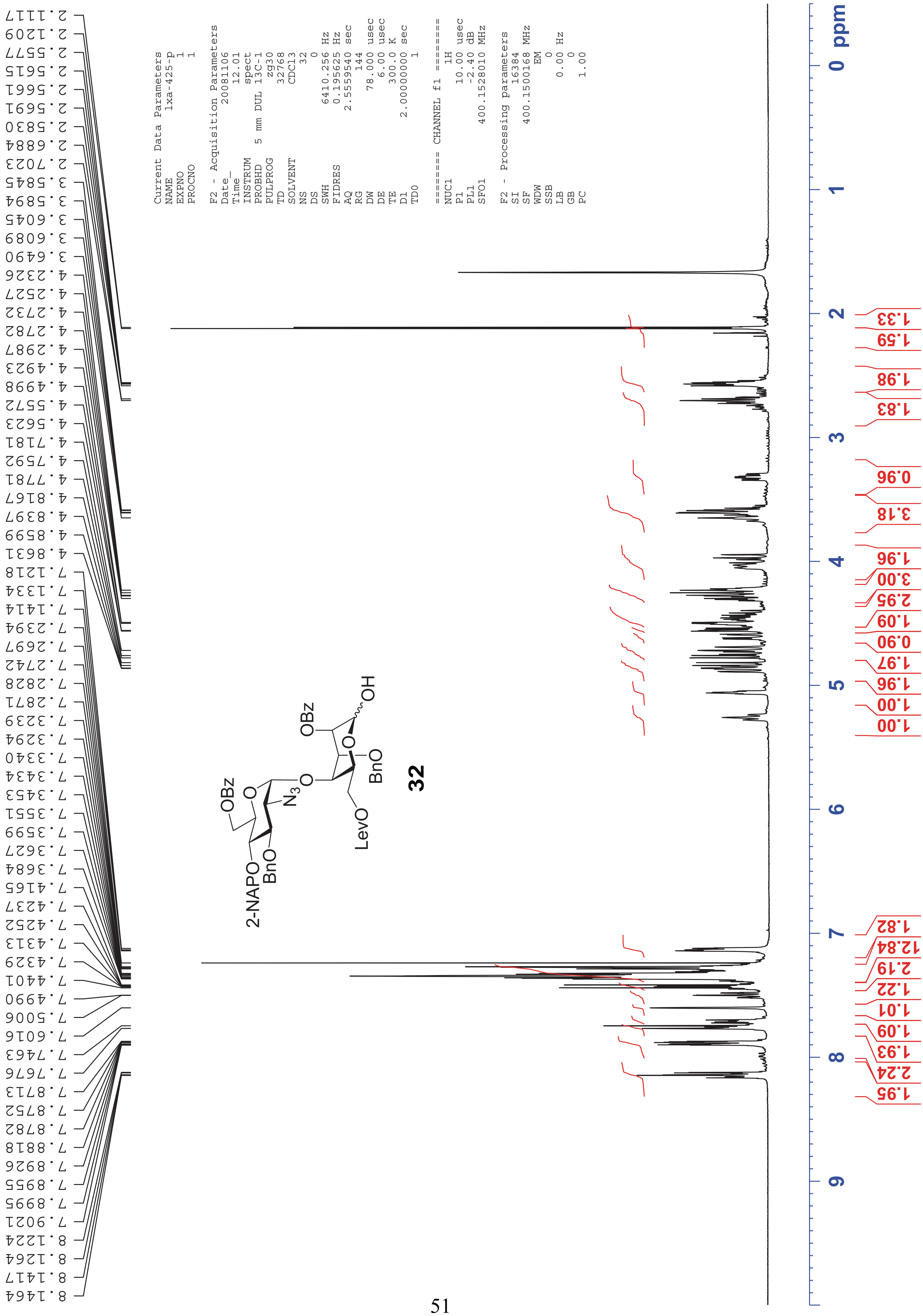
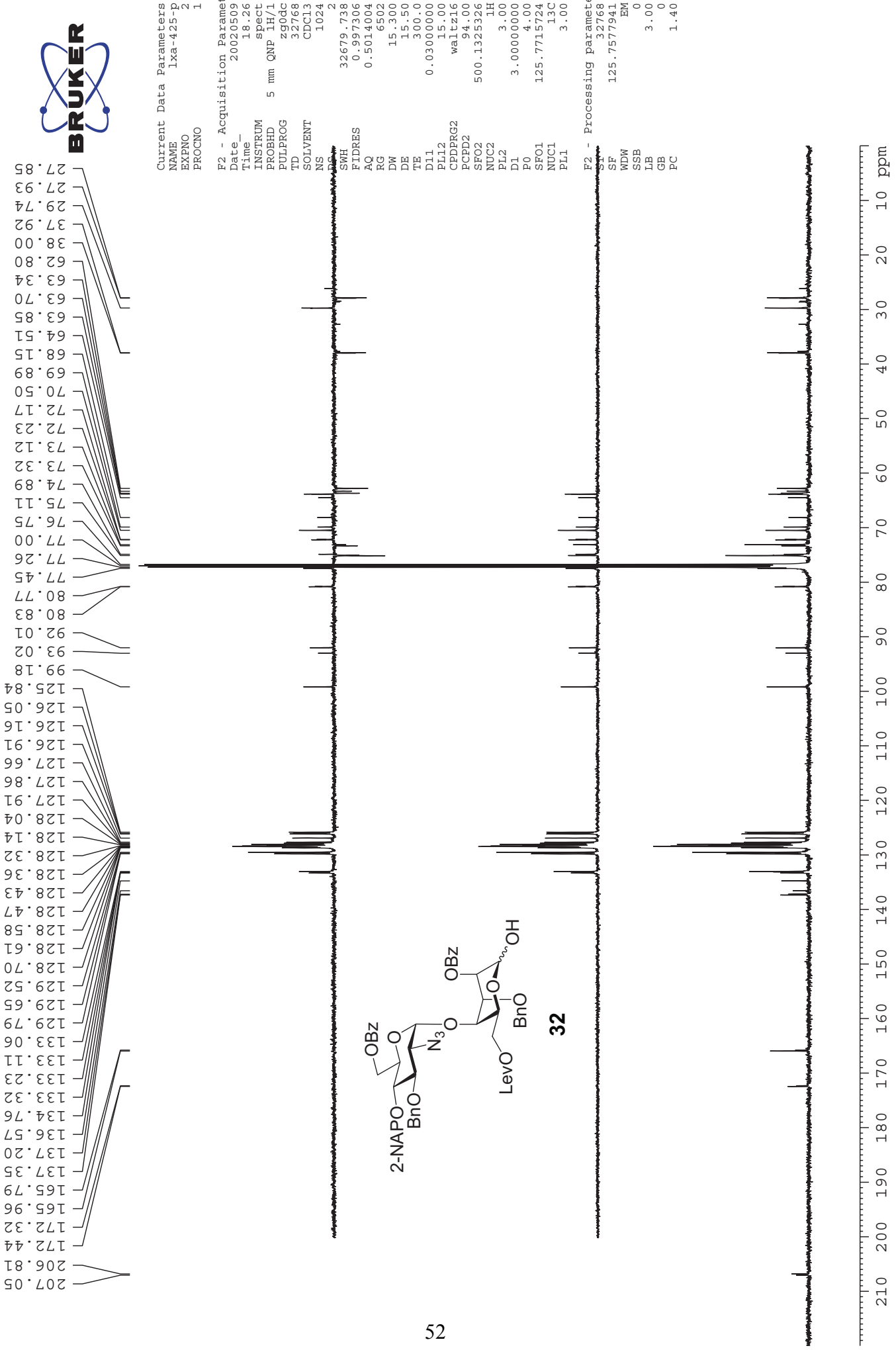
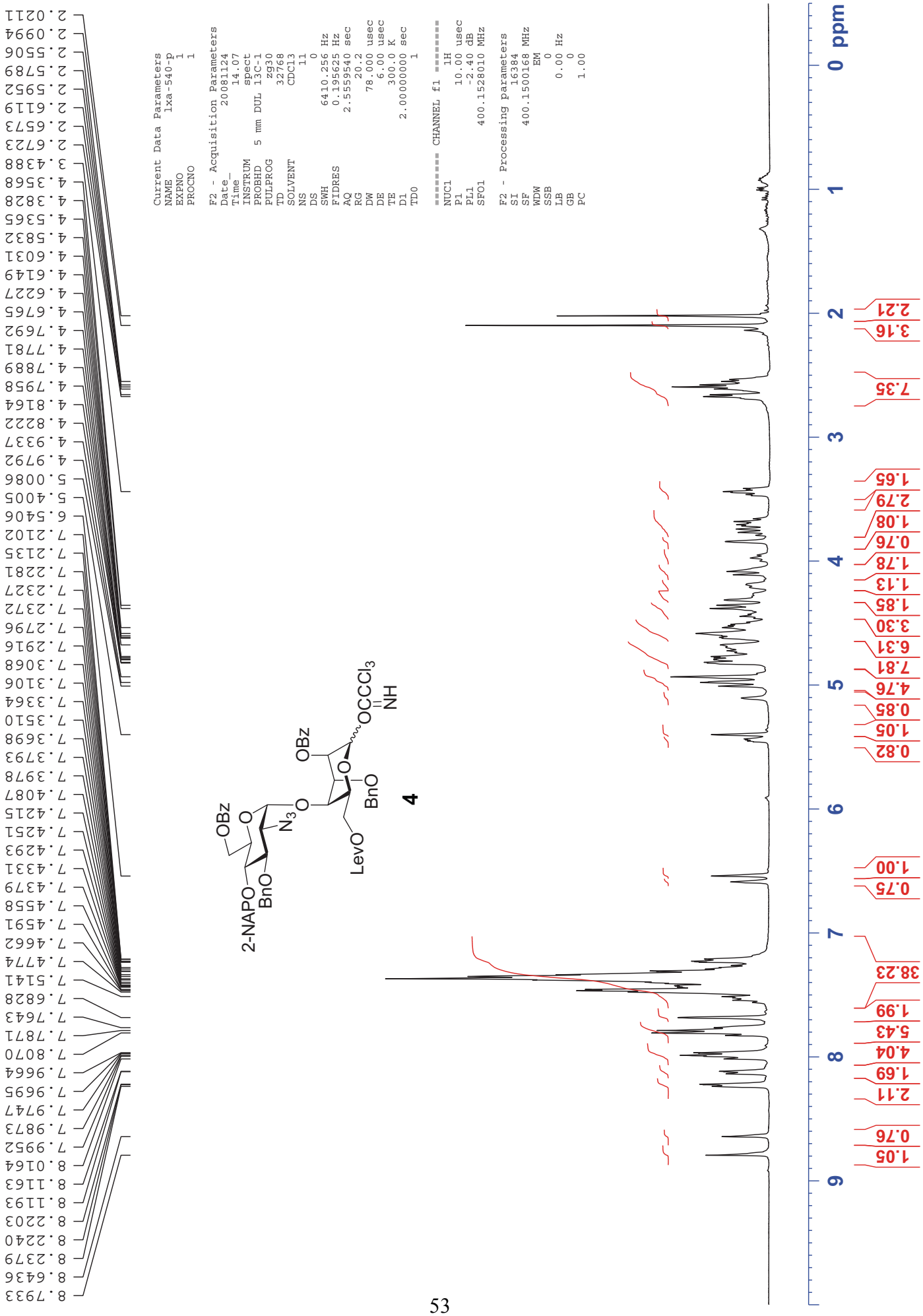
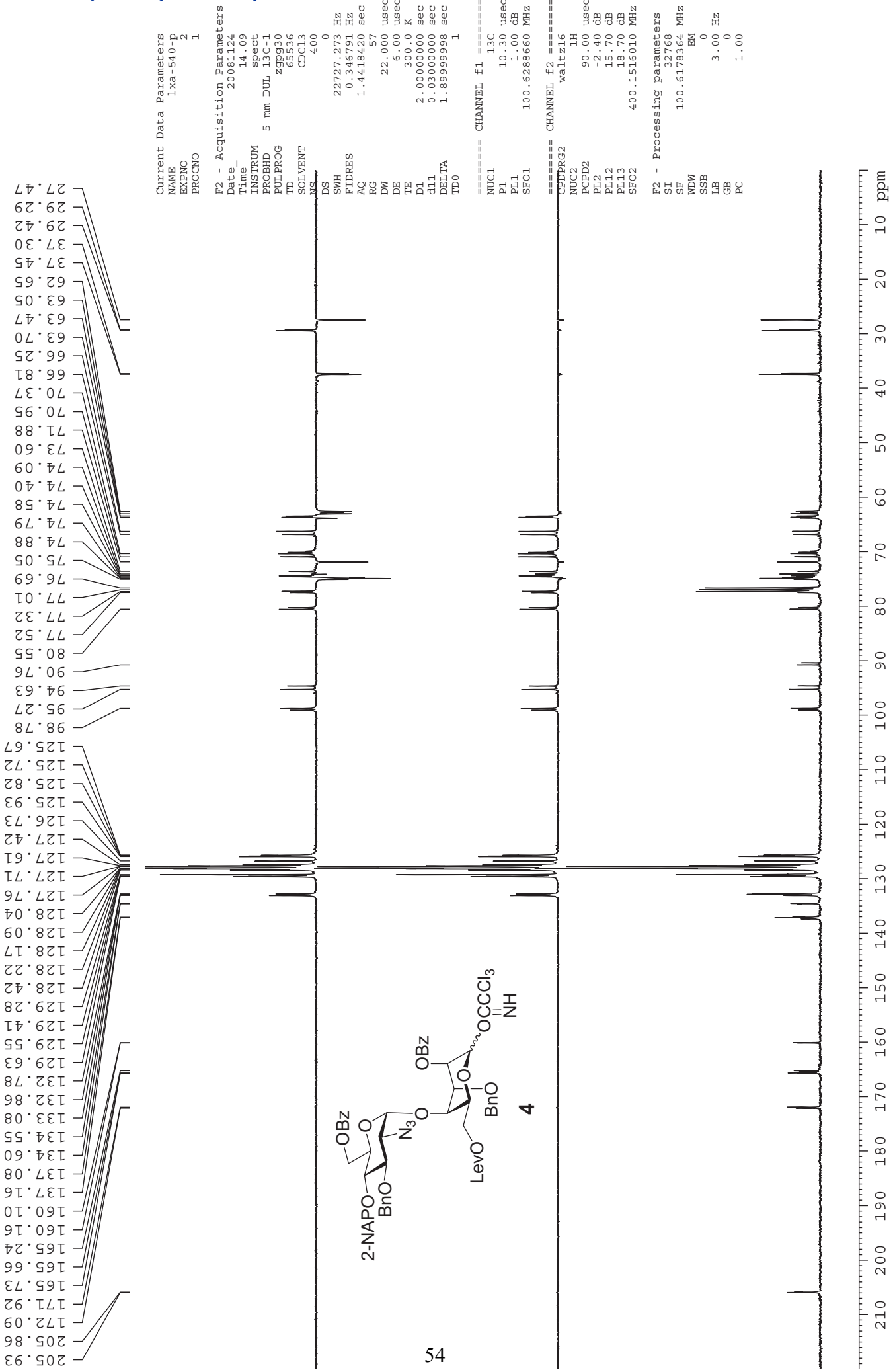
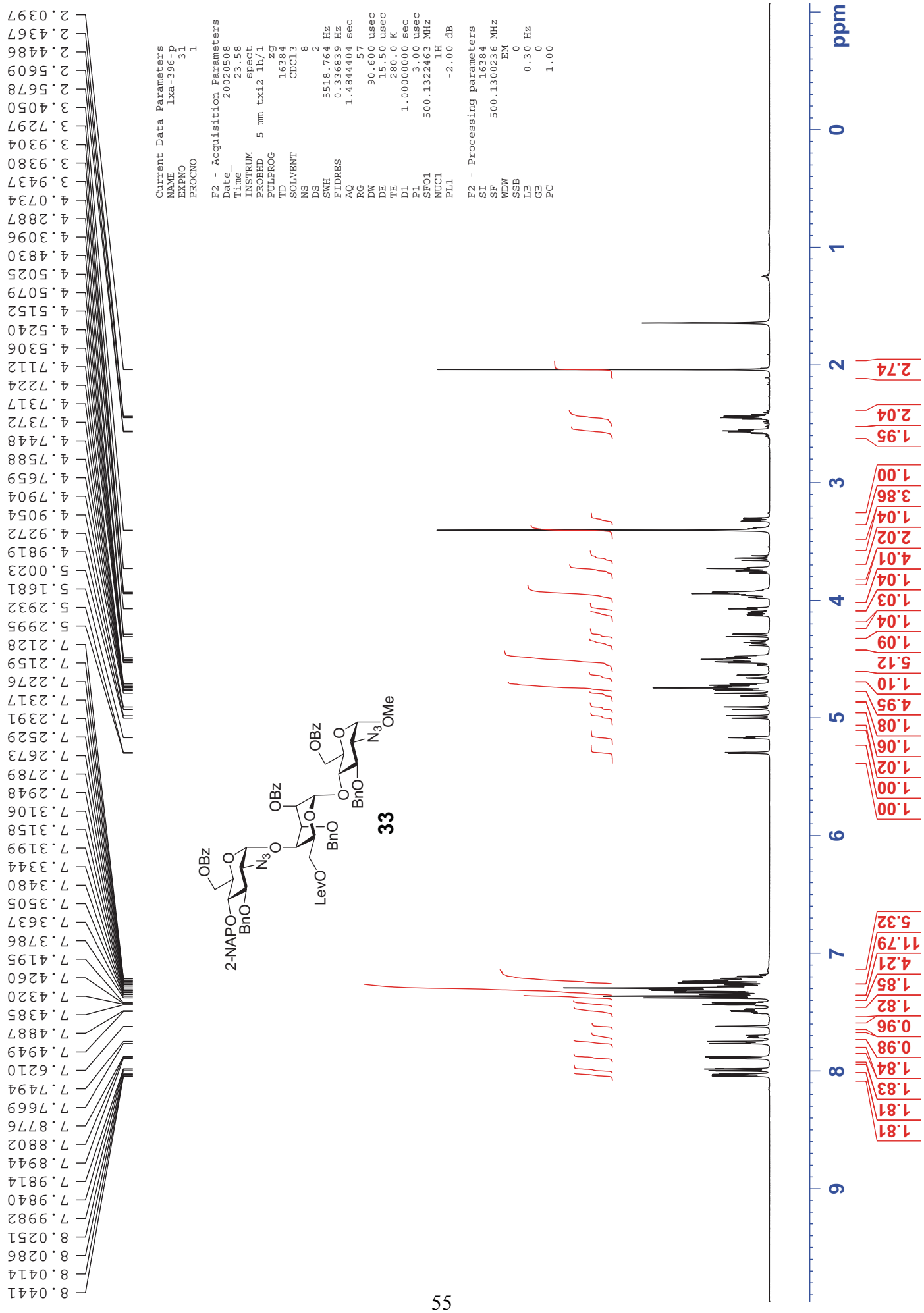
73.24
72.38
71.39
70.51
69.19
68.69
67.19
66.83
63.91
63.82
63.41
63.15
62.84
37.83
37.75
37.64
37.56
29.63
28.08
27.93
27.76
206.38
205.90
172.37
170.63
165.95
165.88
137.45
137.36
137.10
134.78
133.30
133.22
133.11
133.02
129.84
129.64
129.51
128.62
128.43
128.07
128.01
127.90
127.85
127.65
126.92
126.15
126.04
125.88
OBz
O
2-NAPO
BnO
N_3
O
OBz
O
BnO
LevO
OLev
31
ppm

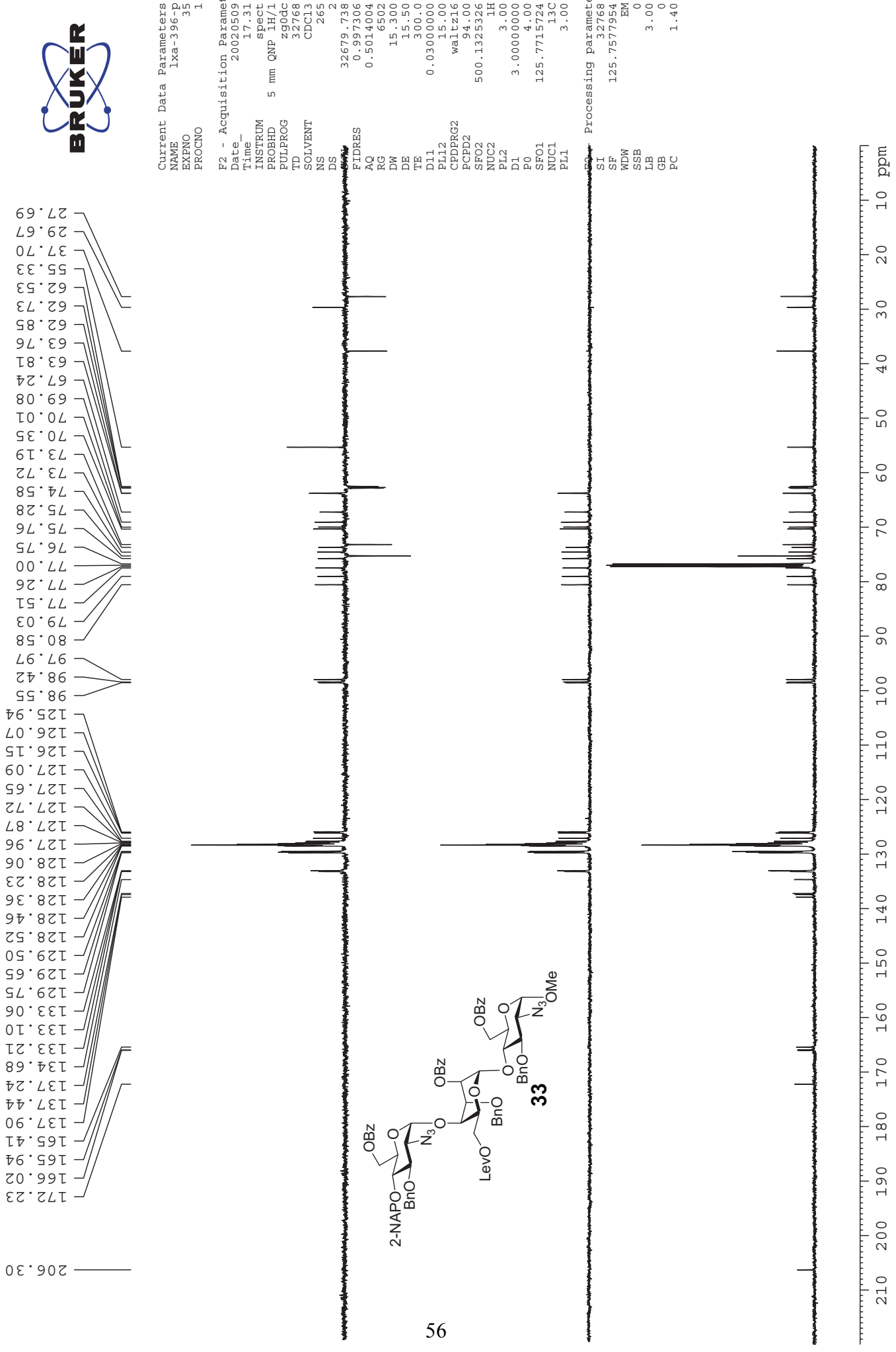

Current Data Parameters
NAME lxa-425-p
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081106
Time 12.01
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 144
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500168 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
OBz
2-NAPO
BnO
O
N3
OBz
O
OH
BnO
LevO
32
ppm

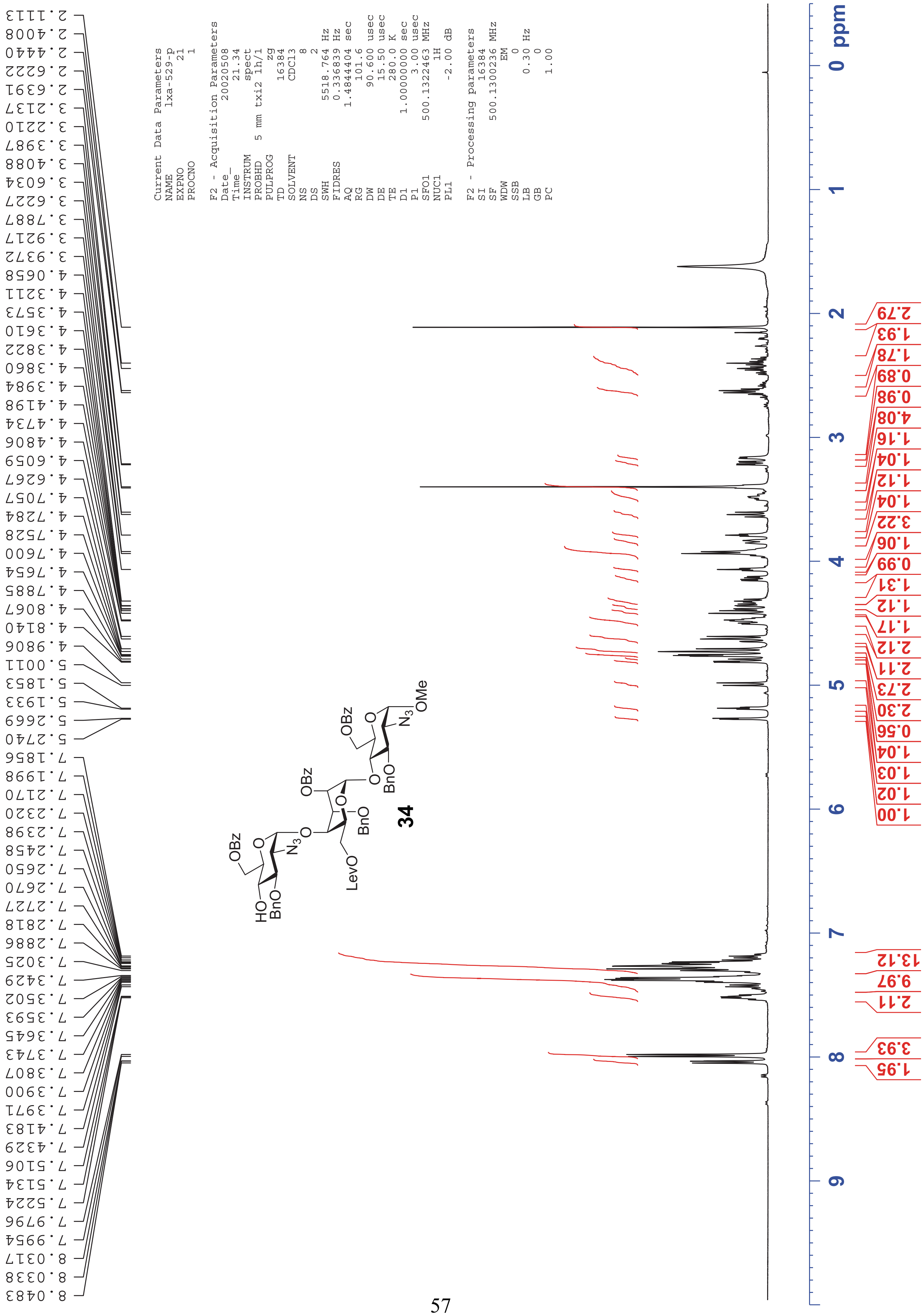


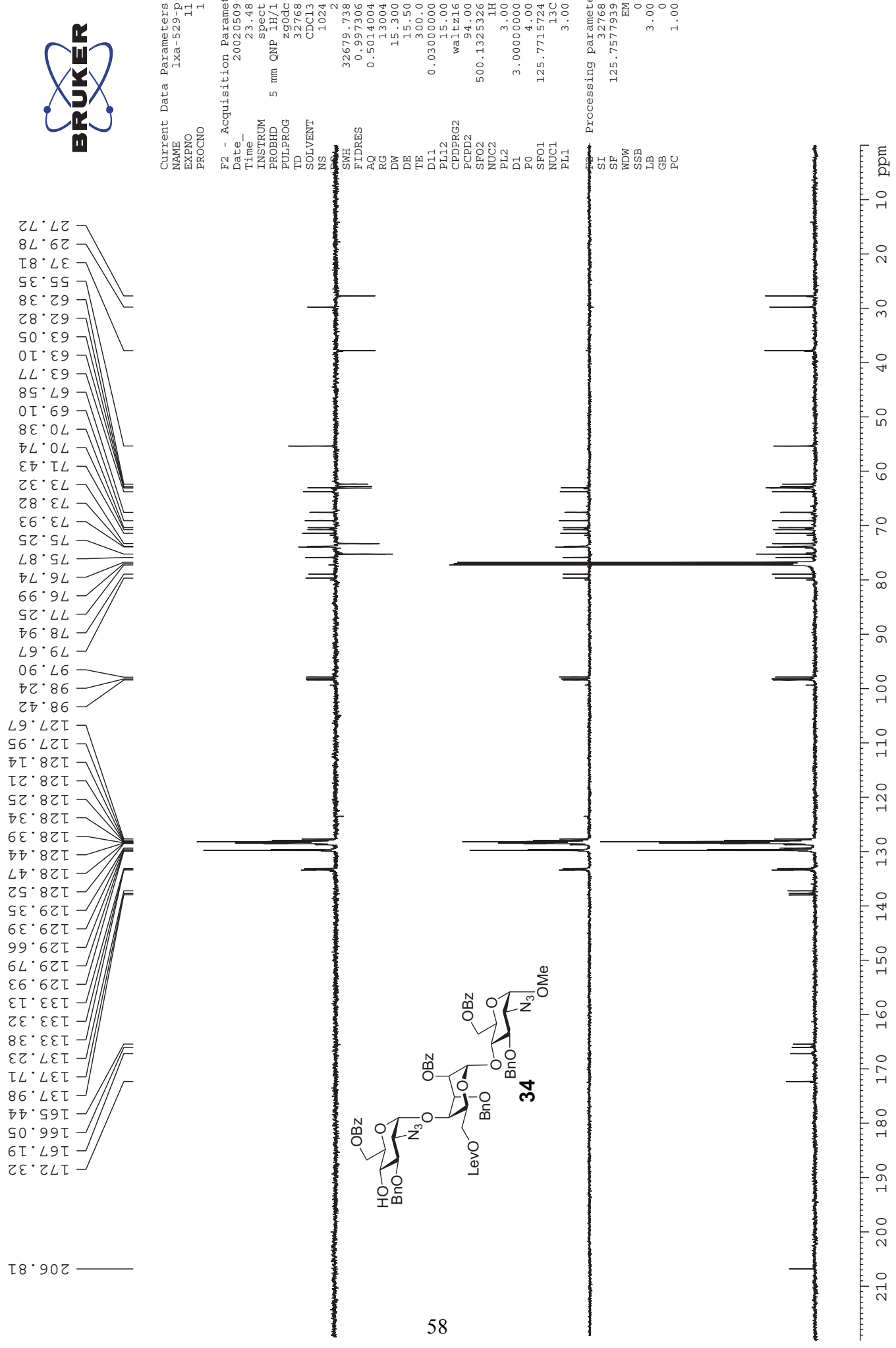

Current Data Parameters
NAME 1xa-540-p
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081124
Time 14.07
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 11
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 20.2
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500168 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
OBz
2-NAPO
BnO
O
N3
OBz
O
BnO
LevO
OCCCl3
NH
4
ppm
0 1 2 3 4 5 6 7 8 9
2.21
3.16
7.35
1.65
2.79
1.08
0.76
1.78
1.13
1.85
3.30
6.31
7.81
4.76
0.85
1.05
0.82
1.00
0.75
38.23
1.99
5.43
4.04
1.69
2.11
0.76
1.05

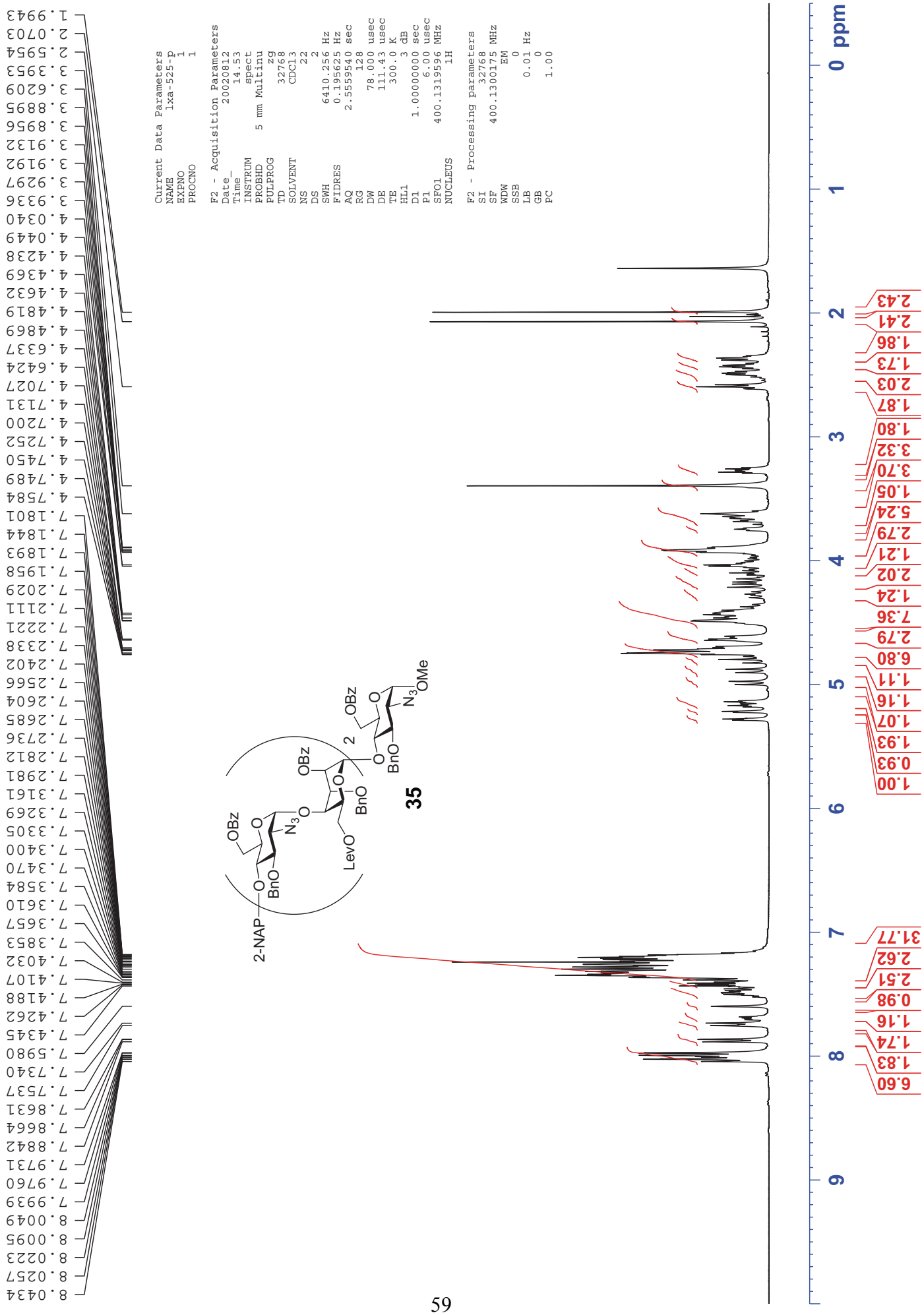


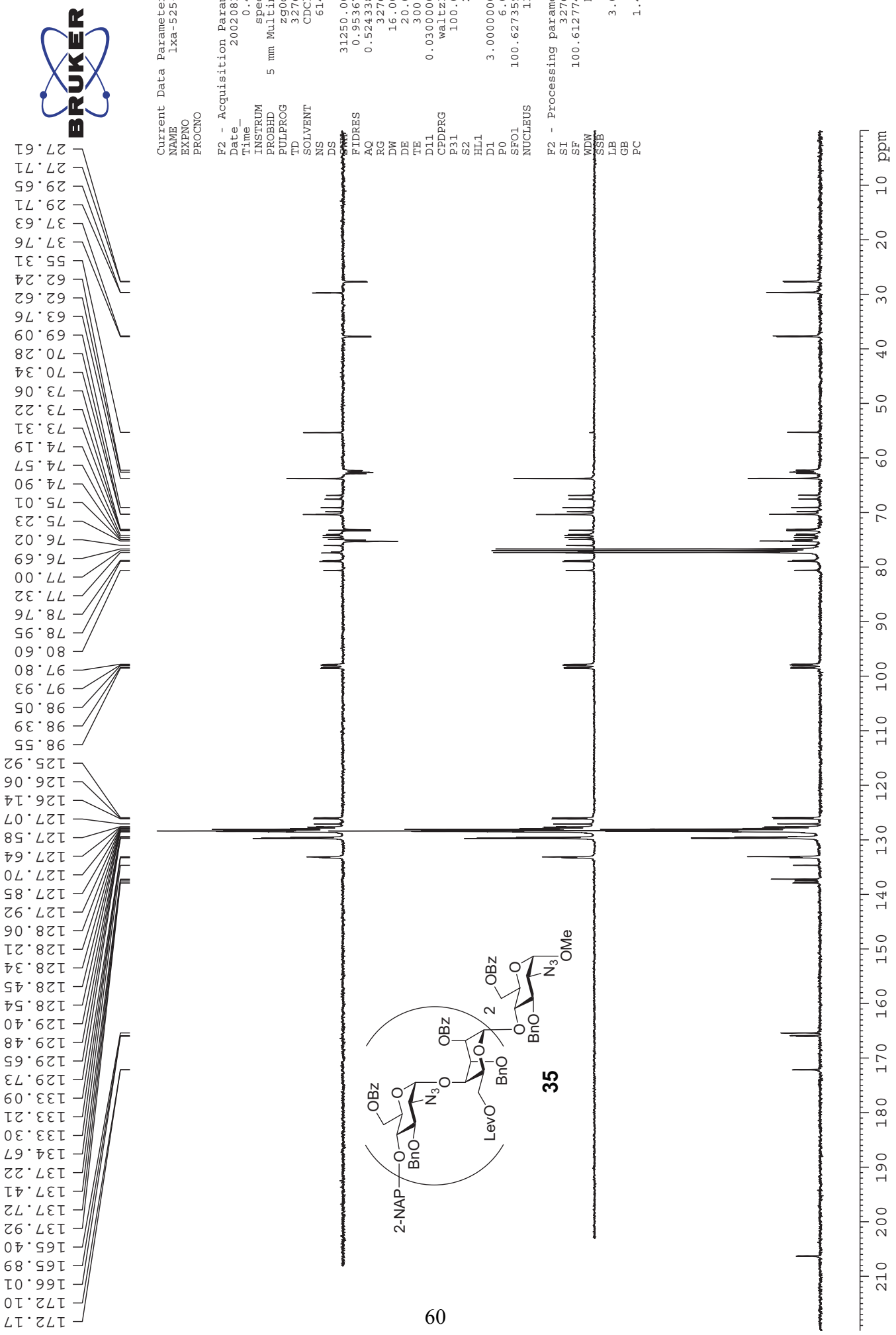

Current Data Parameters
NAME 1xa-396-p
EXPNO 31
PROCNO 1
F2 - Acquisition Parameters
Date_ 20020508
Time 23.58
INSTRUM spect
PROBHD 5 mm txi2 1h/1
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 8
DS 2
SWH 5518.764 Hz
FIDRES 0.336839 Hz
AQ 1.4844404 sec
RG 57
DW 90.600 usec
DE 15.50 usec
TE 280.0 K
D1 1.00000000 sec
P1 3.00 usec
SFO1 500.1322463 MHz
NUC1 1H
PL1 -2.00 dB
F2 - Processing parameters
SI 16384
SF 500.1300236 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
2.74
2.04
1.95
1.00
3.86
1.04
2.02
4.01
1.04
1.03
1.04
1.09
5.12
1.10
4.95
1.08
1.06
1.02
1.00
1.00
5.32
11.79
4.21
1.85
1.82
0.96
0.98
1.84
1.83
1.81
1.81
OBz
2-NAPO
BnO
LevO
BnO
OBz
BnO
OBz
OMe
33

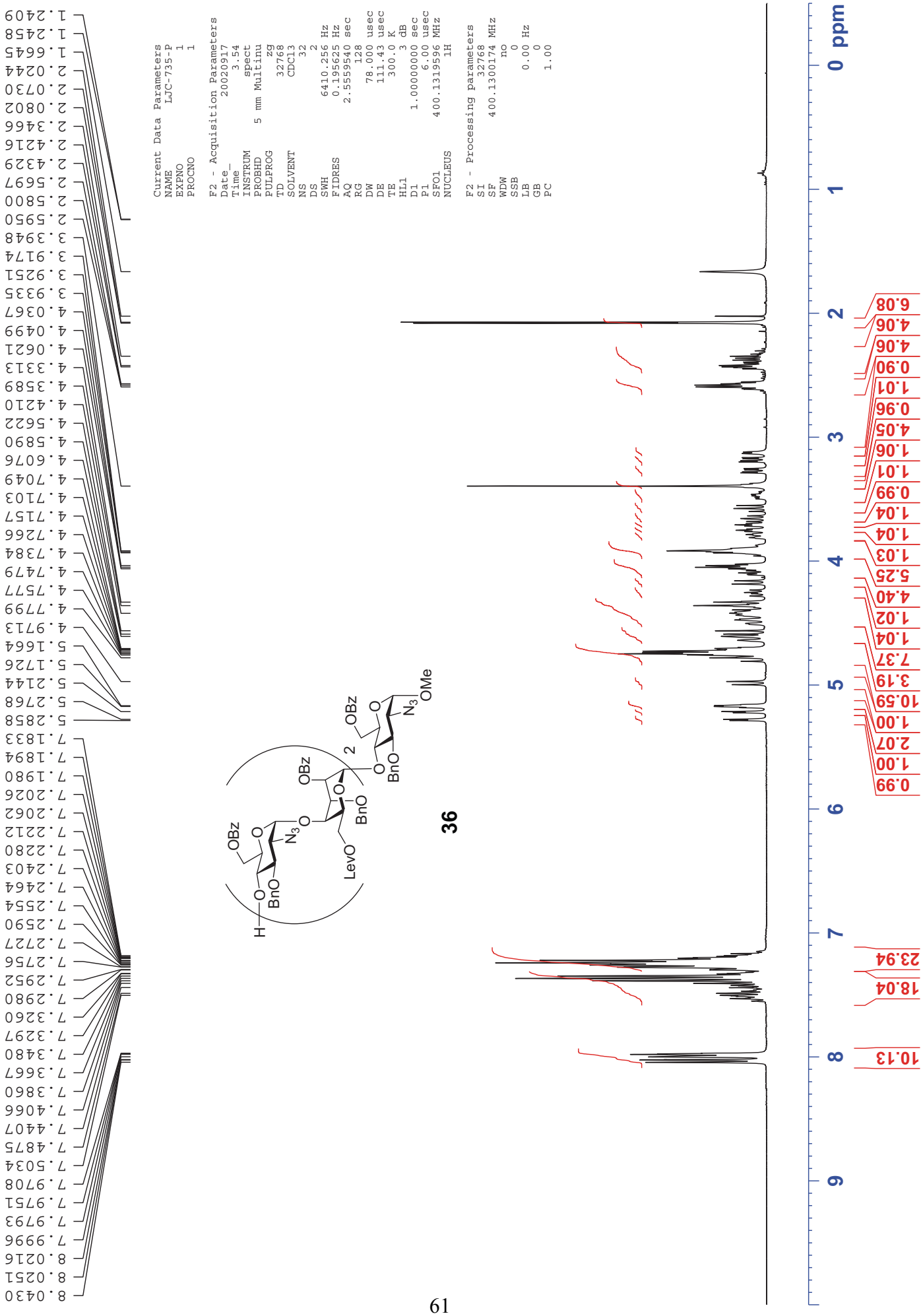


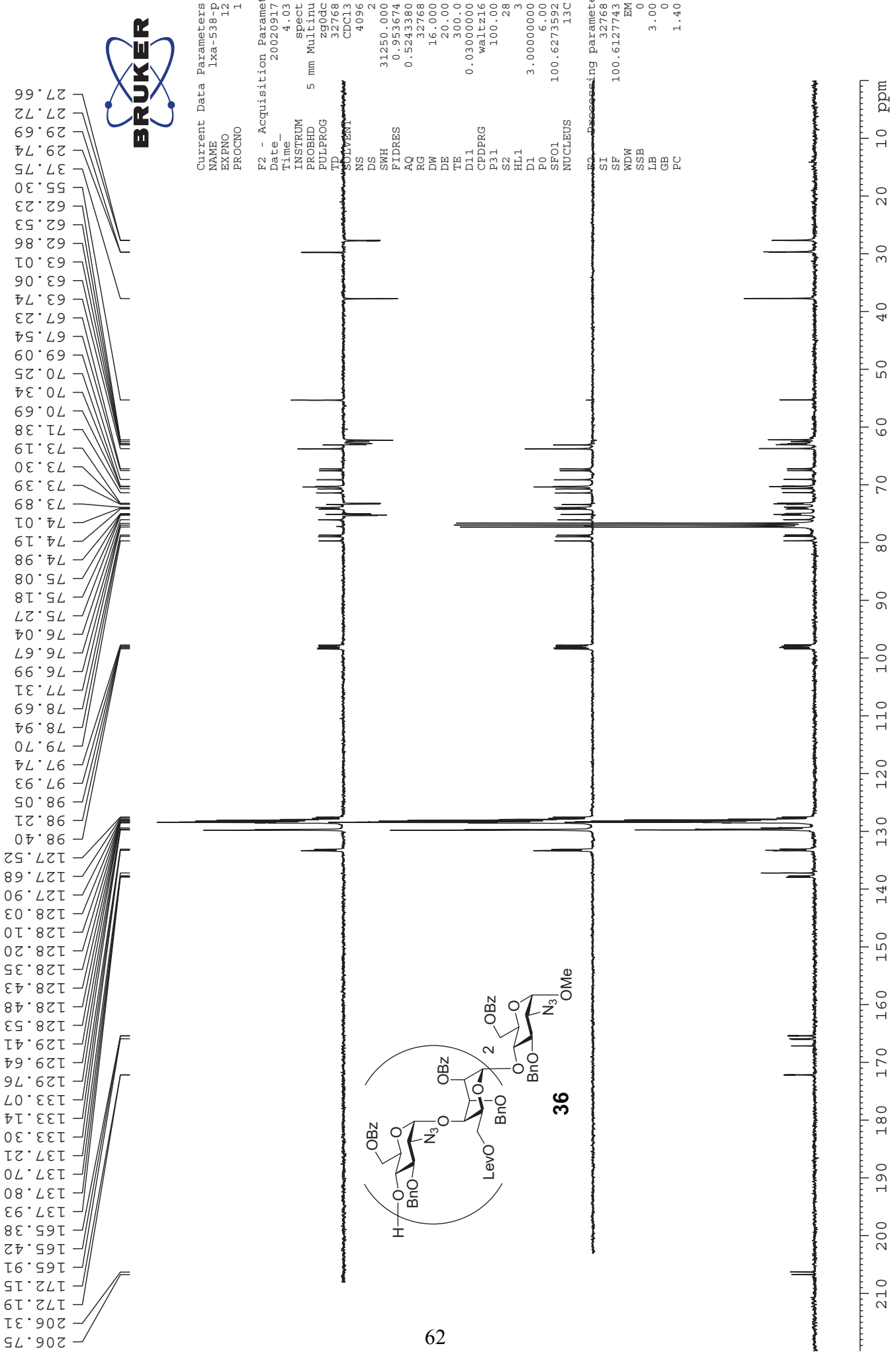

Current Data Parameters
NAME lxa-529-p
EXPNO 21
PROCNO 1
F2 - Acquisition Parameters
Date_ 20020508
Time 21.34
INSTRUM spect
PROBHD 5 mm txi2 1h/1
PULPROG zg
TD 16384
SOLVENT CDC13
NS 8
DS 2
SWH 5518.764 Hz
FIDRES 0.336839 Hz
AQ 1.484404 sec
RG 101.6
DW 90.600 usec
DE 15.50 usec
TE 280.0 K
D1 1.00000000 sec
P1 3.00 usec
SFO1 500.1322463 MHz
NUC1 1H
PL1 -2.00 dB
F2 - Processing parameters
SI 16384
SF 500.1300236 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
34
ppm

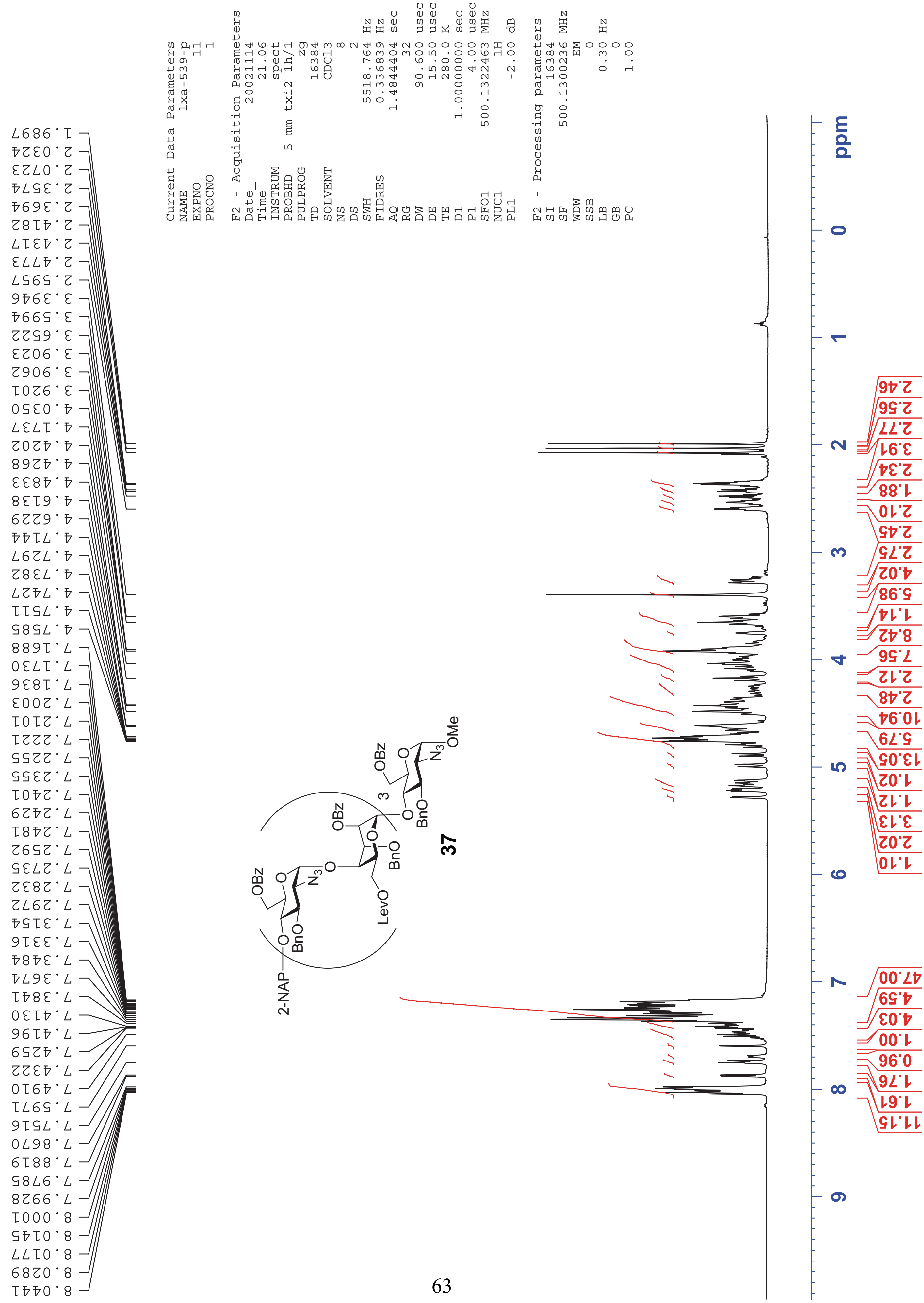


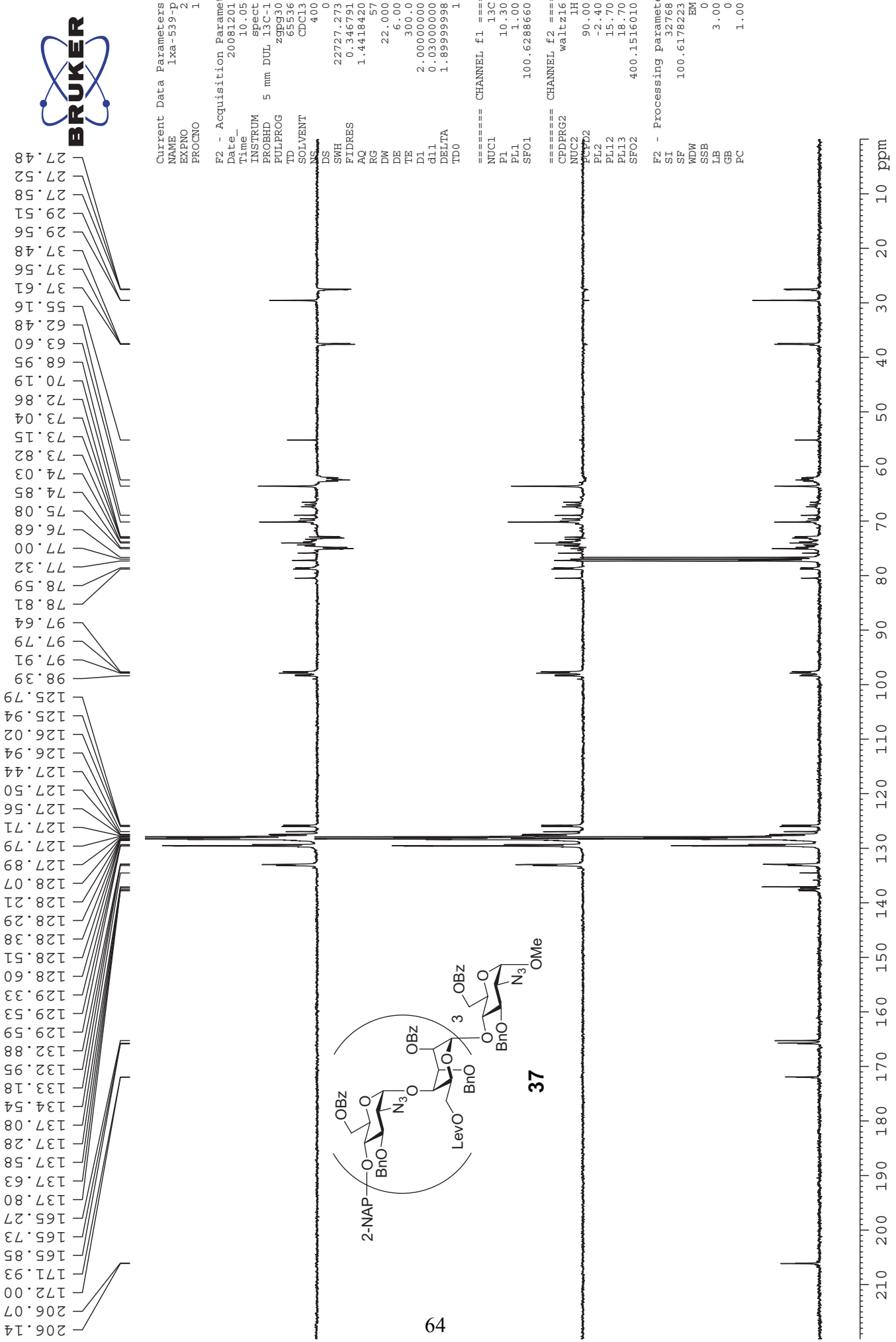

Current Data Parameters
NAME 1xa-525-p
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20020812
Time 14.53
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 22
DS 2
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 128
DW 78.000 usec
DE 111.43 usec
TE 300.0 K
HL1 3 dB
D1 1.00000000 sec
P1 6.00 usec
SFO1 400.1319596 MHz
NUCLEUS 1H
F2 - Processing parameters
SI 32768
SF 400.1300175 MHz
WDW EM
SSB 0
LB 0.01 Hz
GB 0
PC 1.00
35
2-NAP
OBz
BnO
LevO
OMe
ppm

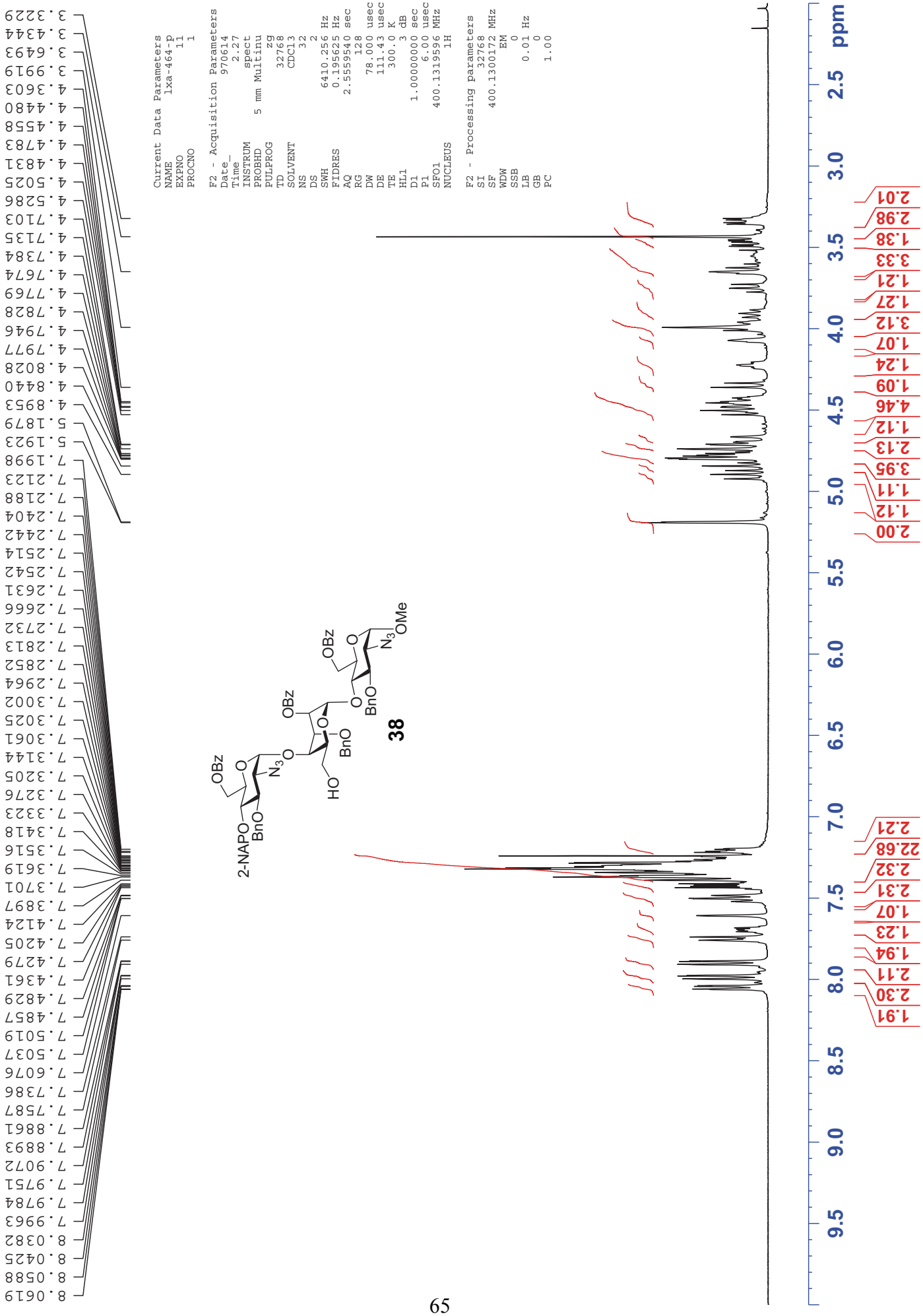


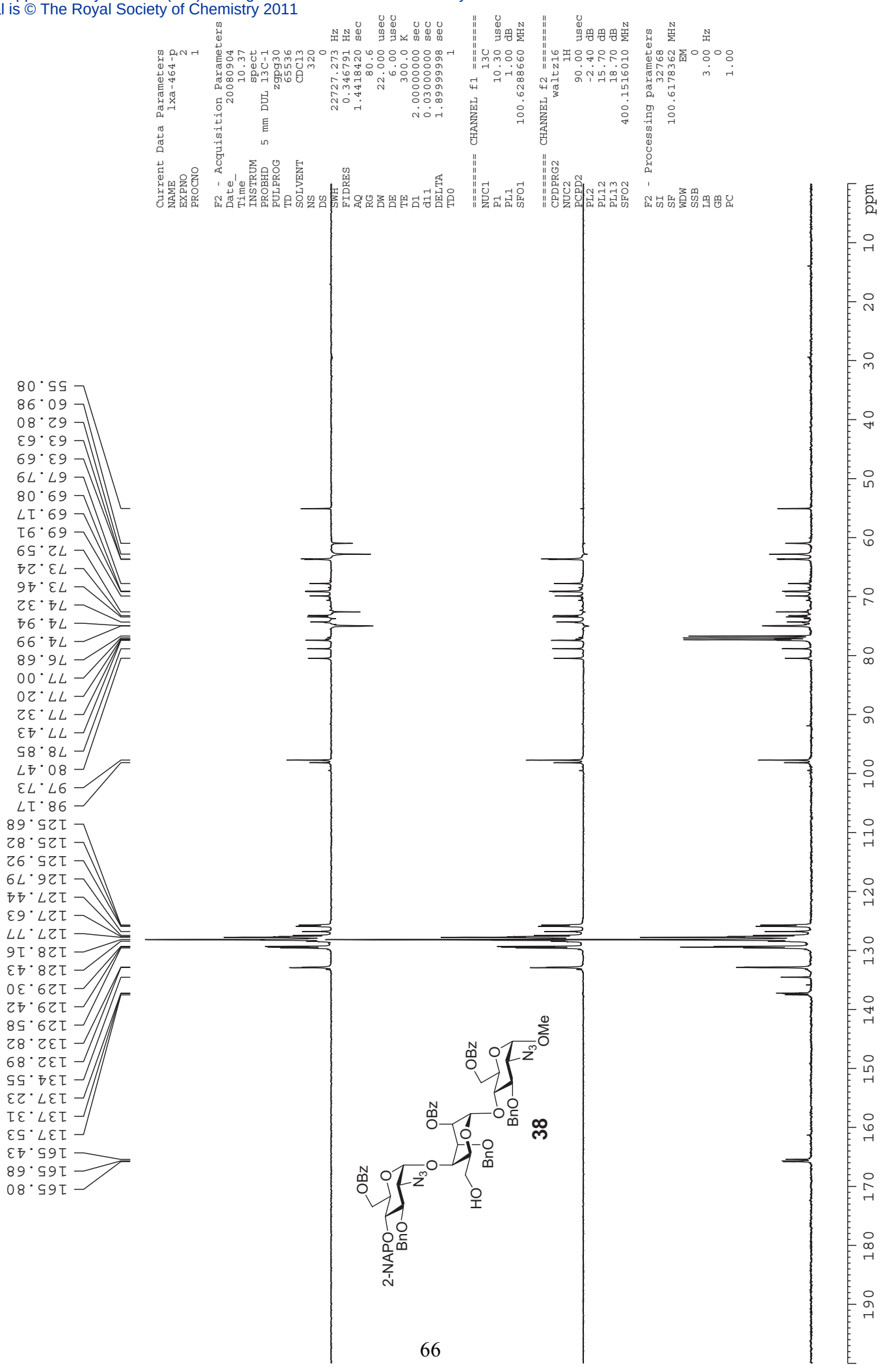


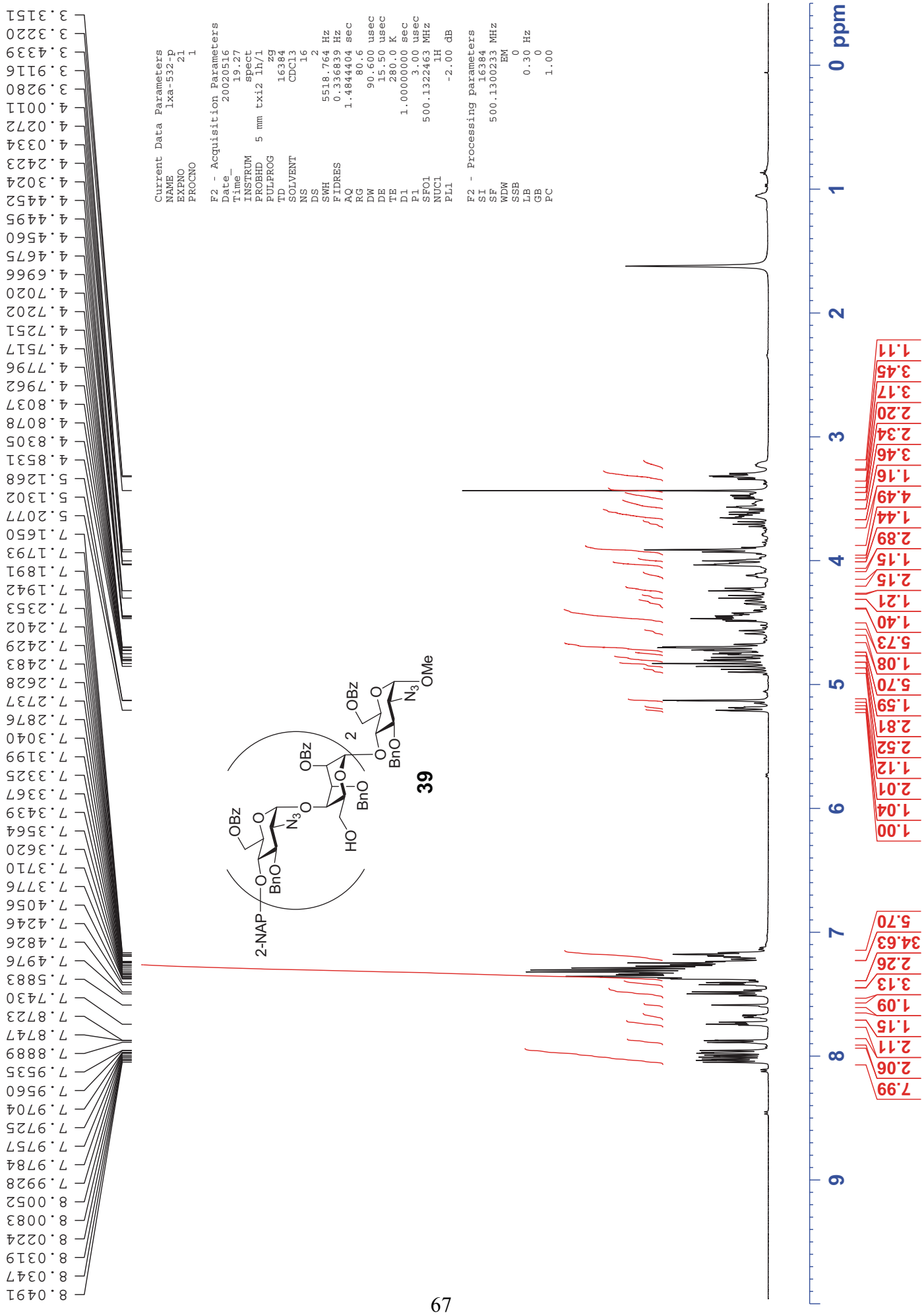


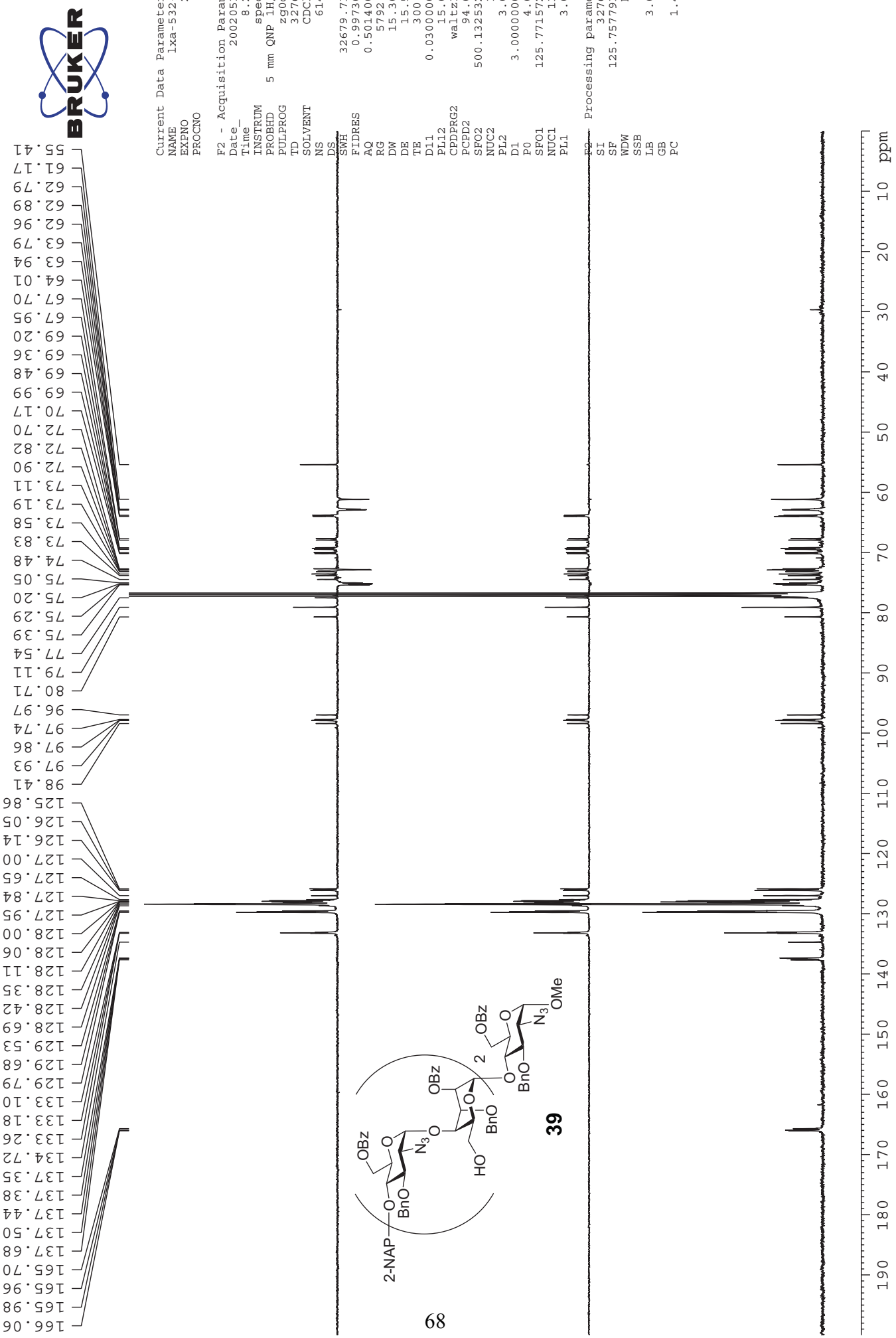

Current Data Parameters
NAME lxa-539-p
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20021114
Time 21.06
INSTRUM spect
PROBHD 5 mm txi2 1h/1
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 8
DS 2
SWH 5518.764 Hz
FIDRES 0.336839 Hz
AQ 1.4844404 sec
RG 32
DW 90.600 usec
DE 15.50 usec
TE 280.0 K
D1 1.00000000 sec
P1 4.00 usec
SFO1 500.1322463 MHz
NUC1 1H
PL1 -2.00 dB
F2 - Processing parameters
SI 16384
SF 500.1300236 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
2-NAP
OBz
BnO
N3
LevO
BnO
OBz
3
BnO
N3
OMe
37
ppm

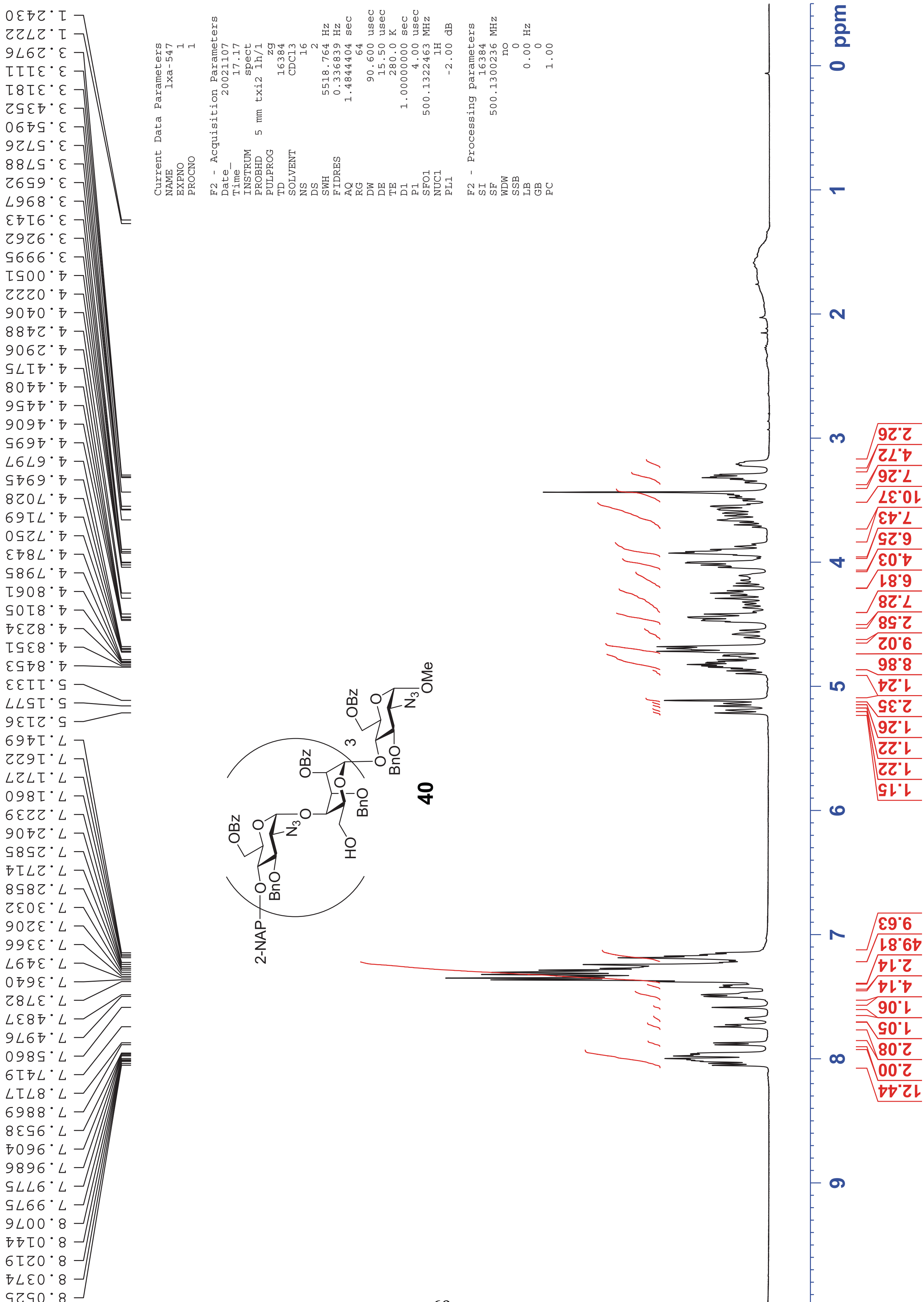


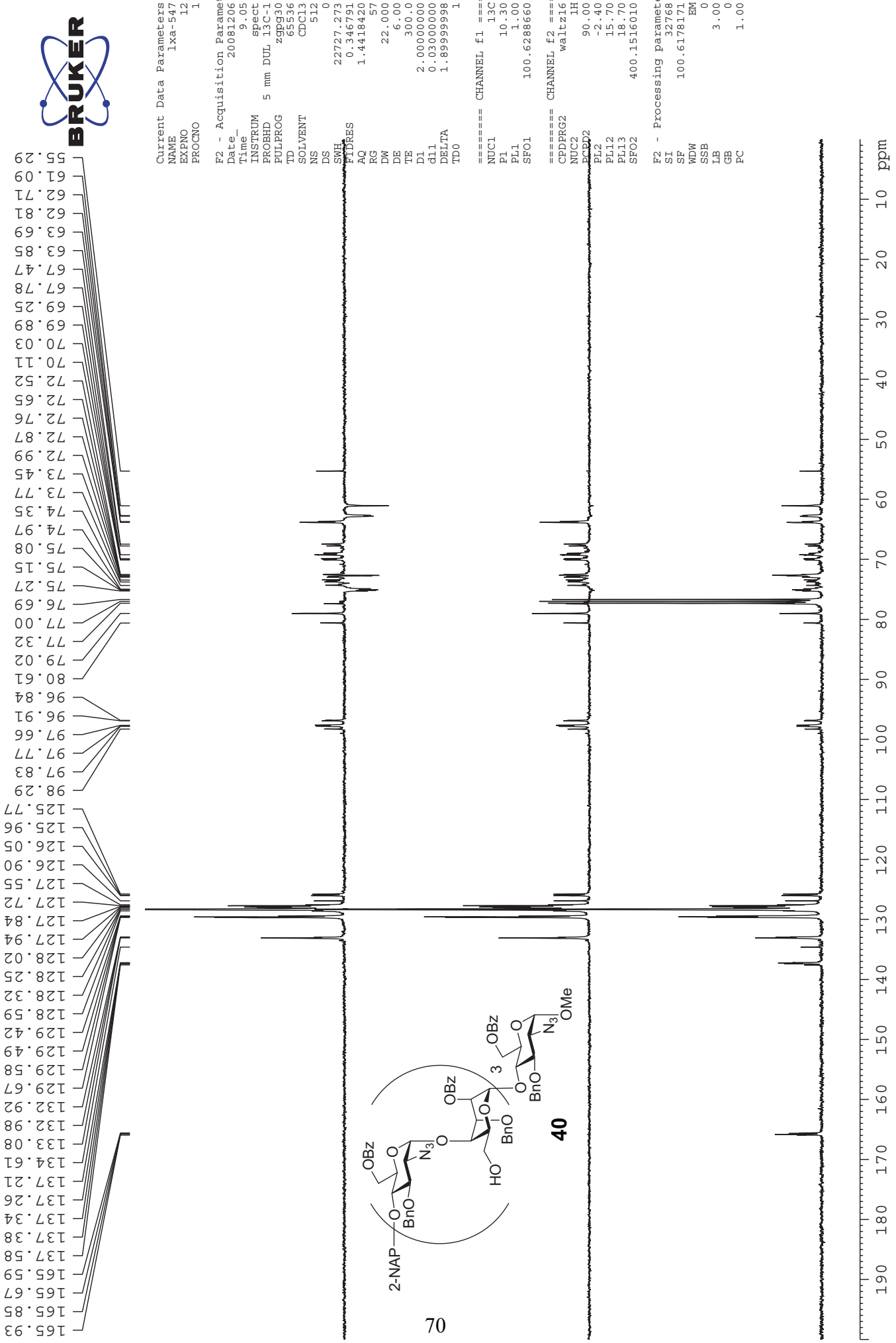

Current Data Parameters
NAME lxa-464-p
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 970614
Time 2.27
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 128
DW 78.000 usec
DE 111.43 usec
TE 300.0 K
HL1 3 dB
D1 1.00000000 sec
P1 6.00 usec
SFO1 400.1319596 MHz
NUCLEUS 1H
F2 - Processing parameters
SI 32768
SF 400.1300172 MHz
WDW EM
SSB 0
LB 0.01 Hz
GB 0
PC 1.00
OBz
2-NAPO
BnO
N3
O
OBz
BnO
HO
OBz
BnO
N3
OMe
38
ppm
2.00
1.12
1.11
3.95
2.13
1.12
4.46
1.09
1.24
1.07
3.12
1.27
1.21
3.33
1.38
2.98
2.01
1.91
2.30
2.11
1.94
1.23
1.07
2.31
2.32
22.68
2.21

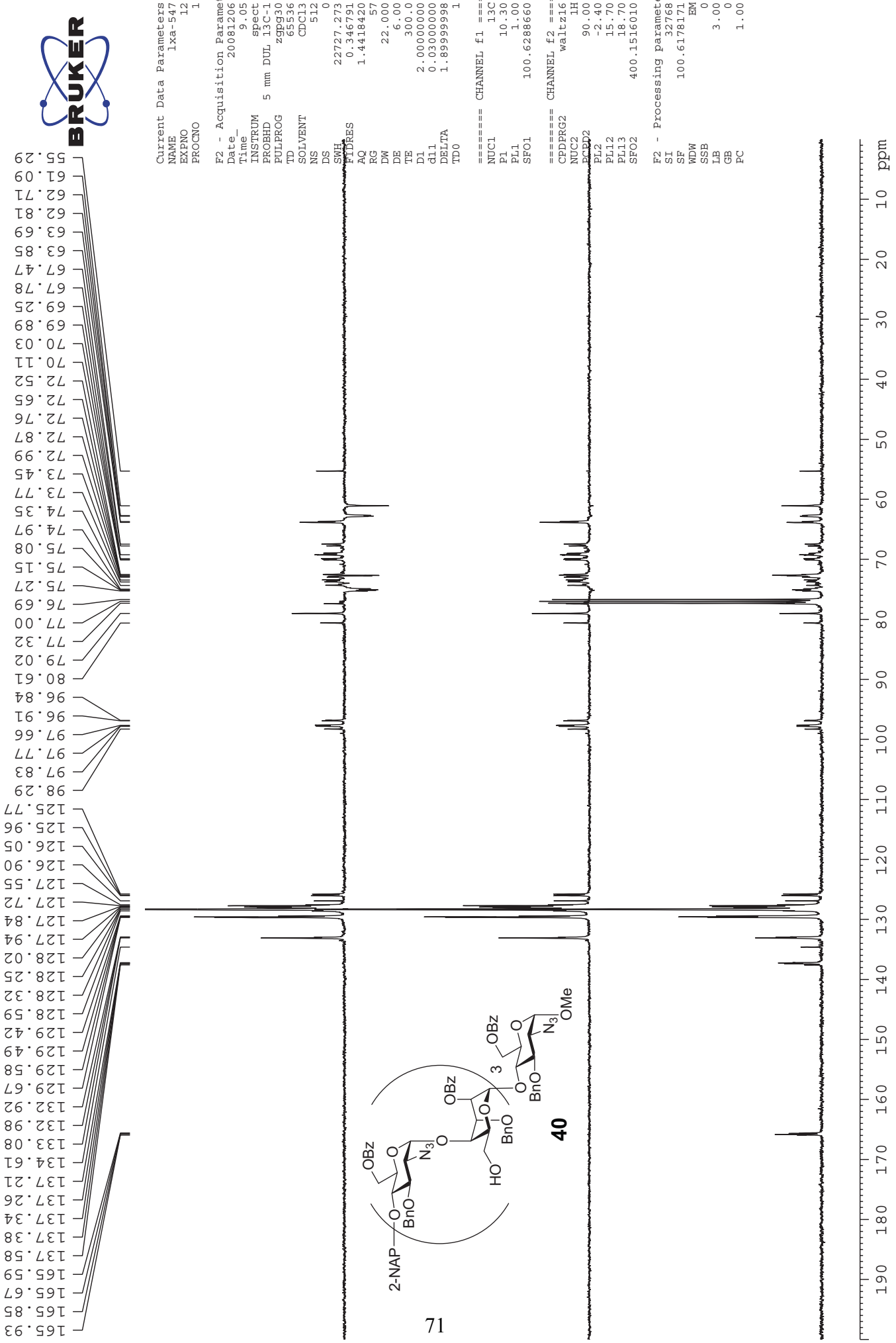


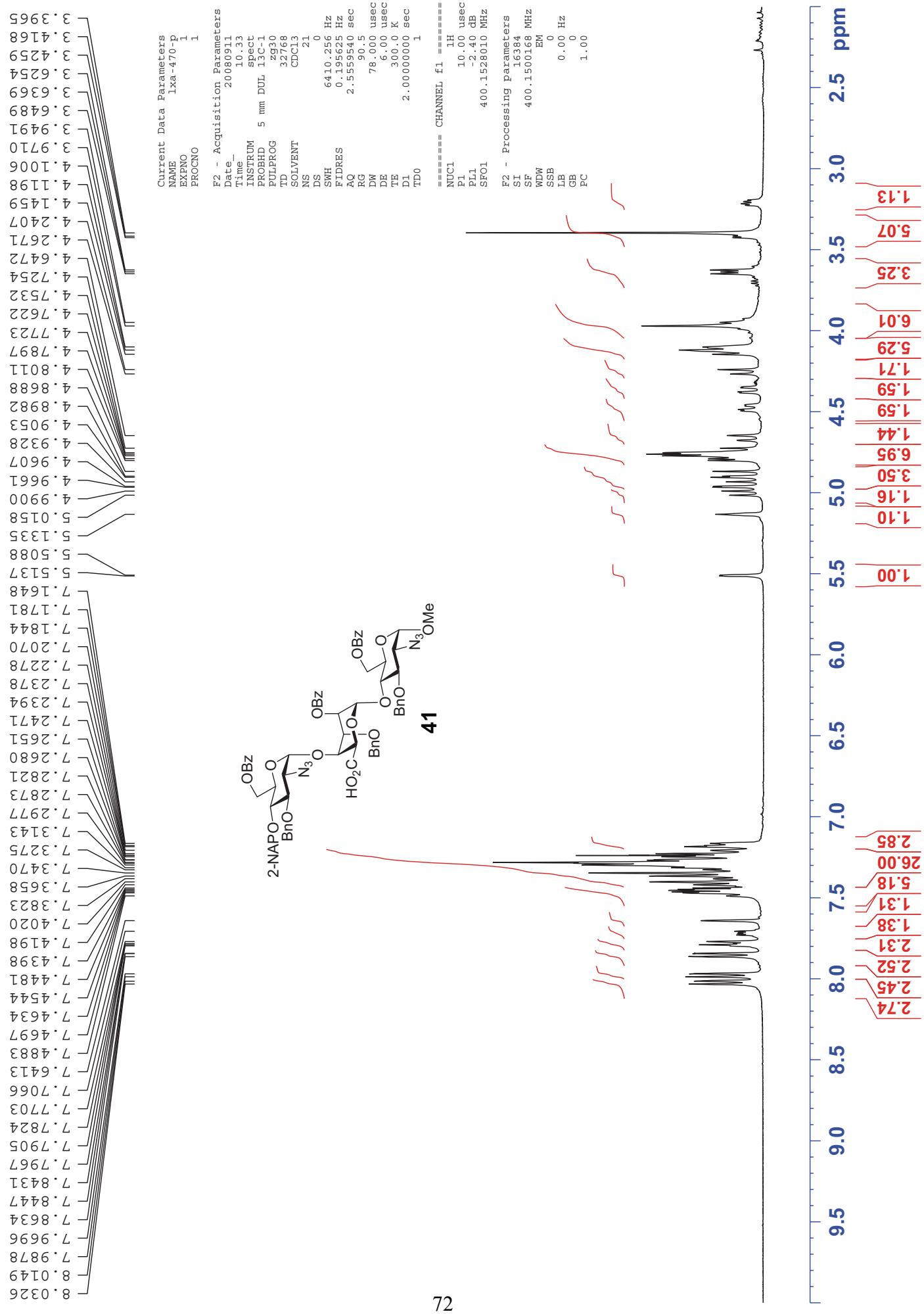

Current Data Parameters
NAME 1xa-532-p
EXPNO 21
PROCNO 1
F2 - Acquisition Parameters
Date_ 20020516
Time 19.27
INSTRUM spect
PROBHD 5 mm txi2 1h/1
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 16
DS 2
SWH 5518.764 Hz
FIDRES 0.336839 Hz
AQ 1.4844404 sec
RG 80.6
DW 90.600 usec
DE 15.50 usec
TE 280.0 K
D1 1.00000000 sec
P1 3.00 usec
SFO1 500.1322463 MHz
NUC1 1H
PL1 -2.00 dB
F2 - Processing parameters
SI 16384
SF 500.1300233 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
2-NAP
BnO
OBz
N3
HO
OMe
39
ppm
0
1
2
3
4
5
6
7
8
9
1.00
1.04
2.01
1.12
2.52
2.81
1.59
5.70
1.08
5.73
1.40
1.21
2.15
1.15
2.89
1.44
4.49
1.16
3.46
2.34
2.20
3.17
3.45
1.11
5.70
34.63
2.26
3.13
1.09
1.15
2.11
2.06
7.99




Current Data Parameters
NAME lxa-547
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20021107
Time 17.17
INSTRUM spect
PROBHD 5 mm txi2 1h/1
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 16
DS 2
SWH 5518.764 Hz
FIDRES 0.336839 Hz
AQ 1.4844404 sec
RG 64
DW 90.600 usec
DE 15.50 usec
TE 280.0 K
D1 1.00000000 sec
P1 4.00 usec
SFO1 500.1322463 MHz
NUC1 1H
PL1 -2.00 dB
F2 - Processing parameters
SI 16384
SF 500.1300236 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1.2430 1.2722 3.2976 3.3111 3.3181 3.4352 3.5490 3.5726 3.5788 3.6592 3.8967 3.9143 3.9262 3.9995 4.0051 4.0222 4.0406 4.2488 4.2906 4.4175 4.4408 4.4456 4.4606 4.4695 4.6797 4.6945 4.7028 4.7169 4.7250 4.7843 4.7985 4.8061 4.8105 4.8234 4.8351 4.8453 5.1133 5.1577 5.2136 7.1469 7.1622 7.1727 7.1860 7.2239 7.2406 7.2585 7.2714 7.2858 7.3032 7.3206 7.3366 7.3497 7.3640 7.3782 7.4837 7.4976 7.5860 7.7419 7.8717 7.8869 7.9538 7.9604 7.9686 7.9775 7.9965 8.0076 8.0144 8.0219 8.0374 8.0525
2-NAP
BnO
OBz
N3
O
HO
BnO
OBz
3
OBz
BnO
OMe
40
2.26 4.72 7.26 10.37 7.43 6.25 4.03 6.81 7.28 2.58 9.02 8.86 1.24 2.35 1.26 1.22 1.22 1.15 9.63 49.81 2.14 4.14 1.06 1.05 2.08 2.00 12.44
0 1 2 3 4 5 6 7 8 9 ppm




BRUKER
Current Data Parameters
NAME 1xa-547
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081206
Time_ 9.05
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 57
DW 22.000 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 10.30 usec
PL1 1.00 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.70 dB
PL13 18.70 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178171 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
ppm
2-NAP
OBz
BnO
N3
HO
3
OMe
40


Current Data Parameters
NAME 1xa-470-p
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080911
Time 10.33
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 21
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 90.5
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500168 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
41
ppm
1.13
5.07
3.25
6.01
5.29
1.71
1.59
1.59
1.44
6.95
3.50
1.16
1.10
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2.85
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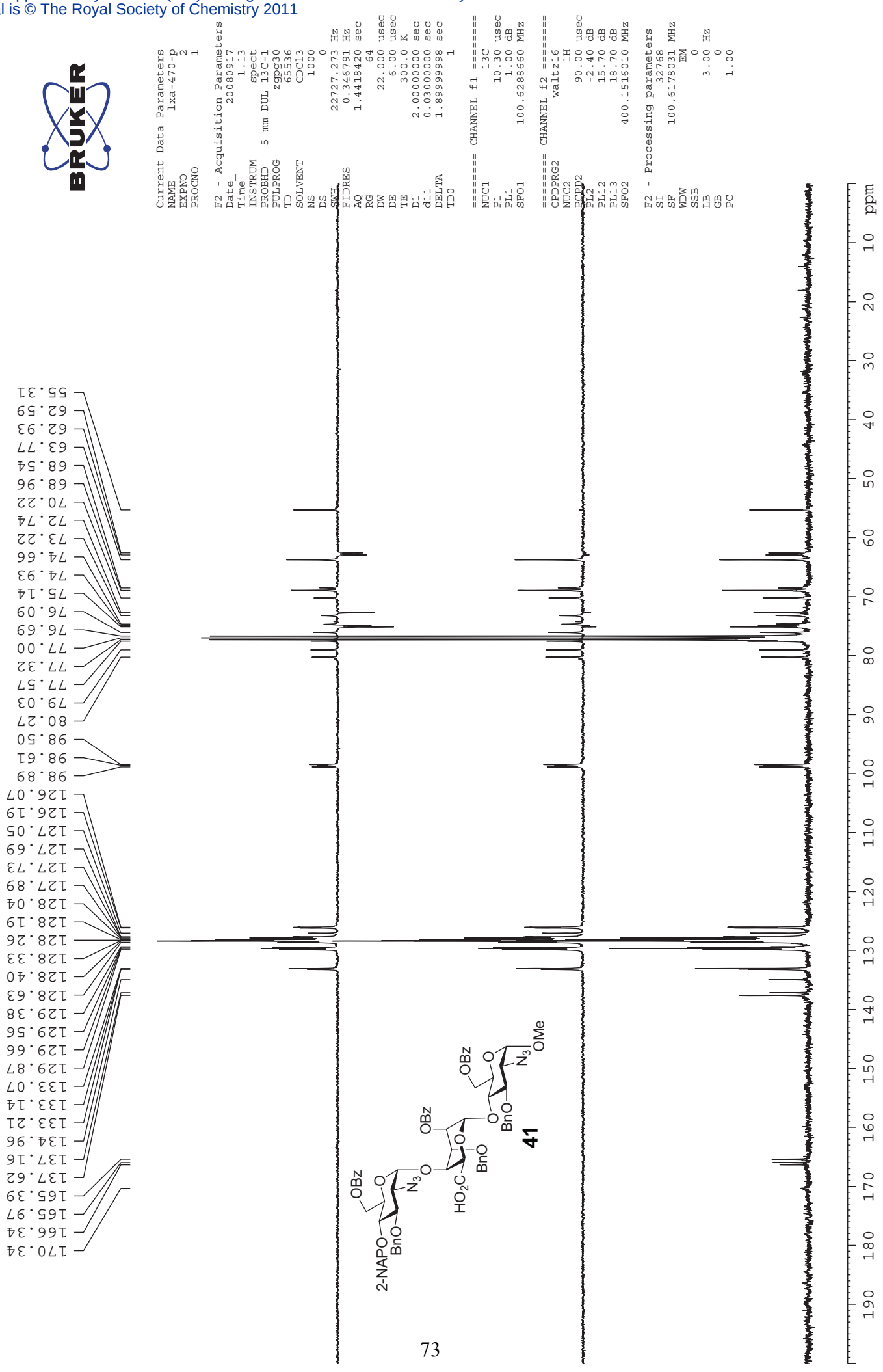
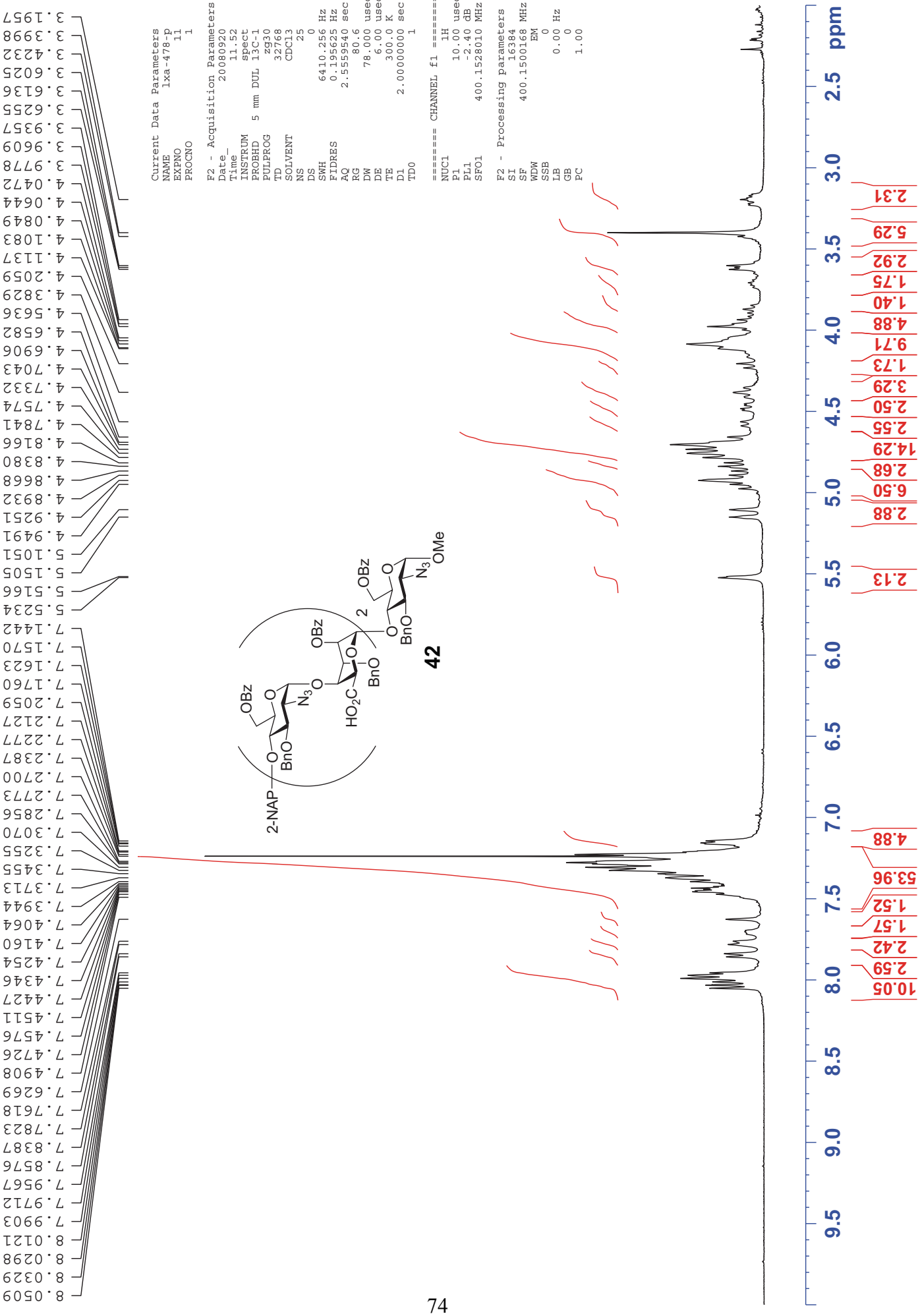
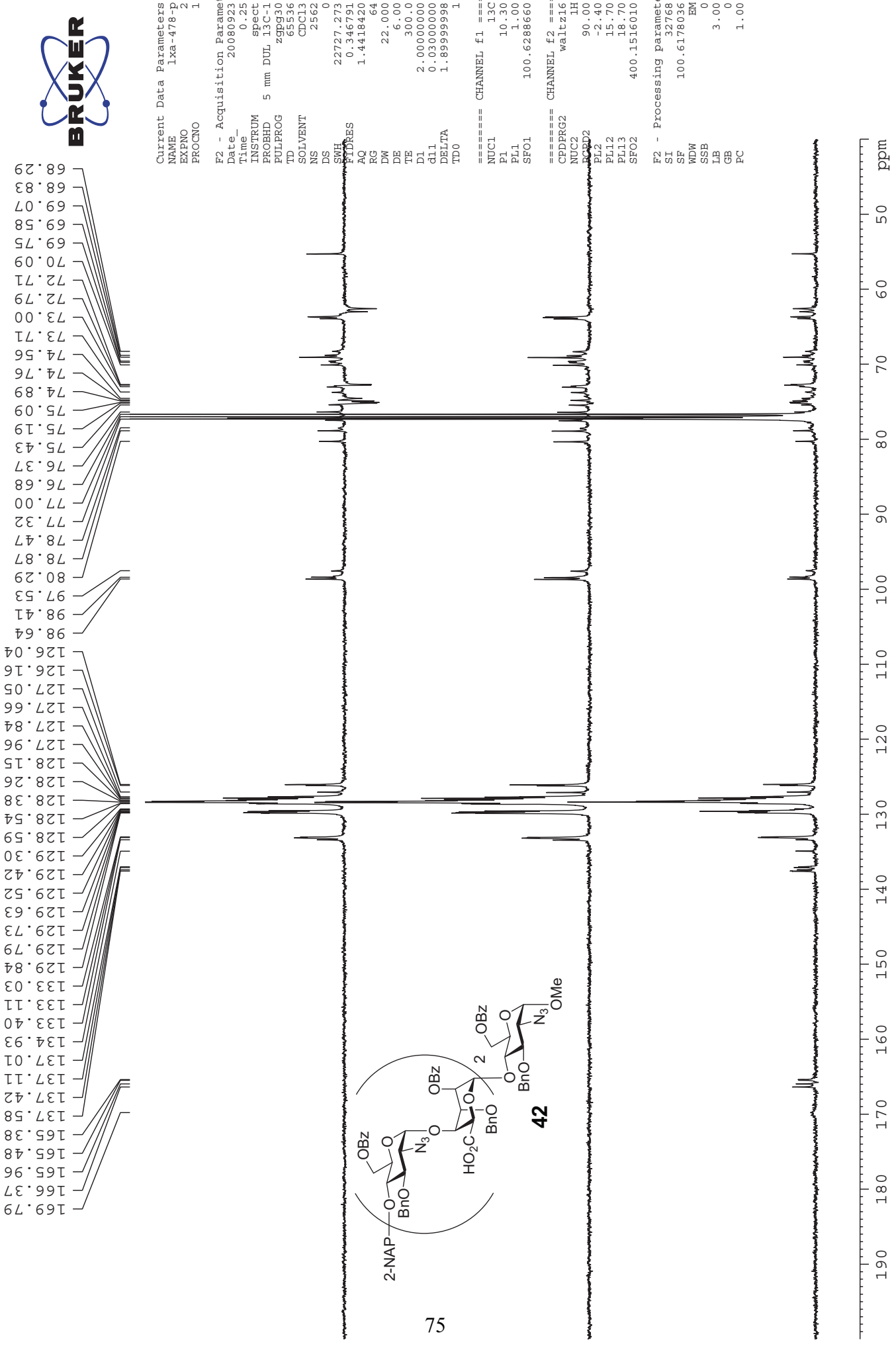
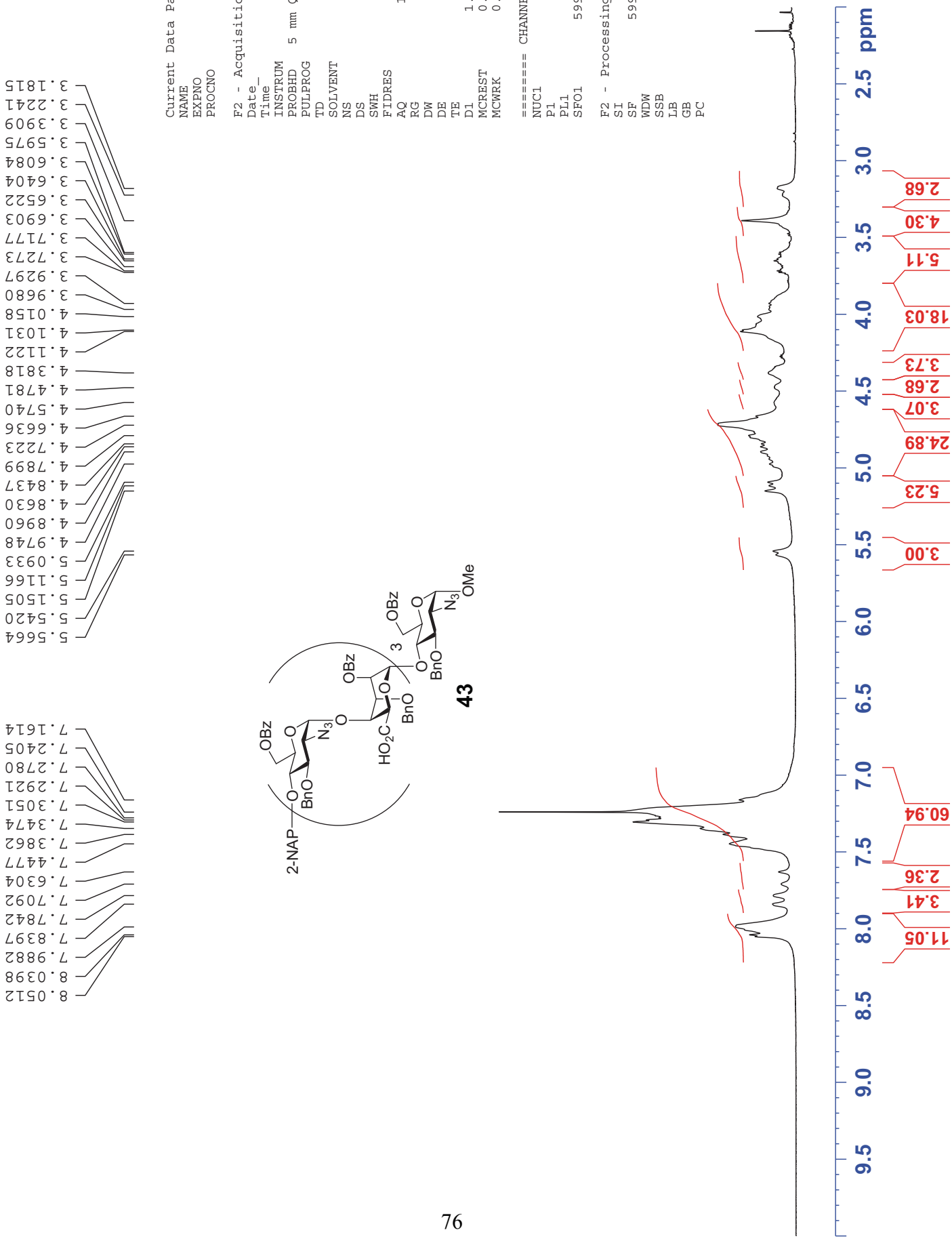
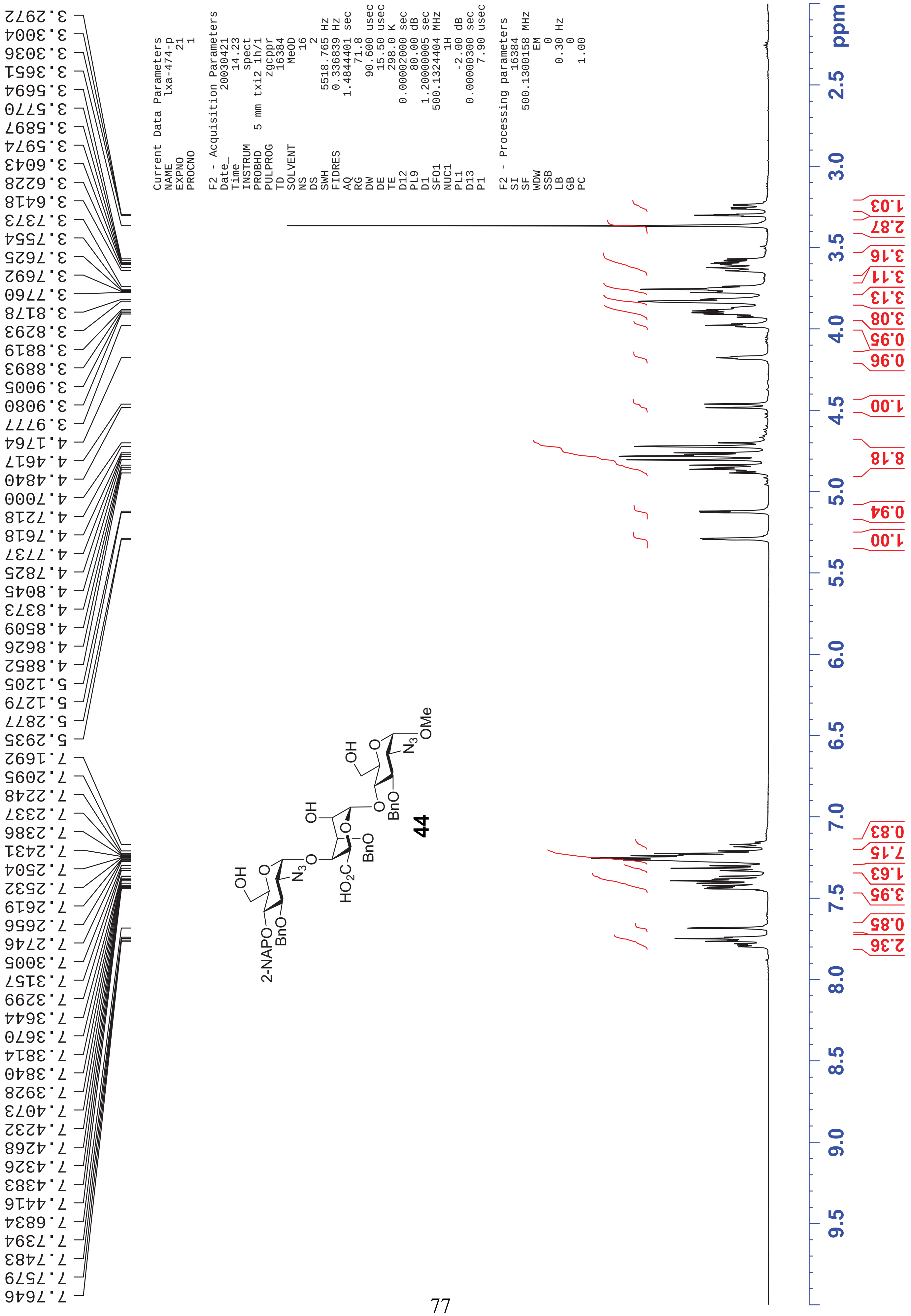
5.18
1.31
1.38
2.31
2.52
2.45
2.74

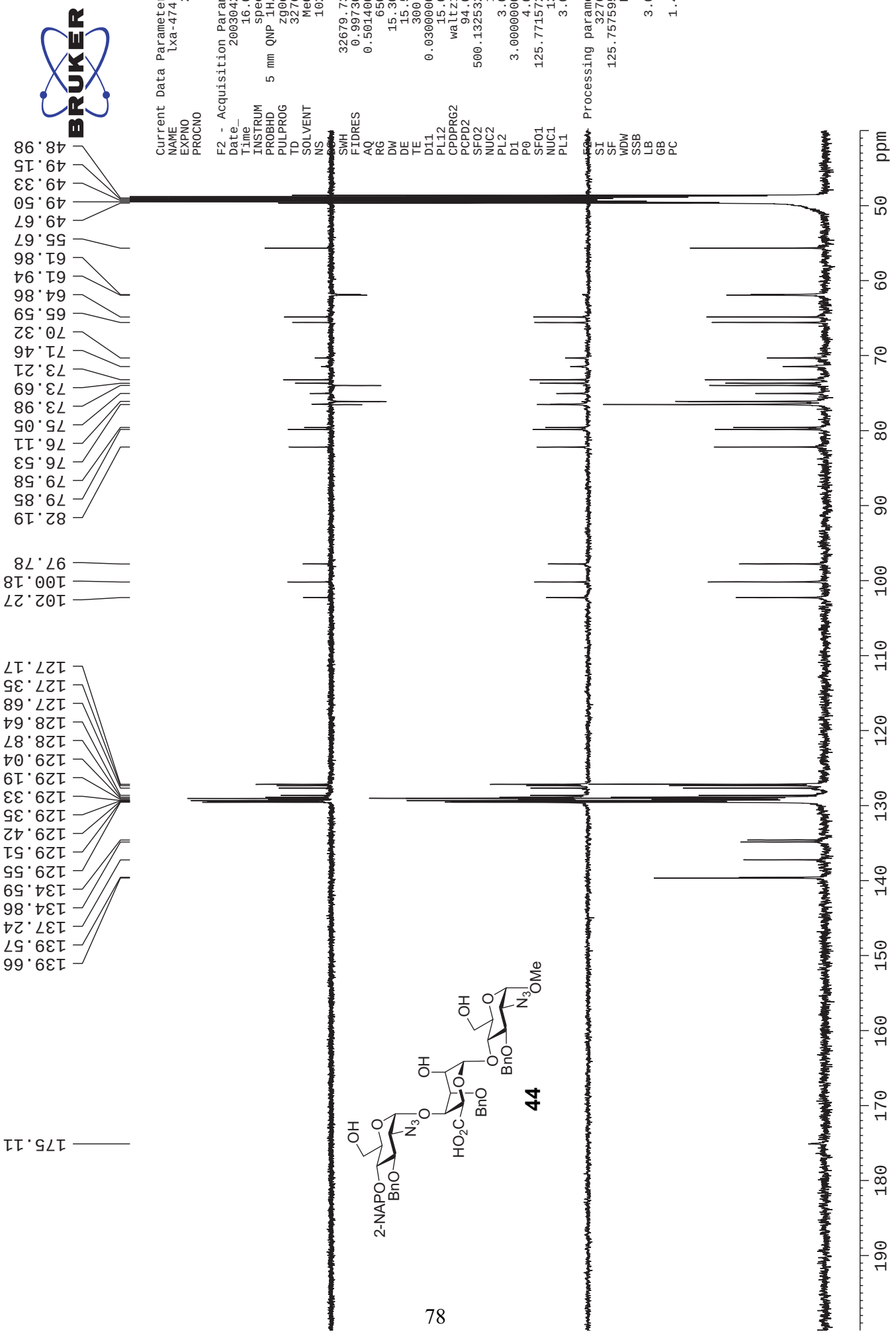


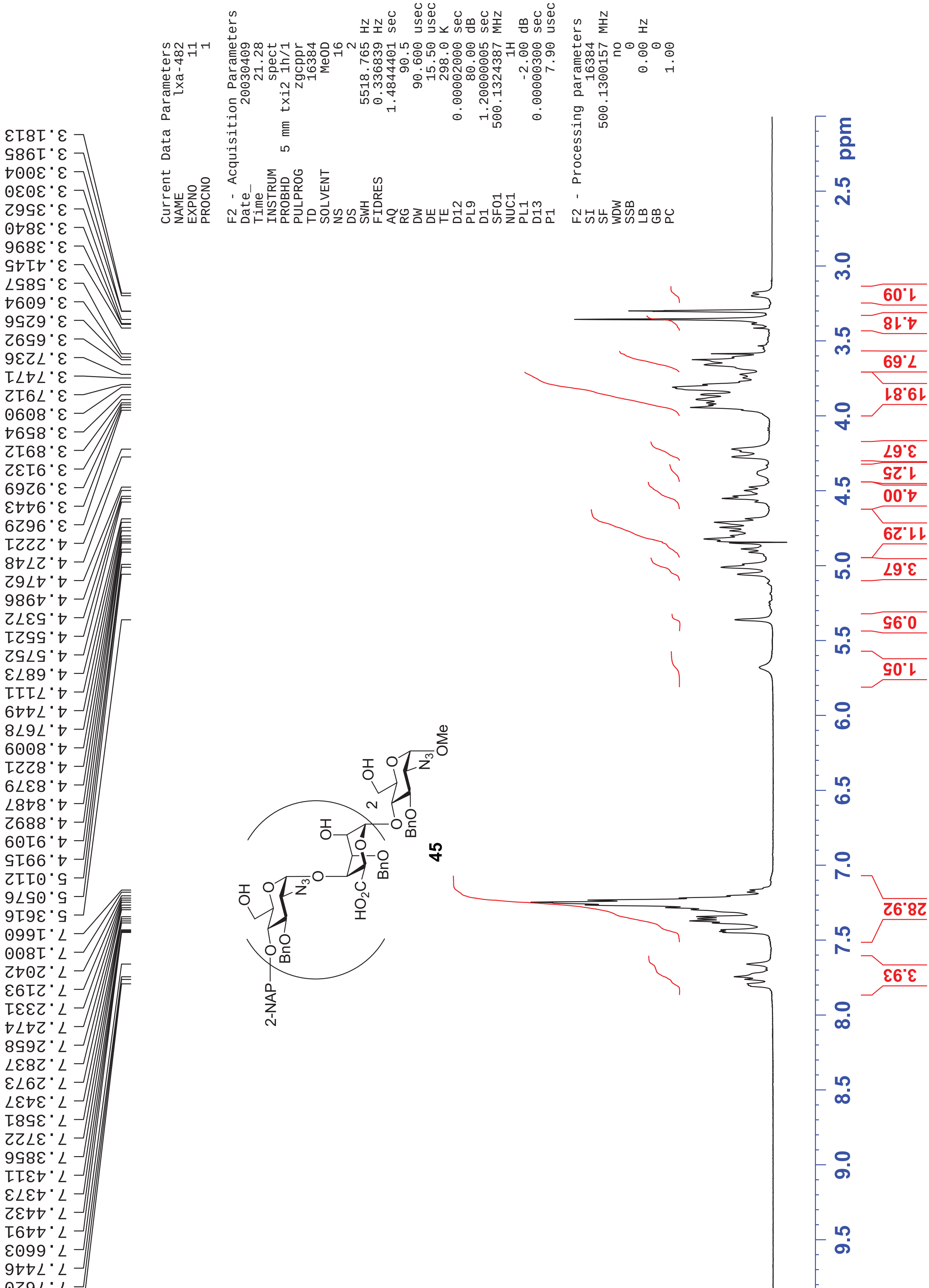

Current Data Parameters
NAME 1xa-478-p
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080920
Time 11.52
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 25
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 80.6
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500168 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
2-NAP
BnO
OBz
N3
HO2C
2
OMe
42
ppm
2.31
5.29
2.92
1.75
1.40
4.88
9.71
1.73
3.29
2.50
2.55
14.29
2.68
6.50
2.88
2.13
4.88
53.96
1.52
1.57
2.42
2.59
10.05

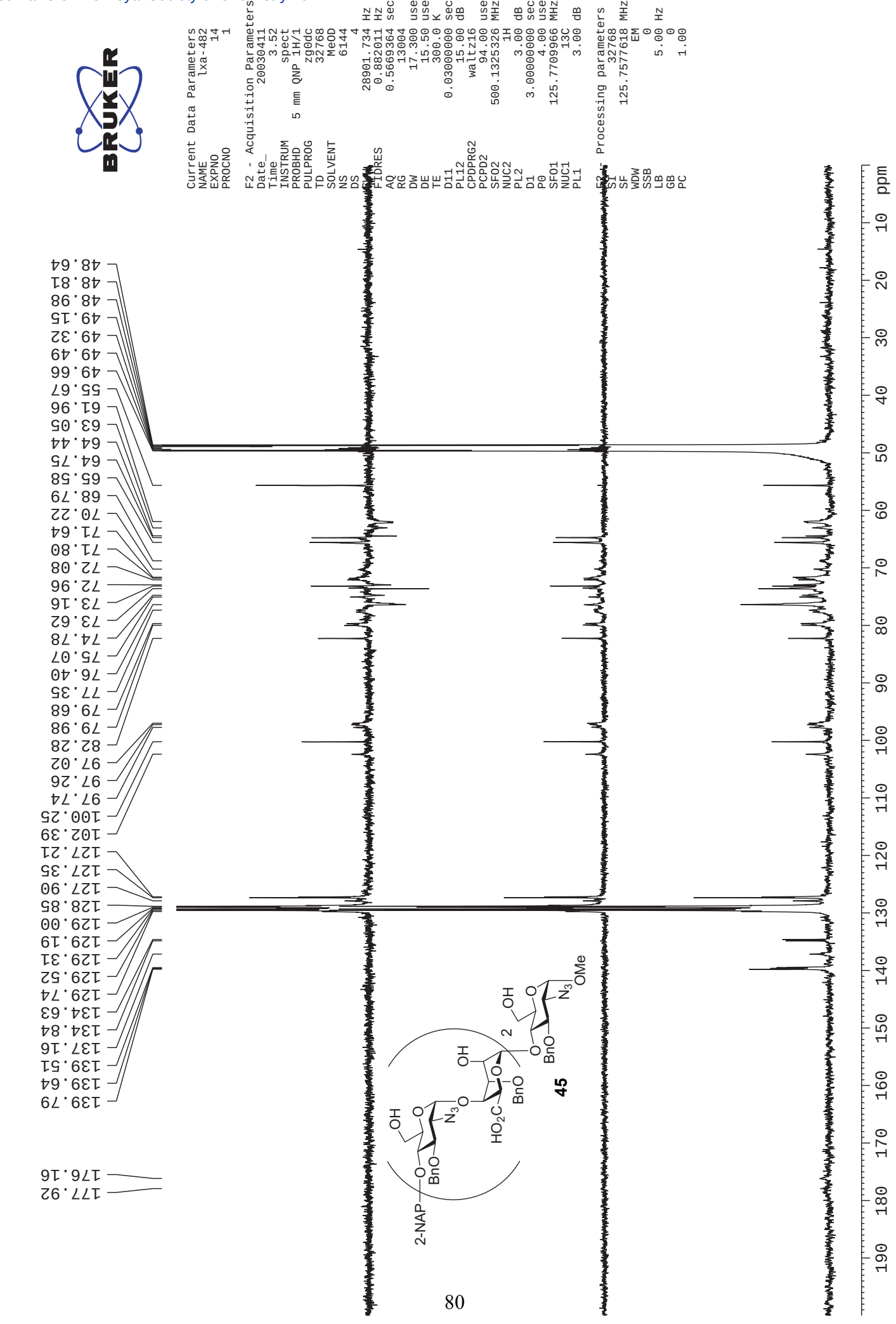


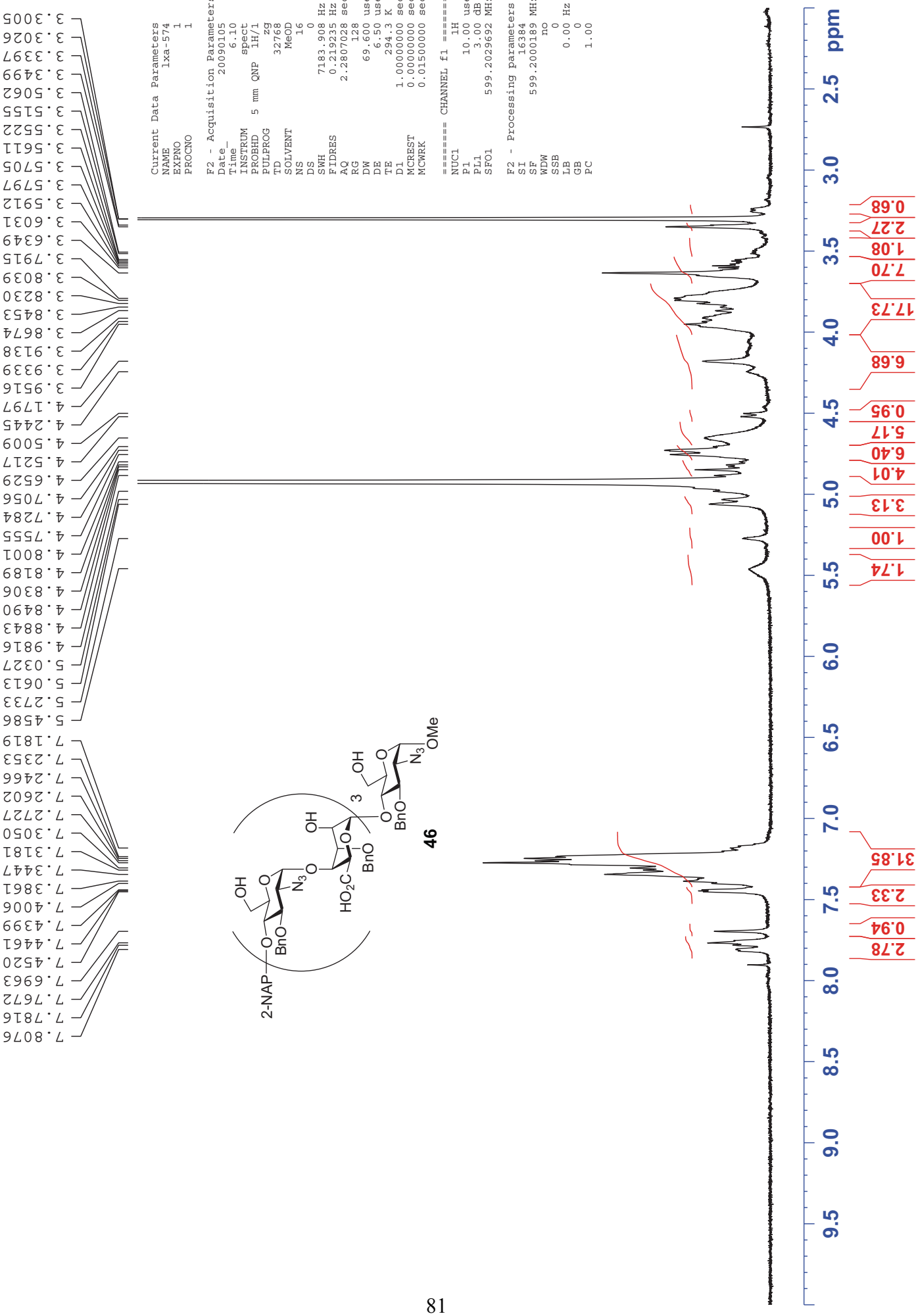

Current Data Parameters
NAME lxa-572
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090103
Time_ 7.20
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 9615.385 Hz
FIDRES 0.293438 Hz
AQ 1.7039860 sec
RG 128
DW 52.000 usec
DE 6.50 usec
TE 292.6 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 3.00 dB
SFO1 599.2035952 MHz
F2 - Processing parameters
SI 16384
SF 599.2000246 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
2-NAP
OBz
BnO
N_3
HO_2C
OMe
43
ppm
2.68
4.30
5.11
18.03
3.73
2.68
3.07
24.89
5.23
3.00
60.94
2.36
3.41
11.05

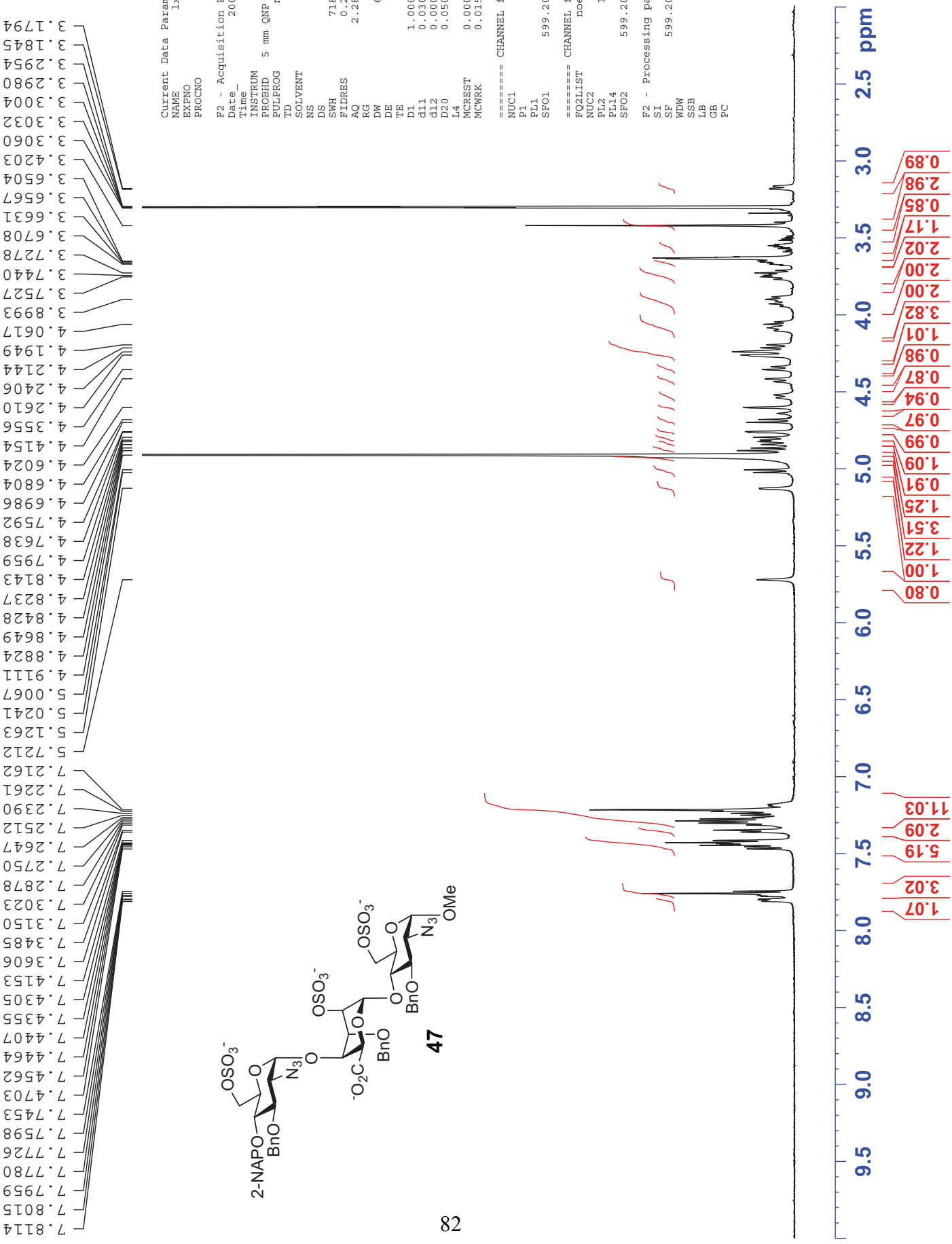

Current Data Parameters
NAME lxa-474-p
EXPNO 21
PROCNO 1
F2 - Acquisition Parameters
Date_ 20030421
Time 14.23
INSTRUM spect
PROBHD 5 mm txi2 1h/1
PULPROG zgcppr
TD 16384
SOLVENT MeOD
NS 16
DS 2
SWH 5518.765 Hz
FIDRES 0.336839 Hz
AQ 1.4844401 sec
RG 71.8
DW 90.600 usec
DE 15.50 usec
TE 298.0 K
D12 0.00002000 sec
PL9 80.00 dB
D1 1.20000005 sec
SFO1 500.1324404 MHz
NUC1 1H
PL1 -2.00 dB
D13 0.00000300 sec
P1 7.90 usec
F2 - Processing parameters
SI 16384
SF 500.1300158 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
44
ppm

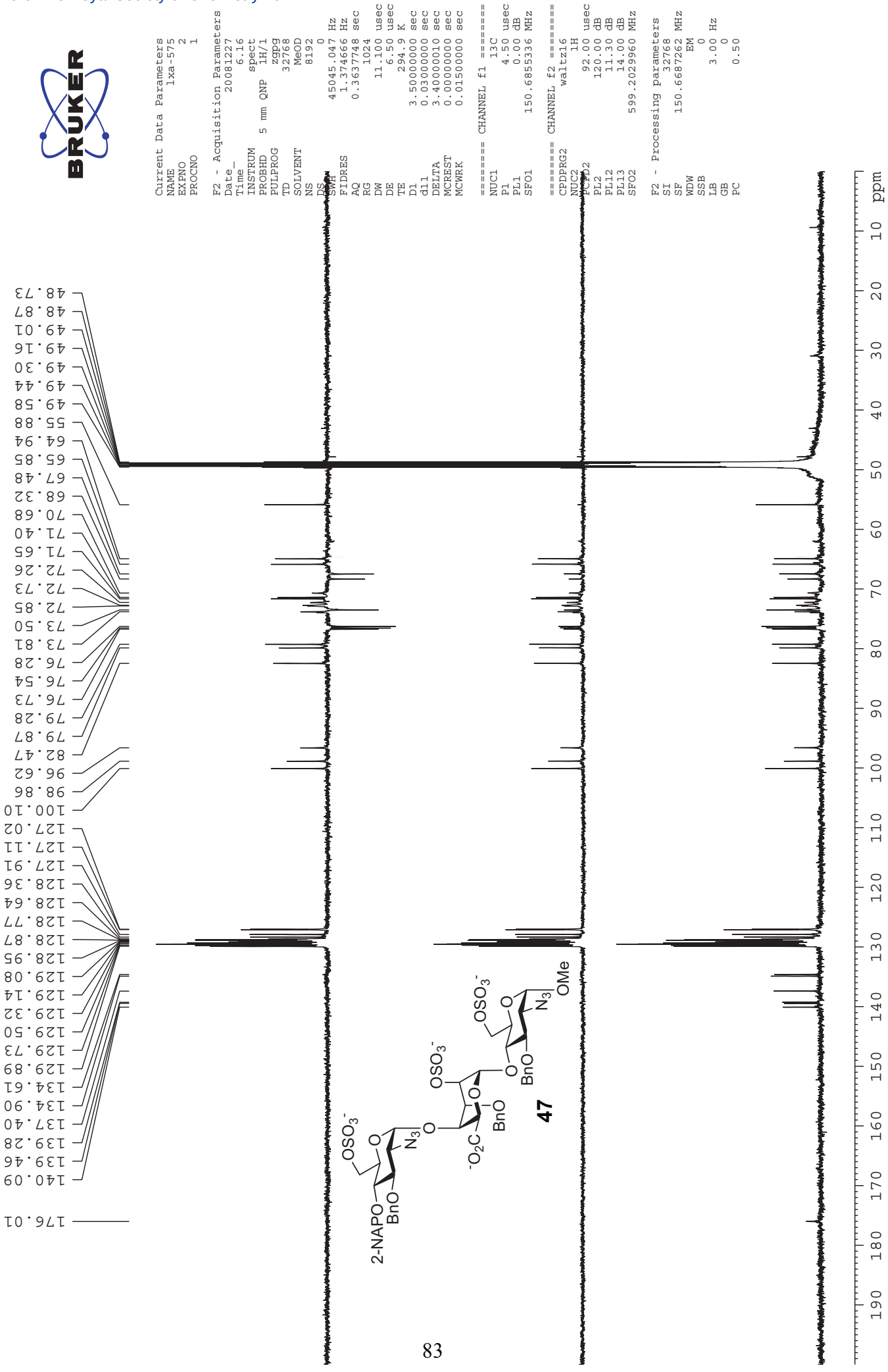


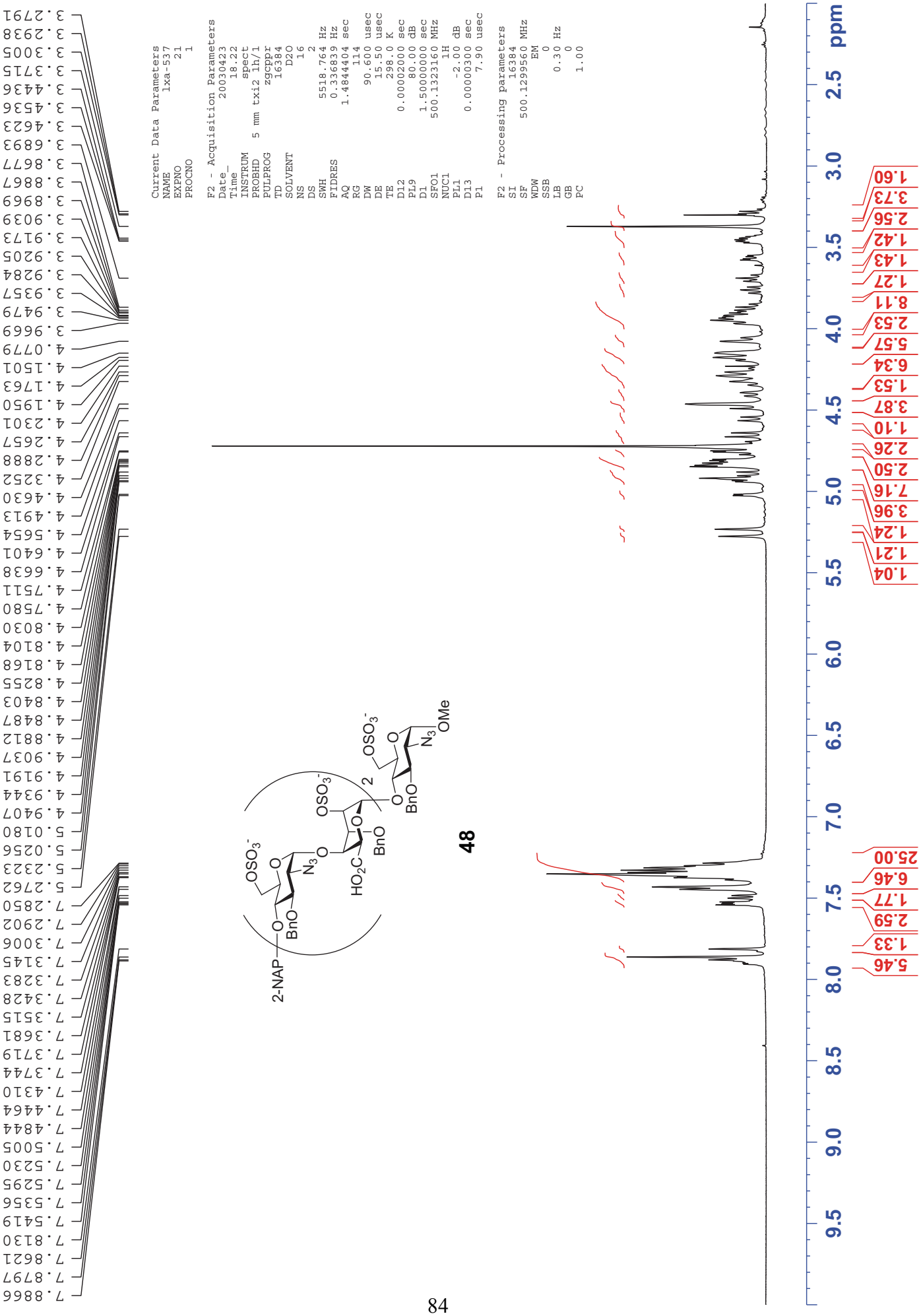

Current Data Parameters
NAME lxa-482
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20030409
Time 21.28
INSTRUM spect
PROBHD 5 mm txi2 1h/1
PULPROG zgcppr
TD 16384
SOLVENT MeOD
NS 16
DS 2
SWH 5518.765 Hz
FIDRES 0.336839 Hz
AQ 1.4844401 sec
RG 90.5
DW 90.600 usec
DE 15.50 usec
TE 298.0 K
D12 0.00002000 sec
PL9 80.00 dB
D1 1.20000005 sec
SFO1 500.1324387 MHz
NUC1 1H
PL1 -2.00 dB
D13 0.00000300 sec
P1 7.90 usec
F2 - Processing parameters
SI 16384
SF 500.1300157 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
3.1813
3.1985
3.3004
3.3030
3.3562
3.3840
3.3896
3.4145
3.5857
3.6094
3.6256
3.6592
3.7236
3.7471
3.7912
3.8090
3.8594
3.8912
3.9132
3.9269
3.9443
3.9629
4.2221
4.2748
4.4762
4.4986
4.5372
4.5521
4.5752
4.6873
4.7111
4.7449
4.7678
4.8009
4.8221
4.8379
4.8487
4.8892
4.9109
4.9915
5.0112
5.0576
5.3616
7.1660
7.1800
7.2042
7.2193
7.2331
7.2474
7.2658
7.2837
7.2973
7.3437
7.3581
7.3722
7.3856
7.4311
7.4373
7.4432
7.4491
7.6603
7.7446
7.7620
7.7920
1.09
4.18
7.69
19.81
3.67
1.25
4.00
11.29
3.67
0.95
1.05
28.92
3.93
ppm
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
9.0
9.5
OH
O
N3
OMe
2
OH
O
BnO
O
HO2C
BnO
O
N3
O
OH
O
BnO
O
2-NAP
45

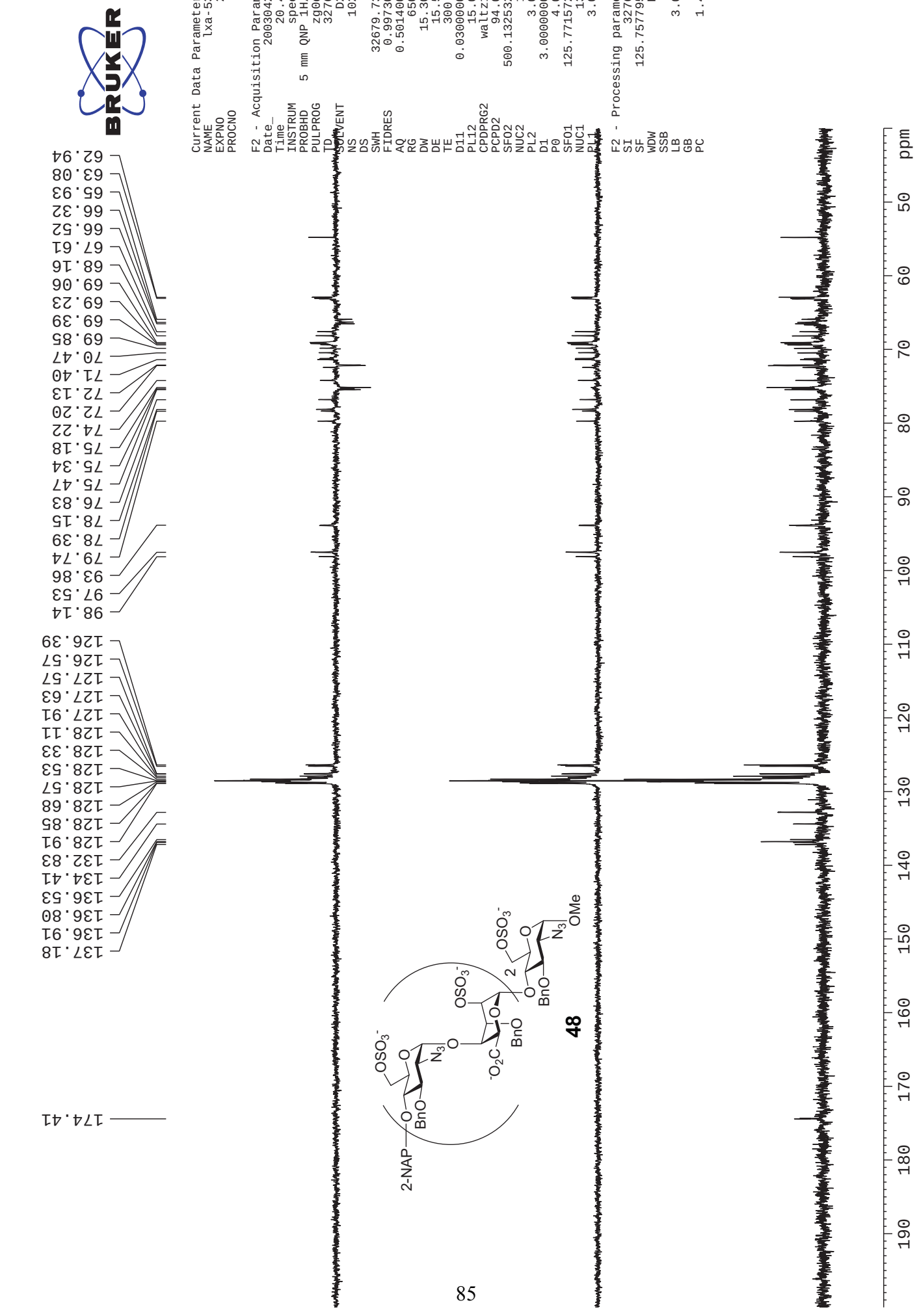


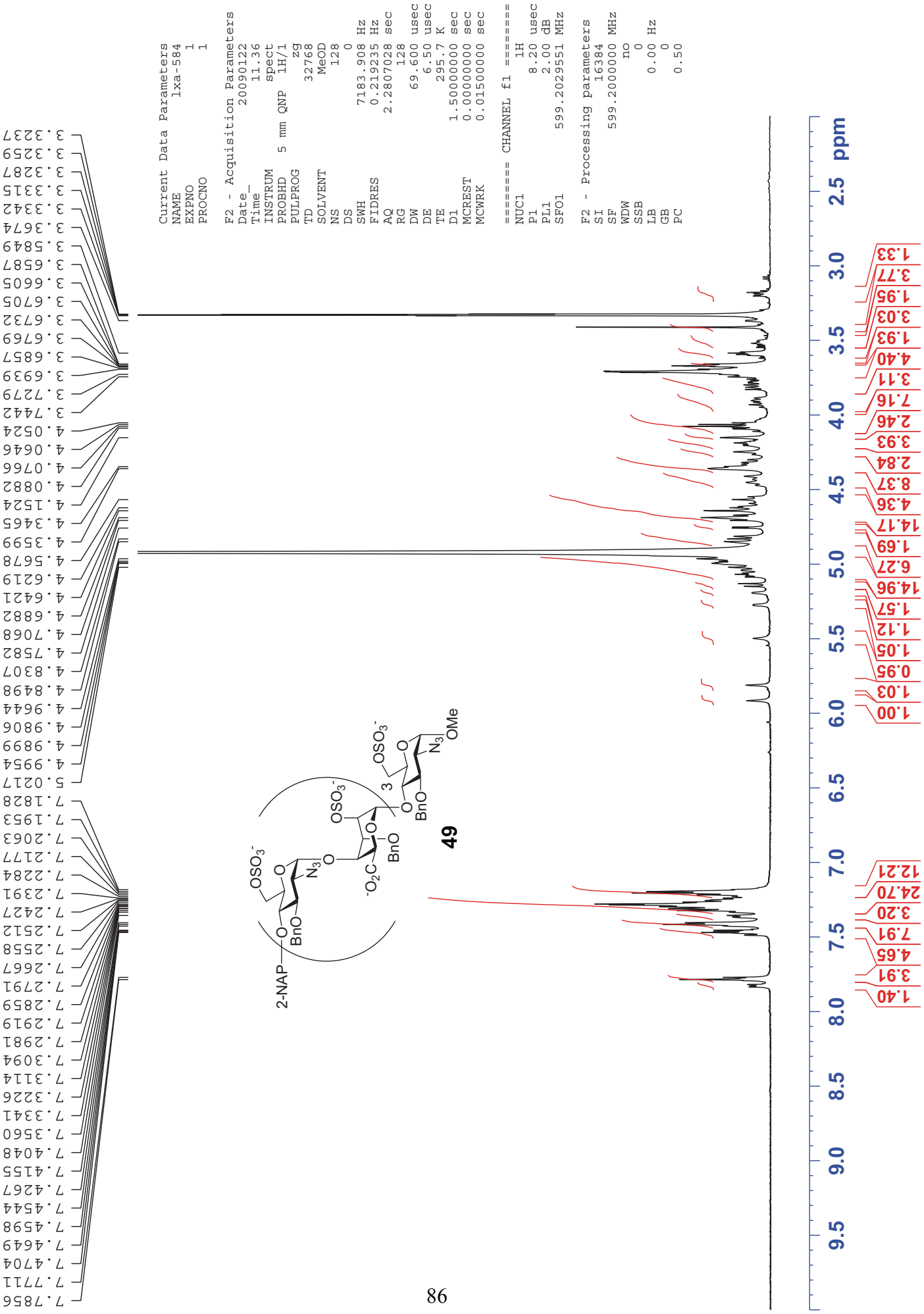

Current Data Parameters
NAME 1xa-574
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090105
Time 6.10
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 32768
SOLVENT MeOD
NS 16
DS 0
SWH 7183.908 Hz
FIDRES 0.219235 Hz
AQ 2.2807028 sec
RG 128
DW 69.600 usec
DE 6.50 usec
TE 294.3 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 3.00 dB
SFO1 599.2029692 MHz
F2 - Processing parameters
SI 16384
SF 599.2000189 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
ppm
0.68
2.27
1.08
7.70
17.73
6.68
0.95
5.17
6.40
4.01
3.13
1.00
1.74
31.85
2.33
0.94
2.78
2-NAP
BnO
N3
OH
HO2C
46
OMe

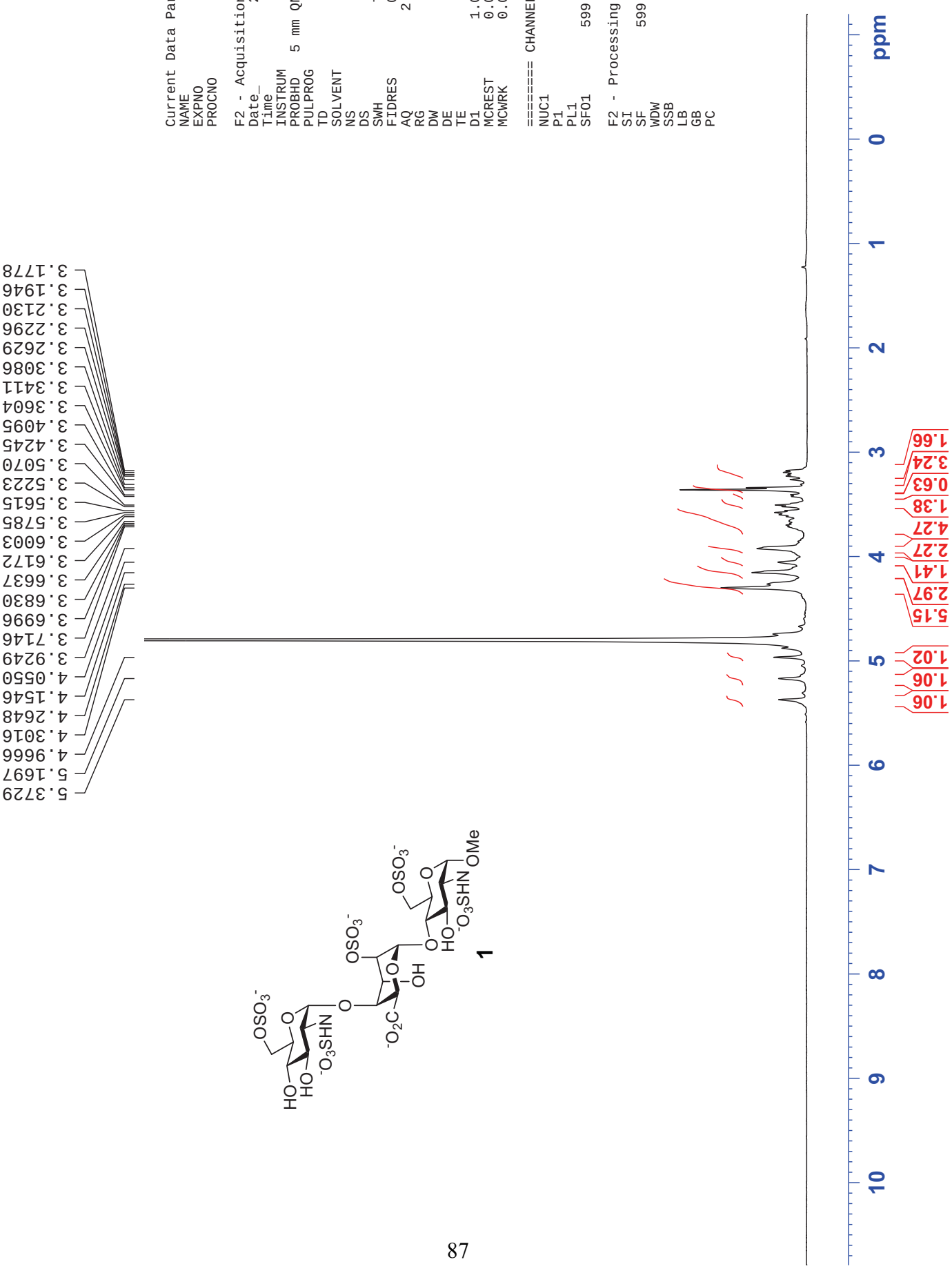

Current Data Parameters
NAME lxa-575
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081227
Time_ 6.13
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG noemul
TD 32768
SOLVENT MeOD
NS 32
DS 0
SWH 7183.908 Hz
FIDRES 0.219235 Hz
AQ 2.2807028 sec
RG 128
DW 69.600 usec
DE 6.50 usec
TE 294.9 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
D20 0.05000000 sec
L4 50
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 3.00 dB
SFO1 599.2028135 MHz
======== CHANNEL f2 ========
FQ2LIST noedif.1
NUC2 1H
PL2 120.00 dB
PL14 40.00 dB
SFO2 599.2032000 MHz
F2 - Processing parameters
SI 16384
SF 599.2000173 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
ppm
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
0.80
1.00
1.22
3.51
1.25
0.91
1.09
0.99
0.97
0.94
0.87
0.98
1.01
3.82
2.00
2.00
2.02
1.17
0.85
2.98
0.89
11.03
2.09
5.19
3.02
1.07
2-NAPO
BnO
N3
OSO3-
-O2C
BnO
OSO3-
BnO
N3
OSO3-
OMe
47

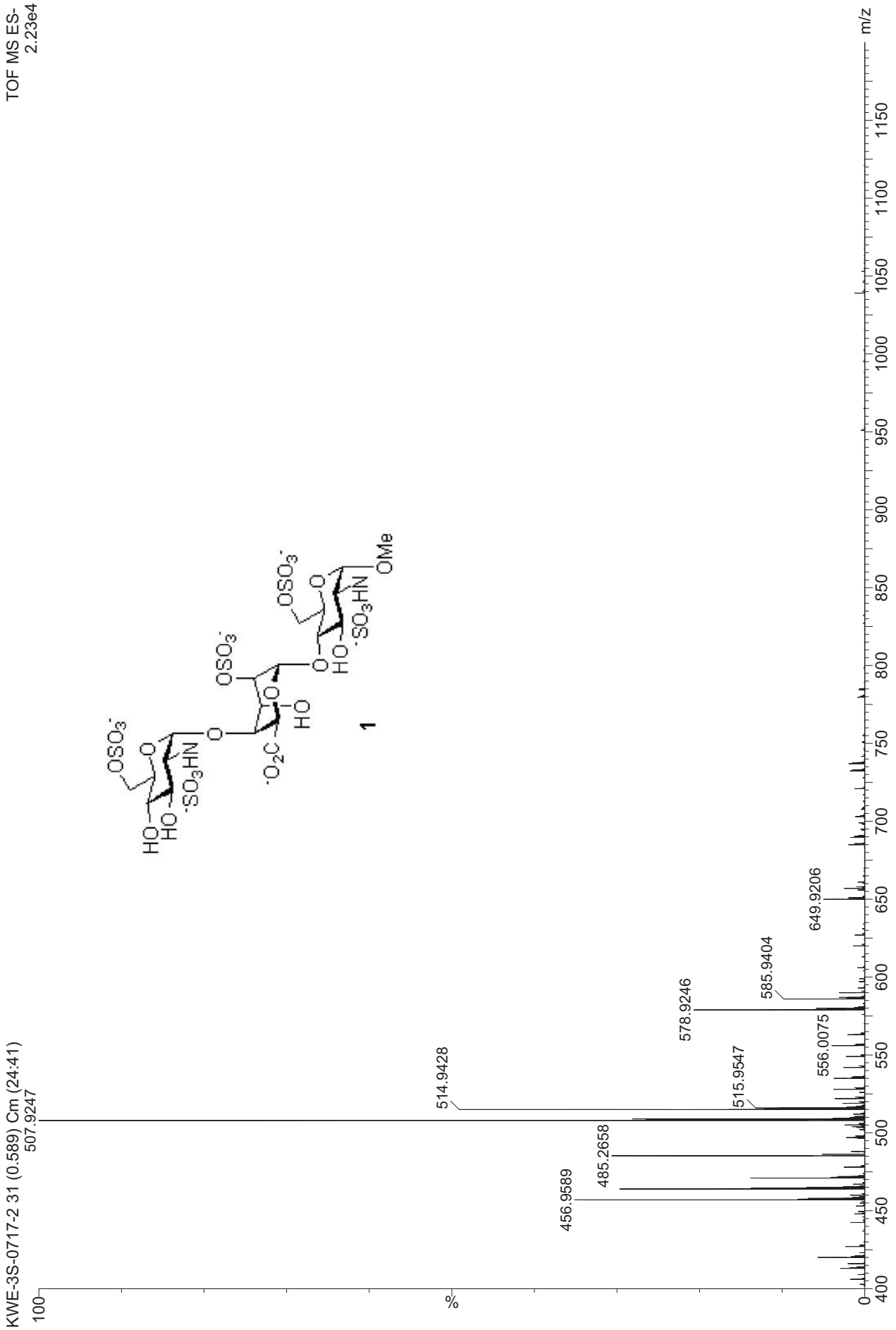


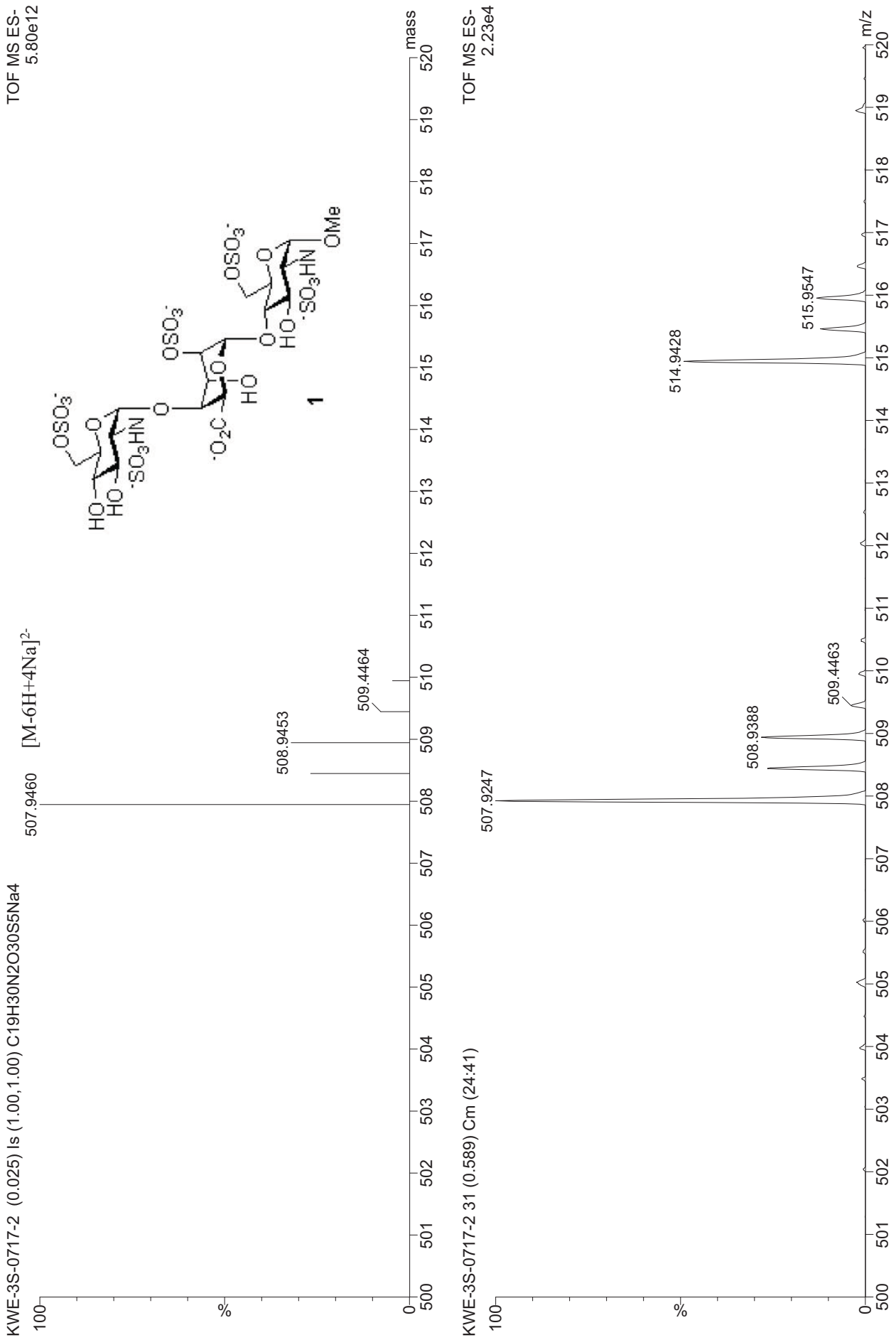

Current Data Parameters
NAME 1xa-537
EXPNO 21
PROCNO 1
F2 - Acquisition Parameters
Date_ 20030423
Time 18.22
INSTRUM spect
PROBHD 5 mm txi2 1h/1
PULPROG zgcppr
TD 16384
SOLVENT D2O
NS 16
DS 2
SWH 5518.764 Hz
FIDRES 0.336839 Hz
AQ 1.4844404 sec
RG 114
DW 90.600 usec
DE 15.50 usec
TE 298.0 K
D12 0.00002000 sec
PL9 80.00 dB
D1 1.50000000 sec
SFO1 500.1323160 MHz
NUC1 1H
PL1 -2.00 dB
D13 0.00000300 sec
P1 7.90 usec
F2 - Processing parameters
SI 16384
SF 500.1299560 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
2-NAP
BnO
OSO3-
N3
HO2C
OMe
48
ppm
1.04
1.21
1.24
3.96
7.16
2.50
2.26
1.10
3.87
1.53
6.34
5.57
2.53
8.11
1.27
1.43
1.42
2.56
3.73
1.60
5.46
1.33
2.59
1.77
6.46
25.00

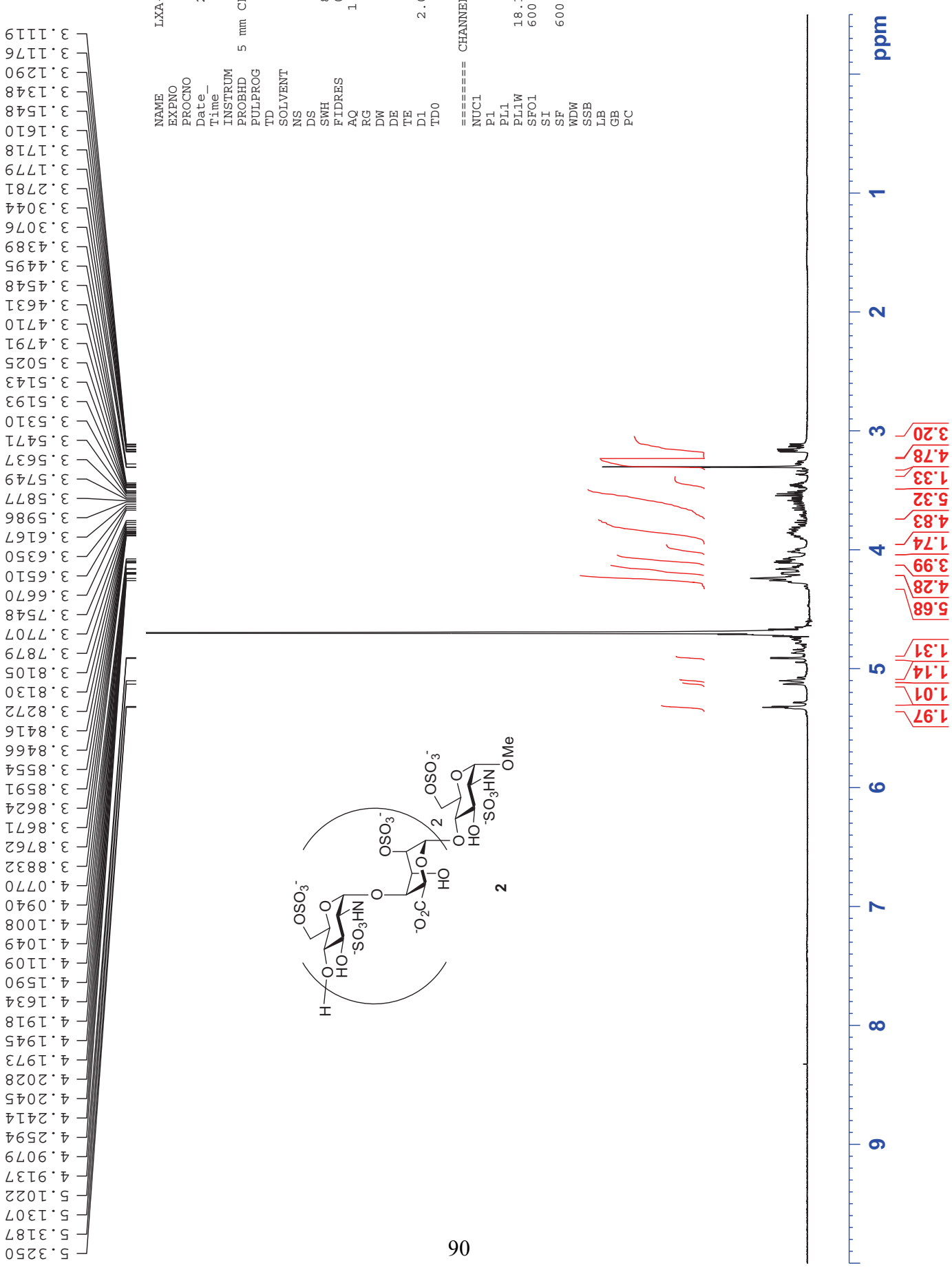


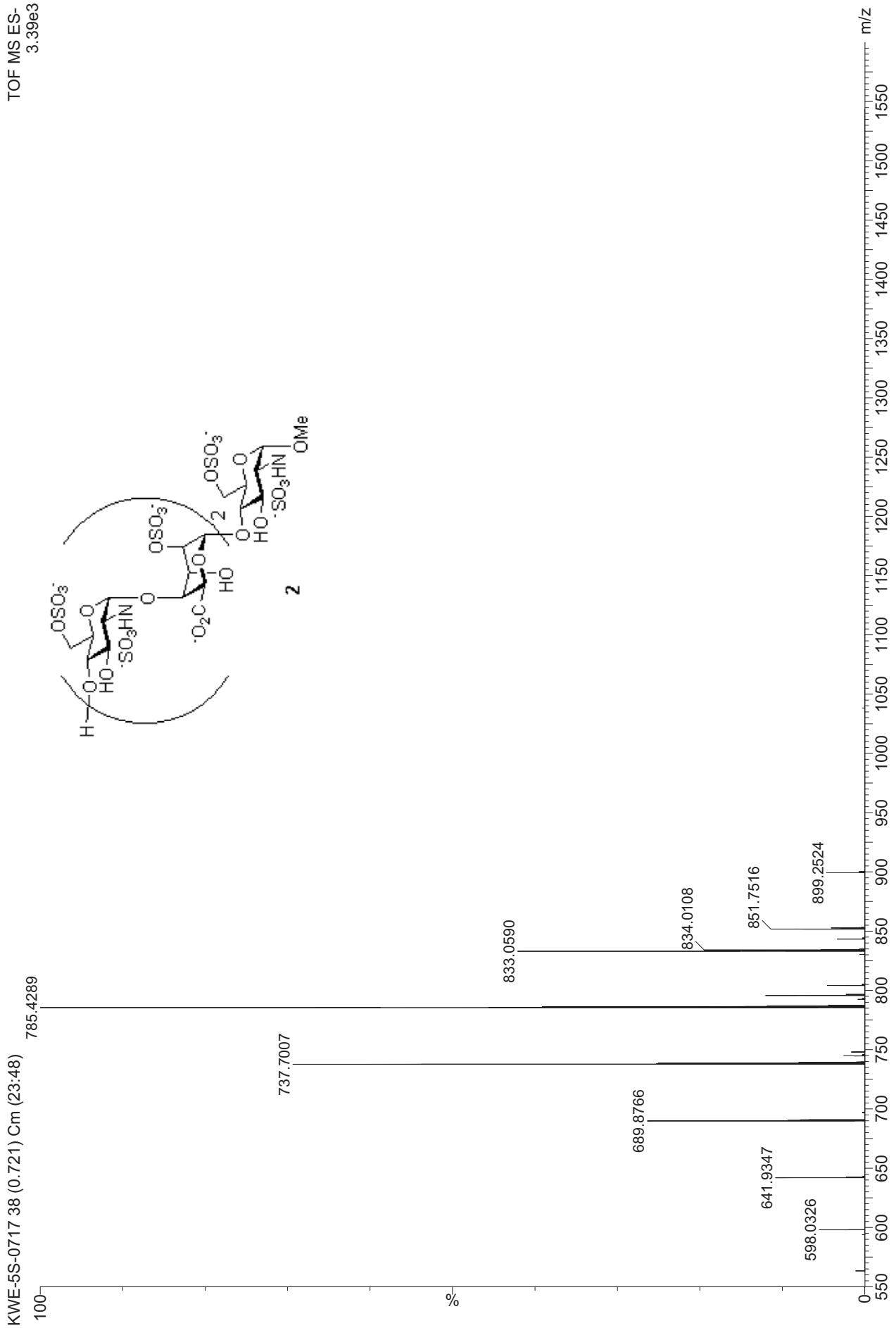

Current Data Parameters
NAME lxa-584
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090122
Time_ 11.36
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 32768
SOLVENT MeOD
NS 128
DS 0
SWH 7183.908 Hz
FIDRES 0.219235 Hz
AQ 2.2807028 sec
RG 128
DW 69.600 usec
DE 6.50 usec
TE 295.7 K
D1 1.50000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.20 usec
PL1 2.00 dB
SFO1 599.2029551 MHz
F2 - Processing parameters
SI 16384
SF 599.2000000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 0.50
ppm
49
2-NAP
BnO
OSO3-
N3
OMe
-O2C
3
1.00
1.03
0.95
1.05
1.12
1.57
14.96
1.69
6.27
14.17
4.36
8.37
2.84
3.93
2.46
7.16
3.11
4.40
1.93
3.03
1.95
3.77
1.33
12.21
24.70
3.20
7.91
4.65
3.91
1.40

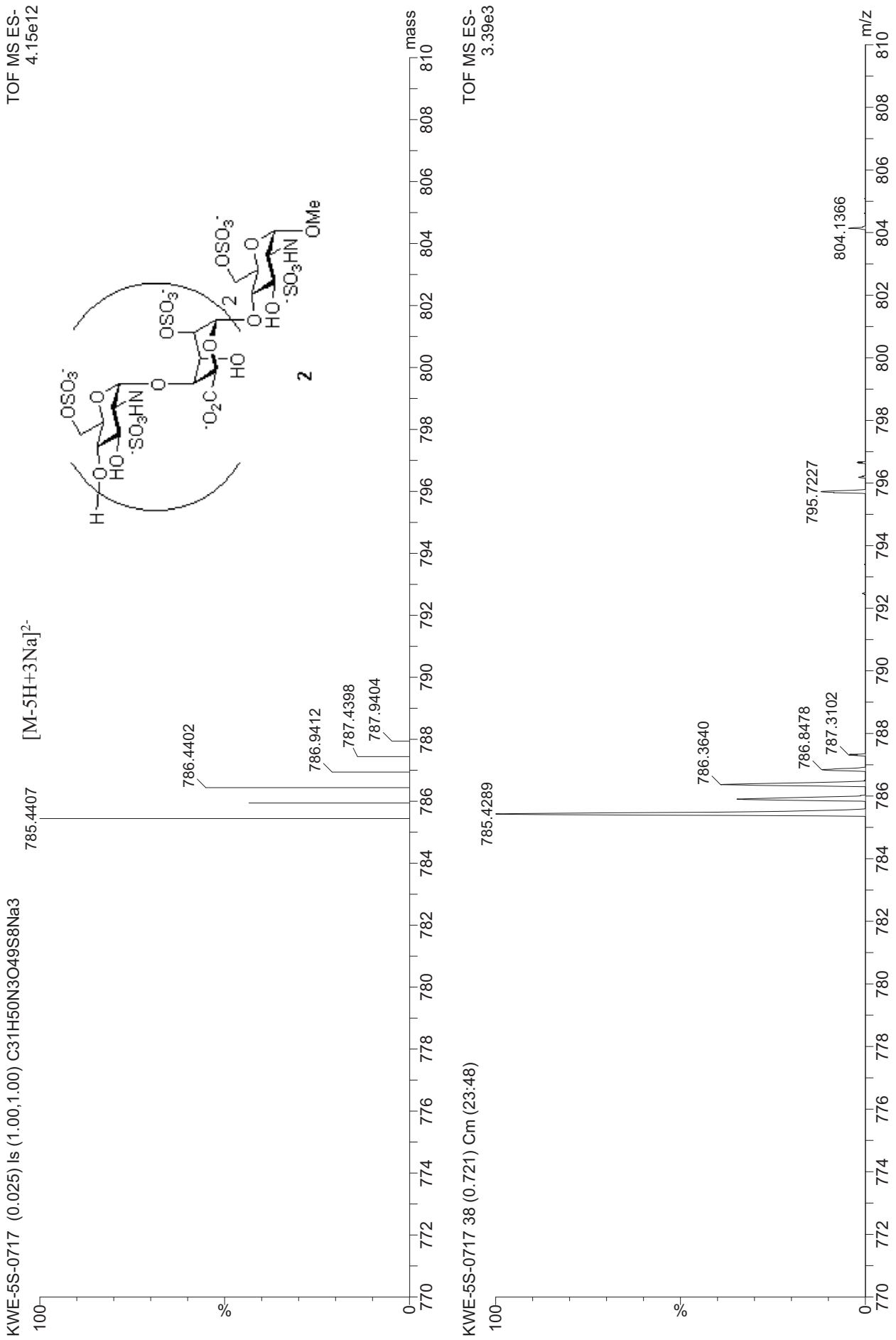


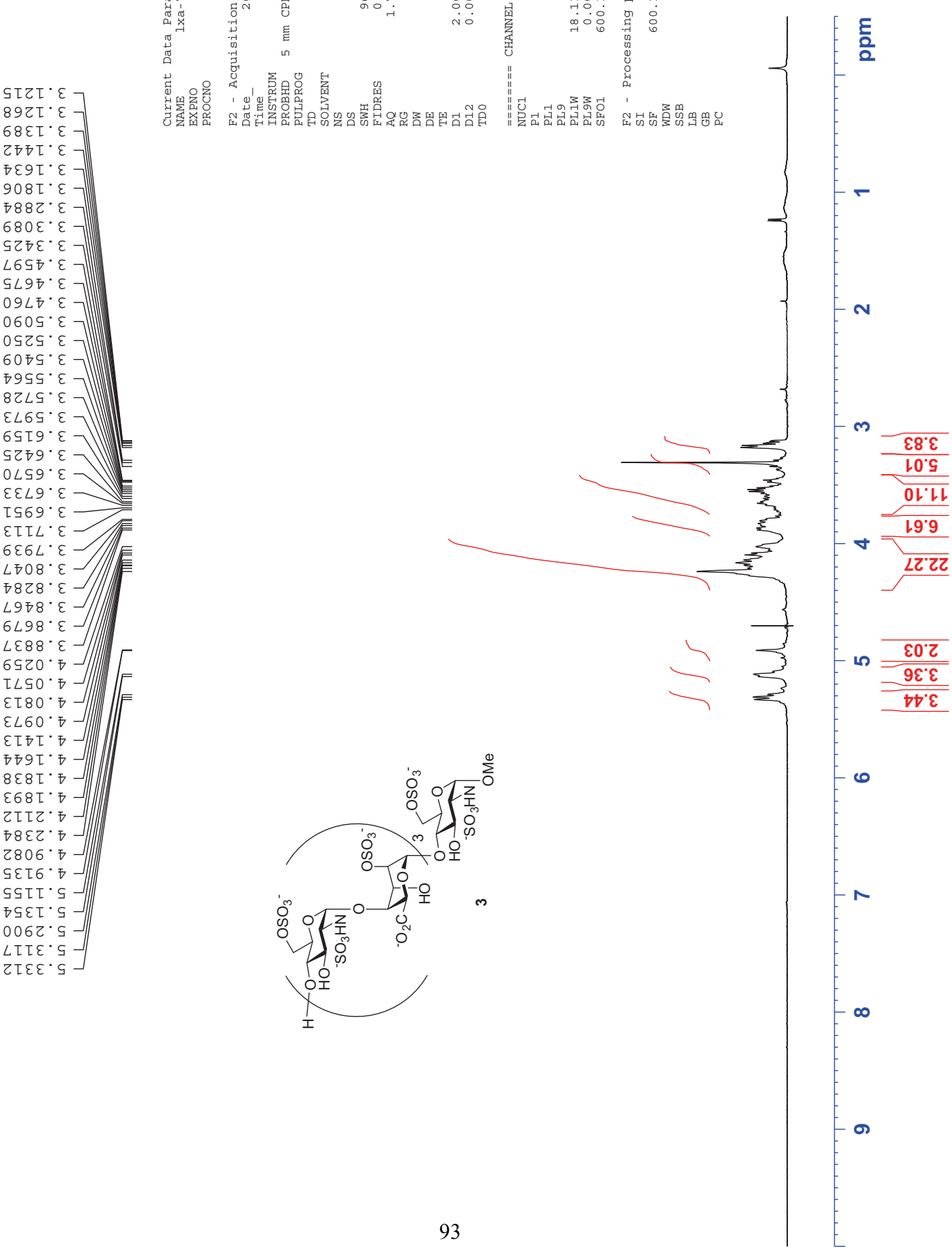


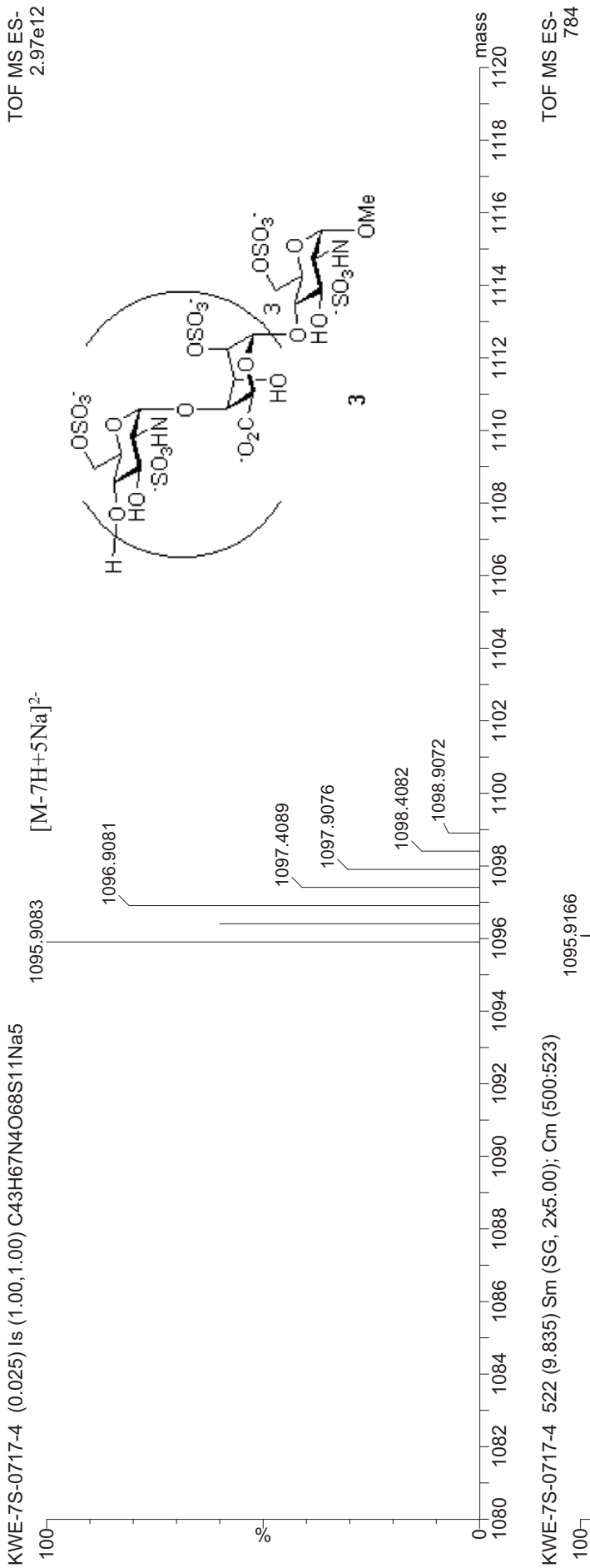

TOF MS ES-
5.80e12
KWE-3S-0717-2 (0.025) Is (1.00,1.00) C19H30N2O30S5Na4
[M-6H+4Na]2-
507.9460
508.9453
509.4464
1
TOF MS ES-
2.23e4
KWE-3S-0717-2 31 (0.589) Cm (24:41)
507.9247
508.9388
509.4463
514.9428
515.9547
mass
m/z
%
100
0

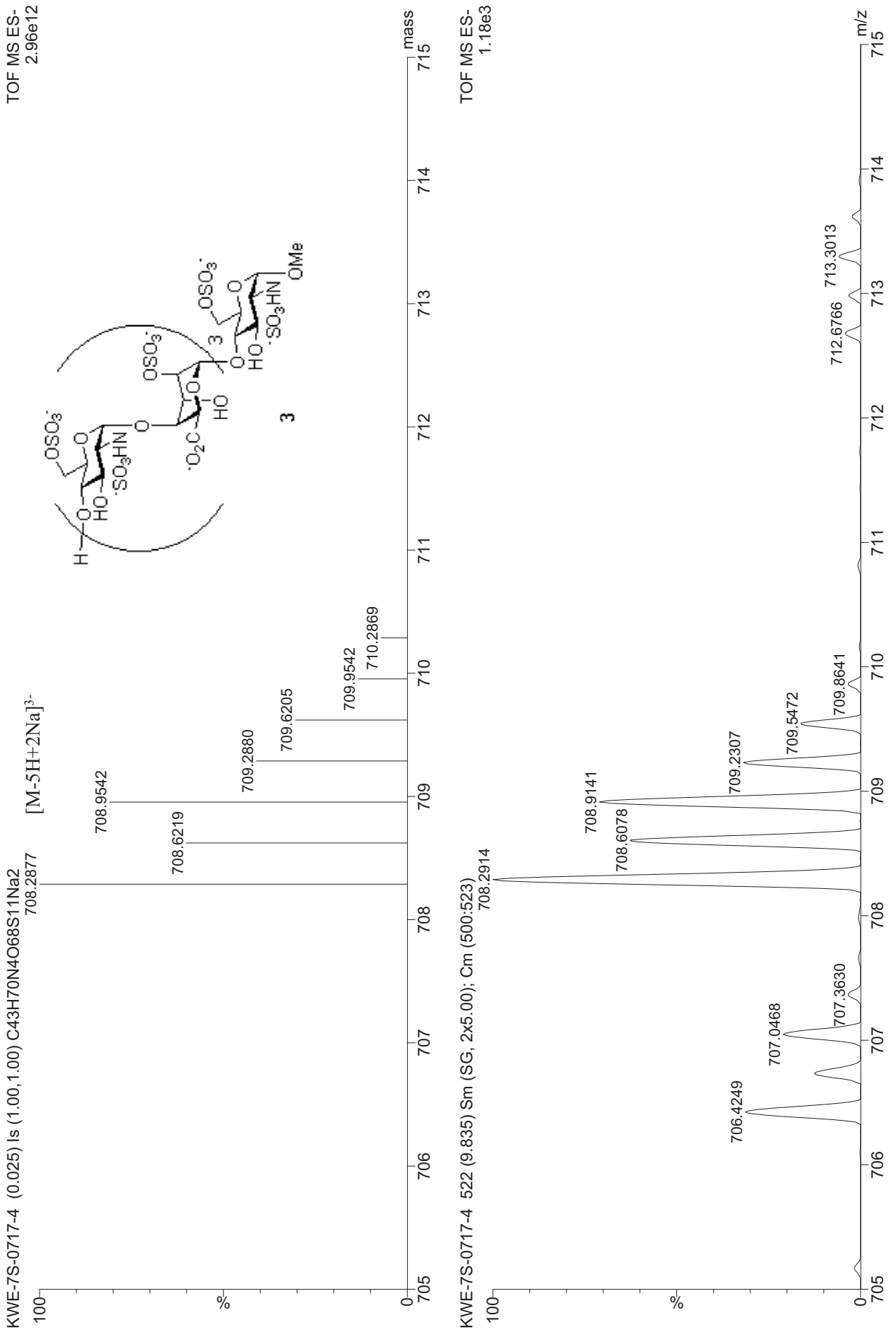

NAME LXA-5-Final
EXPNO 1
PROCNO 1
Date_ 20100630
Time 15.58
INSTRUM spect
PROBHD 5 mm CPDCH 13C
PULPROG zg30
TD 32768
SOLVENT D2O
NS 35
DS 0
SWH 8389.262 Hz
FIDRES 0.256020 Hz
AQ 1.9530824 sec
RG 36
DW 59.600 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -0.80 dB
PL1W 18.11971092 W
SFO1 600.1536010 MHz
SI 16384
SF 600.1500017 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
ppm
1.97
1.01
1.14
1.31
5.68
4.28
3.99
1.74
4.83
5.32
1.33
4.78
3.20
2


TOF MS ES-
3.39e3
KWE-5S-0717 38 (0.721) Cm (23:48)
100
%
0
m/z
550
600
650
700
750
800
850
900
950
1000
1050
1100
1150
1200
1250
1300
1350
1400
1450
1500
1550
785.4289
737.7007
689.8766
641.9347
598.0326
833.0590
834.0108
851.7516
899.2524
OSO3-
SO3HN
OMe
2


TOF MS ES-
4.15e12
[M-5H+3Na]2-
785.4407
786.4402
786.9412
787.4398
787.9404
KWE-5S-0717 (0.025) Is (1.00,1.00) C31H50N3O49S8Na3
100
%
0
770 772 774 776 778 780 782 784 786 788 790 792 794 796 798 800 802 804 806 808 810
mass
OSO3-
SO3HN
O2C
HO
OMe
2
TOF MS ES-
3.39e3
785.4289
786.3640
786.8478
787.3102
795.7227
804.1366
KWE-5S-0717 38 (0.721) Cm (23:48)
m/z


Current Data Parameters
NAME lxa-7-final
EXPNO 1111
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100708
Time 21.09
INSTRUM spect
PROBHD 5 mm CPDCH 13C
PULPROG zgpr
TD 32768
SOLVENT D2O
NS 32
DS 4
SWH 9615.385 Hz
FIDRES 0.293438 Hz
AQ 1.7040380 sec
RG 18
DW 52.000 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D12 0.00002000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -0.80 dB
PL9 54.25 dB
PL1W 18.11971092 W
PL9W 0.00005664 W
SFO1 600.1528227 MHz
F2 - Processing parameters
SI 16384
SF 600.1500000 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
3.44
3.36
2.03
22.27
6.61
11.10
5.01
3.83
ppm
3
5.3312
5.3117
5.2900
5.1354
5.1155
4.9135
4.9082
4.2384
4.2112
4.1893
4.1838
4.1644
4.1413
4.0973
4.0813
4.0571
4.0259
3.8837
3.8679
3.8467
3.8284
3.8047
3.7939
3.7113
3.6951
3.6733
3.6570
3.6425
3.6159
3.5973
3.5728
3.5564
3.5409
3.5250
3.5090
3.4760
3.4675
3.4597
3.4425
3.3089
3.2884
3.1806
3.1634
3.1442
3.1389
3.1268
3.1215




TOF MS ES-
2.96e12
KWE-7S-0717-4 (0.025) Is (1.00,1.00) C43H70N4O68S11Na2
[M-5H+2Na]3-
708.2877
708.6219
708.9542
709.2880
709.6205
709.9542
710.2869
3
mass
TOF MS ES-
1.18e3
KWE-7S-0717-4 522 (9.835) Sm (SG, 2x5.00); Cm (500:523)
706.4249
707.0468
707.3630
708.2914
708.6078
708.9141
709.2307
709.5472
709.8641
712.6766
713.3013
m/z