Chemoselective Per-*O*-trimethylsilylation and Homogeneous *N*-functionalisation of Amino Sugars

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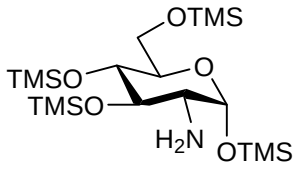
SUPPORTING INFORMATION

General Information

All reactions were conducted in flame-dried glassware, under nitrogen atmosphere. Dichloromethane, *N*,*N*-dimethyformamide, methanol and acetonitrile were dried in a safe purification system containing activated Al_2O_3. All reagents obtained from commercial sources were used without purification, unless otherwise mentioned. Flash column chromatography was carried out on Silica Gel Geduran® Si 60 (0.040-0.063, E. Merck) and DAVISIL® (LC60A 40-63 micron), TLC was performed on pre-coated glass plates of Silica Gel 60 F254 (0.25mm, E. Merck); detection was executed by spraying with a solution of cerium(IV) sulfate, ammonium molybdate and H_2SO_4 in water and subsequent heating on a hot plate. Optical rotations were measured on Jasco DIP-370 using a 100 mm cell at 589 nm. IR spectra were taken with a Perkin-Elmer Paragon 1000 FT-IR spectrometer and Bruker ALPHA ECO-ATR. ^{1}H, ^{13}C NMR, DEPT, ^{1}H-^{1}H COSY, ^{1}H-^{13}C COSY, and NOESY spectrum were recorded with Bruker AV 400, AVIII 400 and AV 500 MHz instruments. Chemical shifts are in ppm from Me_4Si, generated from the $CDCl_3$ lock signal at 7.24, CD_3OD lock signal at 4.78 and D_2O lock signal at 4.80. All ^{13}C NMR spectra contain the ^{13}C at the bottom trace, and the middle and the upper traces are DEPT 90, 135 respectively. Multiplicities are reports by using the following abbreviations: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad, pentet, *J* = coupling constant values in Hertz. Mass spectra was analyzed on a Waters Premier XE instrument with ESI source.



2

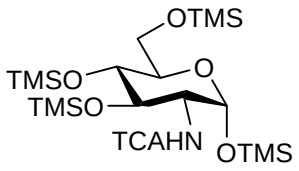
2-Amino-2-deoxy-1,3,4,6-tetra-*O*-trimethylsilyl-α-D-glucopyranose (2). To a suspension of D-Glucosamine hydrochloride (100 mg) in CH_3CN (1 mL) was added HMDS (245 μL, 2.5 eq) at rt under N_2 atmosphere, and the mixture was allowed to stir at rt for 3 h. The reaction was monitored by TLC or ^1H NMR. Upon finishing, the mixture was filtered and the filtrate was evaporated *in vacuo* to furnish the desired product **2** as a colorless crystalline form (211 mg, quantitative).

This reaction is easily scaled up. To a suspension of D-glucosamine hydrochloride (25 g) in CH_3CN (250 mL) was added HMDS (61.6 mL, 2.5 eq) at rt under N_2 atmosphere, and the mixture was allowed to stir at rt for 3 h. The reaction was monitored by TLC or ^1H NMR. Upon finishing, the mixture was filtered and the filtrate was evaporated *in vacuo* to furnish the desired product **2** as a colorless crystalline form (54 g, quantitative). $[\alpha]^{28}_D$ +0.1 (*c* 0.5, $CHCl_3$); IR ($CHCl_3$) ν 3382, 3207, 2957, 2923, 1575, 1384, 1251, 1150 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 5.08 (d, *J* = 3.2 Hz, 1H, H-1), 3.61 (ddd, *J* = 9.4, 4.3, 2.5 Hz, 1H, H-5), 3.50 (t, *J* = 8.9 Hz, 1H, H-3), 3.43 (t, *J* = 8.9 Hz, 1H, H-4), 2.50 (dd, 1H, *J* = 9.0, 3.2 Hz, H-2), 3.71-3.66 (m, 2H, H-6a, H-6b), 0.17 (s, 9H, TMS), 0.14 (s, 9H, TMS), 0.13 (s, 9H, TMS), 0.06 (s, 9H, TMS); ^{13}C NMR (100 MHz, $CDCl_3$) δ 94.5 (CH), 76.9 (CH), 73.0 (CH), 71.0 (CH), 63.7 (CH_2), 57.0 (CH), 1.5 (CH_3), 1.0 (CH_3), 0.1 (CH_3), -0.04 (CH_3); HRMS (ESI) calcd for $C_{18}H_{46}NO_5Si_4$ $[M+H]^+$ 468.2453, found 468.2460.

General Procedures for Homogeneous *N*-functionalization of Amino Sugars

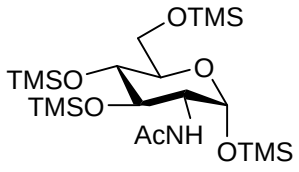
To a solution of compound **2** (100 mg, 1 eq) in CH_2Cl_2/Pyr (7/3) (1 mL) was added the functionalization reagent (1.1 eq), such as trichloroacetyl chloride, acetic anhydride, triflouro acetic anhydride, 2,2,2-trichloroethoxycarbonyl chloride, benzyl chloroformate, methanesulfonyl chloride, *p*-toluenesulfonyl chloride, dansyl chloride, benzenesulfonyl chloride, *p*-nitrobenzenesulfonyl chloride, 2,4-dinitrobenzenesulfonyl chloride and lauryl chloride, respectively at 0 °C, and the mixture was allowed to stir for 2 h. The mixture was concentrated *in vacuo* and was then filtered through a short pad of silica gel, which was washed with 10:1 (Hex:EA). The filtrate was evaporated *in vacuo* to furnish the desired

products.



4

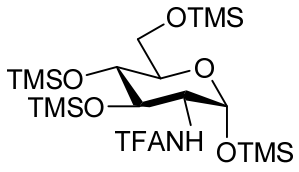
2-Deoxy-2-trichloroacetamido-1,3,4,6-tetra-*O*-trimethylsilyl-α-D-glucopyranose (4). As a yellowish syrup (113 mg, 87%). $[\alpha]^{28}_{D}$ 76.6 (*c* 0.5, $CHCl_3$); IR ($CHCl_3$) ν 1725, 1507, 1251, 1145, 1060, 978, 842, 750 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 6.80 (d, *J* = 9.6 Hz, 1H, N-H), 5.09 (d, *J* = 3.4, 1H, H-1), 3.91 (td, *J* = 9.6, 3.4 Hz, 1H, H-2), 3.76 (dd, *J* = 9.6, 7.6 Hz, 1H, H-3), 3.57-3.73 (m, 4H, H-4, H-5, H-6a, H-6b), 0.16 (s, 9H, TMS), 0.14 (s, 9H, TMS), 0.13 (s, 9H, TMS), 0.08(s, 9H, TMS); ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.9 (C), 91.9 (CH), 73.6 (CH), 73.2 (CH), 71.98 (CH), 61.7 (CH_2), 57.12 (CH), 1.27 (CH_3), 1.01 (CH_3), 0.04 (CH_3), -0.10(CH_3); HRMS (ESI) calcd for $C_{20}H_{44}NO_6Si_4NaCl_3$ $[M+Na]^+$ 634.1209, found 634.1206.



5

2-Acetamido-2-deoxy-1,3,4,6-tetra-*O*-trimethylsilyl-α-D-glucopyranose (5).

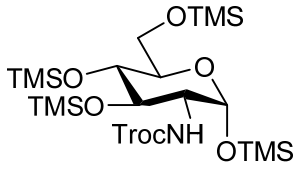
As a white solid (83 mg, 77%), $[\alpha]^{28}_{D}$ +0.1 (*c* 0.5, $CHCl_3$); IR ($CHCl_3$) ν 3278, 2956, 1650, 1249, 843 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 5.31 (d, *J* = 9.8 Hz, 1H, N-H), 5.02 (d, *J* =3.4 Hz, 1H, H-1), 3.99 (td, *J* =, 9.6, 3.4 Hz, 1H, H-2), 3.57-3.71 (m, 5H, H-3, H-4, H5, H-6a, H-6b), 0.14 (s, 9H, TMS), 0.12 (s, 9H, TMS), 0.11 (s, 9H, TMS), 0.06 (s, 9H, TMS); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.8 (C), 92.7 (CH), 74.2 (CH), 72.7 (CH), 72.2 (CH), 61.9 (CH_2), 54.7 (CH), 24.0 (CH), 1.32 (CH_3), 1.03 (CH_3), 0.07 (CH_3), -0.08 (CH_3); HRMS (ESI) calcd for $C_{20}H_{47}NO_6Si_4Na$ $[M+Na]^+$ 532.2378, found 532.2387.



6

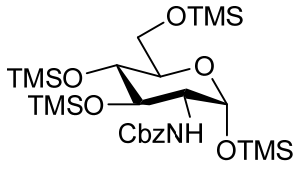
2-Deoxy-2-trifluoroacetamido-1,3,4,6-tetra-*O*-trimethylsilyl-α-D-glucopyranose (6). As a

colourless syrup (106 mg, 88%). $[\alpha]^{25}_D$ 78.0 (*c* 0.5, $CHCl_3$); IR ($CHCl_3$) ν 3299, 2918, 1697, 1557, 1188, 1042 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 6.28 (d, *J* = 9.4 Hz, 1H, N-H), 5.12 (d, *J* = 3.4 Hz, 1H, H-1), 4.01 (td, *J* = 9.6, 3.4 Hz, 1H, H-2), 3.61-3.78 (m, 5H, H-3, H-4, H-5, H-6a, H-6b), 0.19 (s, 9H, TMS), 0.17 (s, 9H, TMS), 0.15 (s, 9H, TMS), 0.12 (s, 9H, TMS); ^{13}C NMR (100 MHz, $CDCl_3$) δ 91.8 (CH), 74.1 (CH), 73.4 (2xCH), 71.8 (CH), 61.6 (CH_2), 55.4 (CH), 1.03 (CH_3), 0.96 (CH_3), -0.11 (2 x CH_3); HRMS (ESI) calcd for $C_{20}H_{44}NO_6Si_4NaF_3$ $[M+Na]^+$ 586.2096, found 586.2094.



7

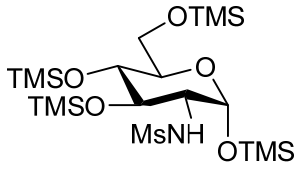
2-Deoxy-2-(2,2,2-Trichloroethoxycarbonylamino)-1,3,4,6-tetra-*O*-trimethylsilyl-α-D-glucopyranose (7). As a colourless syrup (125 mg, 91%). $[\alpha]^{29}_D$ 71.8 (*c* 0.5, $CHCl_3$); IR ($CHCl_3$) ν 1748, 1508, 1250, 1156, 1048, 974, 841, 750 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 5.07 (d, *J* = 2.9 Hz, 1H, H-1), 4.86 (d, *J* = 9.4 Hz, 1H, N-H), 4.58 (ABq *J* = 12.0 Hz, 2H), 3.57-3.73 (m, 6H, H-2, H-3, H-4, H-5, H-6a, H-6b), 0.15 (s, 9H, TMS), 0.13 (s, 9H, TMS), 0.12 (s, 9H, TMS), 0.07 (s, 9H, TMS); ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.4 (C), 92.7 (CH), 74.9 (Troc CH_2), 74.1 (CH), 72.6 (CH), 72.2 (CH), 61.8 (CH_2), 57.0 (CH), 1.2 (CH_3), 1.0 (CH_3), 0.05 (CH_3), -0.07 (CH_3); HRMS (ESI) calcd for $C_{21}H_{46}NO_7Si_4NaCl_3$ $[M+Na]^+$ 664.1315, found 664.1320.



8

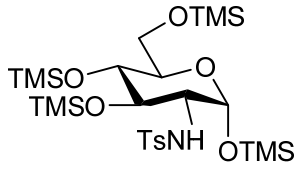
2-Carbobenzoxylamino-2-deoxy-1,3,4,6-tetra-*O*-trimethylsilyl-α-D-glucopyranose (8). As a colourless syrup (104 mg, 81%). $[\alpha]^{28}_D$ +0.1 (*c* 0.5, $CHCl_3$); IR ($CHCl_3$) ν 2956, 1732, 1717, 1507, 1249, 840 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.34-7.26 (m, 5H, Ar), 5.07 (s, 2H, Ph-CH_2), 5.05 (d, *J* = 3.4 Hz, 1H, H-1), 4.68 (d, *J* = 10.0 Hz, NH), 3.72-3.53 (m, 6H, H-2, H-3, H-4, H-5, H-6a, H-6b), 0.15 (s, 9H, TMS), 0.10 (s, 9H, TMS), 0.08 (s, 9H, TMS), 0.07 (s, 9H, TMS); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.1 (C), 136.6 (C), 128.7 (CH), 128.6 (CH), 128.4 (CH), 93.0 (CH), 74.4 (CH), 72.6 (CH), 72.3 (CH), 67.2 (CH_2), 62.0 (CH_2), 56.8 (CH),

1.1 (CH_3), 1.0 (CH_3), 0.0 (CH_3), -0.1 (CH_3); HRMS (ESI) calcd for $C_{26}H_{51}NO_7Si_4Na$ $[M+Na]^+$ 624.2640, found 624.2642.



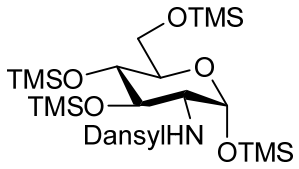
9

2-Deoxy-2-methanesulfonylamino-1,3,4,6-tetra-*O*-trimethylsilyl-α-D-glucopyranose (9). As colourless syrup (99 mg, 85%). $[\alpha]^{28}_D$ +0.1 (*c* 0.5, $CHCl_3$); IR ($CHCl_3$) ν 3294, 2957, 1326, 1251, 1150, 1069, 842 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 5.23 (d, *J* = 3.4 Hz, 1H, H-1), 4.39 (d, = 9.6 Hz, 1H, NH), 3.72-3.63 (m, 2H, H-6a, H-6b), 3.62-3.56 (m, 2H, H-3, H-5), 3.50 (t, *J* = 8.6 Hz, 1H, H-4), 3.16 (td, *J* = 9.6, 3.4 Hz, 1H, H-2), 2.96 (s, 3H), 0.16 (s, 18H, 2 x TMS), 0.16 (s, 9H, TMS), 0.08 (s, 9H, TMS); ^{13}C NMR (100 MHz, $CDCl_3$) δ 93.2 (CH), 73.3 (CH), 73.2 (CH), 72.4 (CH), 61.7 (CH_2), 59.5 (CH), 41.7 (CH_3), 1.4 (CH_3), 1.0 (CH_3), 0.1 (CH_3), -0.1 (CH_3); HRMS (ESI) calcd for $C_{19}H_{46}NO_7Si4S$ $[M-H]^-$ 544.2072, found 544.2079.



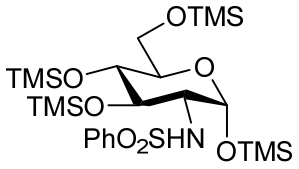
10

2-Deoxy-2-(3-methylbenzensulfonylamino)-1,3,4,6-tetra-*O*-trimethylsilyl-α-D-glucopyranose (10). As a colourless syrup (99 mg, 75%). $[\alpha]^{28}_D$ 0.0 (*c* 0.5, $CHCl_3$); IR ($CHCl_3$) ν 2957, 2922, 1250, 1168, 1069, 843 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.72 (d, *J* = 8.2 Hz, 2H, Ar), 7.26 (d, *J* = 8.2 Hz, 2H, Ar), 4.52 (d, *J* = 10.0 Hz, 1H, NH), 4.49 (d, *J* = 3.5 Hz, 1H, H-1), 3.61 (ddd, *J* = 16.5, 11.3, 2.4 Hz, 2H, H-6 a, H-6b), 3.55 (t, *J* = 9.5 Hz, 1H, H-3), 3.49 (ddd, *J* = 9.3, 6.9, 2.4 Hz, 1H, H-5), 3.42 (t, *J* = 8.2 Hz, 1H, H-4), 3.20 (td, *J* = 10.0, 3.5 Hz, 1H, H-2), 2.39 (s, 3H), 0.15 (s, 9H, TMS), 0.14 (s, 9H, TMS), 0.04 (s, 9H, TMS), -0.03 (s, 9H, TMS); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.7 (C), 139.1 (C), 130.0 (CH), 127.0 (CH), 92.1 (CH), 73.4 (CH), 72.7 (CH), 72.5 (CH), 62.0 (CH_2), 59.3 (CH), 21.6 (CH_3), 1.5 (CH_3), 1.1 (CH_3), -0.1 (CH_3), -0.2 (CH_3); HRMS (ESI) calcd for $C_{25}H_{51}NO_7Si_4NaS$ $[M+Na]^+$ 644.2361, found 644.2357.



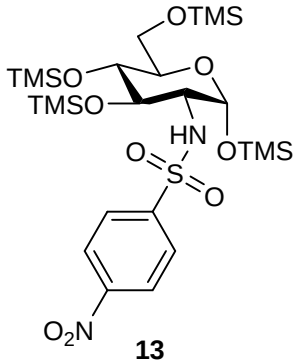
11

2-Deoxy-2-[(5-dimethylamino)napthalene-1-sulfonylamino]-1,3,4,6-tetra-*O*-trimethylsilyl-α-D-glucopyranose (11). As a yellowish syrup (133 mg, 89%). $[\alpha]^{28}_{D}$0.0 (*c* 0.5, $CHCl_3$); IR ($CHCl_3$) ν 2954, 1311, 1201, 1102, 1058, 836 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.50 (d, *J* = 8.5 Hz, Ar), 8.31 (d, *J* = 8.6 Hz, Ar), 8.25 (dd, *J* = 7.3, 1.1 Hz, 1H, Ar), 7.55 (t, *J* = 7.7 Hz, 1H, Ar), 7.48 (dd, *J* = 8.4, 7.4 Hz, Ar), 7.17 (d, *J* = 7.5 Hz, Ar), 4.84 (d, *J* = 10.0 Hz, 1H, NH), 3.97 (d, *J* = 3.5 Hz, 1H, H-1), 3.60 (t, *J* = 9.0 Hz, 1H, H-3), 3.55-3.51 (m, 2H, H-6a, H-6b), 3.44-3.37 (m, 2H, H-4, H-5), 3.20 (td, *J* = 9.0, 3.5 Hz, 1H, H-2), 2.84 (s, 6H), 0.2 (s, 9H, TMS), 0.14 (s, 9H, TMS), -0.01 (s, 9H, TMS), -0.3 (s, 9H, TMS); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.6 (C), 136.9 (C), 130.5 (CH), 130.3 (C), 129.5 (C), 129.2 (CH), 129.1 (CH), 123.7 (CH), 118.9 (CH), 115.6 (CH), 92.0 (CH), 73.4 (CH), 72.7 (CH), 72.5 (CH), 62.0 (CH_2), 59.5 (CH), 45.6 (2 x CH_3), 1.6 (CH_3), 1.1 (CH_3), -0.2 (CH_3), -0.6 (CH_3); HRMS (ESI) calcd for $C_{30}H_{56}N_2O_7Si_4NaS$ $[M+Na]^+$ 723.2783, found 723.2781.



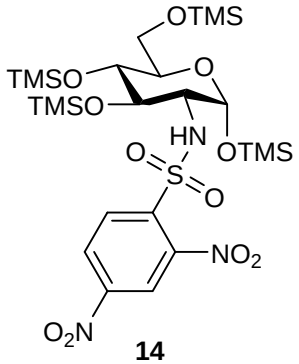
12

2-Benzensulfonylamino-2-deoxy-1,3,4,6-tetra-*O*-trimethylsilyl-α-D-glucopyranose (12). As a colourless syrup (101 mg, 78%). $[\alpha]^{25}_{D}$ 212.5 (*c* 0.5, $CHCl_3$); IR ($CHCl_3$) ν 3301, 2956, 2922, 1251, 1169, 1070, 843 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.86-7.84 (m, 2H, Ar), 7.57-7.53 (m, 1H, Ar), 7.50-7.46 (m, 2H, Ar), 4.56 (d, *J* = 10.0 Hz, 1H, NH), 4.52 (d, *J* = 3.4 Hz, 1H, H-1), 3.61 (ddd, *J* = 14.1, 12.2, 2.8 Hz, 2H, H-6a, H-6b), 3.56 (t, *J* = 8.8 Hz, 1H, H-3), 3.51-3.41 (m, 2H, H-4, H-5), 3.20 (td, *J* = 10.0, 3.4 Hz, 1H, H-2), 0.15 (s, 9H, TMS), 0.14 (s, 9H, TMS), 0.04 (s, 9H, TMS), -0.02 (s, 9H, TMS); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.1 (C), 132.8 (CH), 129.5 (2 x CH), 127.0 (2 x CH), 92.2 (CH), 73.3 (CH), 72.9 (CH), 72.5 (CH), 62.2 (CH_2), 59.4 (CH), 1.47 (CH_3), 1.1 (CH_3), 0.0 (CH_3), -0.1 (CH_3); HRMS (ESI) calcd for $C_{24}H_{49}NO_7Si_4NaS$ $[M+Na]^+$ 630.2205, found 630.2195.



13

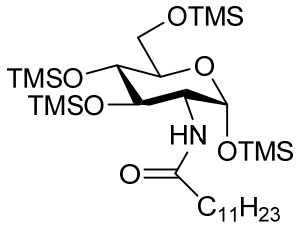
2-Deoxy-2-(4-nitrobenzensulfonylamino)-1,3,4,6-tetra-*O*-trimethylsilyl-α-D-glucopyranose (13). As a yellowish solid (107 mg, 77%). $[\alpha]^{28}_{D}$ 0.0 (*c* 0.5, $CHCl_3$); IR ($CHCl_3$) ν 2957, 1533, 1379, 1251, 1173, 1068, 843 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.32 (d, *J* = 8.6 Hz, Ar), 8.04 (d, *J* = 8.7 Hz, Ar), 4.74 (d, J= 10.0 Hz, 1H, NH), 4.72 (d, J= 3.4 Hz, 1H, H-1), 3.63-3.57 (m, 3H, H-3, H-6a, H-6b), 3.52 (dt, *J* = 3.6, 10.0 Hz, 1H, H-5), 3.46 (t, *J* = 10.0 Hz, 1H, H-4), 3.26 (td, *J* = 10.0, 3.4 Hz, 1H, H-2), 0.1 (s, 9H, TMS), 0.08 (s, 9H, TMS), 0.04 (s, 9H, TMS), 0.01 (s, 9H, TMS); ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.2 (C), 147.6 (C), 128.4 (CH), 124.6 (CH), 92.2 (CH), 73.3 (CH), 73.1 (CH), 72.2 (CH), 61.7 (CH_2), 59.7 (CH), 1.3 (CH_3), 1.0 (CH_3), 0.0 (CH_3), -0.2 (CH_3); HRMS (ESI) calcd for $C_{24}H_{48}N_2O_9Si_4NaS$ $[M+Na]^+$ 675.2055, found 675.2045.



14

2-Deoxy-2-(2,4-dinitrobenzensulfonylamino)-1,3,4,6-tetra-*O*-trimethylsilyl-α-D-glucopyranose (14). As a yellowish solid (116 mg, 78%). $[\alpha]^{28}_{D}$ +0.1 (*c* 0.5, $CHCl_3$); IR ($CHCl_3$) ν 3351, 2957, 1554, 1551, 1348, 1251, 1068, 843 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.64 (d, *J* = 2.2 Hz, Ar), 8.53 (dd, *J* = 8.6, 2.2 Hz, Ar), 8.34 (d, *J* = 8.6 Hz, 1H, Ar), 5.78 (d, *J* = 9.6 Hz, 1H, NH), 4.68 (d, *J* = 3.3 Hz, 1H, H-1), 3.71 (dd, *J* = 9.6, 7.4 Hz, 1H, H-3), 3.64 (ddd, *J* = 15.2, 11.2, 3.7 Hz, 2H, H-6 a, H-6b), 3.58-3.50 (m, 2H, H-5, H-4), 3.74 (td, *J* = 9.6, 3.3 Hz, 1H, H-2), 0.16 (s, 9H, TMS), 0.1 (s, 9H, TMS), 0.05 (s, 9H, TMS), -0.06 (s, 9H, TMS); ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.9 (C), 148.4 (C), 141.2 (C), 132.1 (CH), 127.6

(CH), 120.7 (CH), 92.1 (CH), 73.5 (CH), 73.3 (CH), 72.2 (CH), 61.6 (CH_2), 60.4 (CH), 1.35 (CH_3), 1.0 (CH_3), -0.2 (CH_3), -0.2 (CH_3); HRMS (ESI) calcd for $C_{24}H_{47}N_3O_{11}Si_4NaS$ $[M+Na]^+$ 720.1906, found 720.1905.

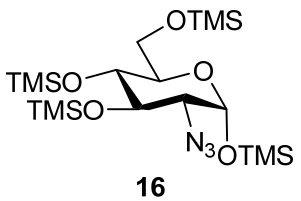


15

2-Deoxy-2-lauricamido-1,3,4,6-tetra-*O*-trimethylsilyl-α-D-glucopyranose (15). As a yellowish solid (119 mg, 86%). $[\alpha]^{25}_D$ 141.5 (*c* 0.5, $CHCl_3$); IR ($CHCl_3$) ν 3308, 2923, 2853, 1698, 1507, 1250, 844 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 5.28 (d, *J* = 9.8 Hz, 1H, NH), 5.0 (d, *J* = 3.5 Hz, 1H, H-1), 3.98 (td, *J* = 9.5, 3.5 Hz, 1H, H-2), 3.70-3.55 (m, 5H, H-3, H-4, H-5, H-6a, H-6b), 2.18 (pentet, *J* = 14.9, 7.2 Hz, 1H), 2.07 (pentet, *J* = 15.2, 8.0, Hz, 1H), 1.58 (m, 2H), 1.25-1.21 (m, 18H), 0.83 (t, *J* = 7.0 Hz, 3H), 0.13 (s, 9H, TMS), 0.10 (s, 9H, TMS), 0.09 (s, 9H, TMS), 0.05 (s, 9H, TMS); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.9 (C), 92.8 (CH), 74.0 (CH), 72.7 (CH), 72.2 (CH), 61.9 (CH_2), 54.5 (CH), 37.4 (CH_2), 32.0 (CH_2), 29.7 (CH_2), 29.6 (CH_2), 29.5 (CH_2), 29.5 (CH_2), 25.8 (CH_2), 22.8 (CH_2), 14.2 (CH_2), 1.3 (CH_3), 0.9 (CH_3), 0.04 (CH_3), -0.11 (CH_3); HRMS (ESI) calcd for $C_{30}H_{67}NO_6Si_4Na$ $[M+Na]^+$ 672.3943, found 672.3948.

General procedure for per-*O*-trimethylsilylation of amino sugars

To a suspension of amino sugar in dry CH_3CN was added hexamethyldisilazane (2.5 eq). The reaction mixture was stirred at rt for 3 h and the reaction mixture was filtered through a pad of Celite and the pad was washed with Ethyl acetate. The filtrate was concentrated *in vacuo* to furnish the per-*O*-trimethylsilylation of amino sugars, which were used without further purification.



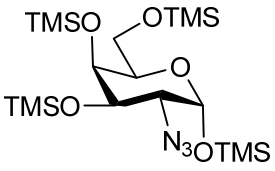
16

2-Azido-2-deoxy-1,3,4,6-tetra-*O*-trimethylsilyl-α-D-glucopyranose(16).

Trifluoromethanesulfonic anhydride (42 μL, 1.2 eq) was slowly added drop wise from an

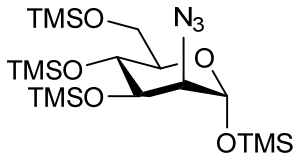
addition funnel to a solution of sodium azide (55 mg, 4 eq) in a mixture of water (1 mL) and CH_2Cl_2 (2 mL) at 0 ºC. After stirring at the same temperature for 2 h, the organic layer was separated, and the aqueous layer was extracted with CH_2Cl_2 (2 x 2 mL). The combined organic layers were neutralized with saturated $NaHCO_3$(aq). The generated trifluoromethanesulfonic azide (TfN_3) was directly used without further purification for the ensuing reaction. 4-Dimethylaminopyridine (78 mg, 3 eq) was added to a CH_2Cl_2 solution of the per-*O*-trimethylsilylated glucosamine **2** (100 mg, 1 eq) and the TfN_3 solution in CH_2Cl_2 was added. The ice-bath was removed and the reaction was kept stirring at room temperature for another 12 h. The mixture was filtered through a short pad of silica gel, which was further washed with hexane. The filtrate was concentrated *in vacuo* to furnish compound **16** (101 mg, 96%) as a colorless syrup.

This reaction is easily scaled up. Trifluoromethanesulfonic anhydride (4.27 mL, 1.2 eq) was slowly added drop wise from an addition funnel to a solution of sodium azide (5.58 g, 4 eq) in a mixture of water (44 mL) and CH_2Cl_2 (80 mL) at 0 ºC. After stirring at the same temperature for 2 h, the organic layer was separated, and the aqueous layer was extracted with CH_2Cl_2 (2 x 20 mL). The combined organic layers were neutralized with saturated $NaHCO_3$(aq). The generated trifluoromethanesulfonic azide (TfN_3) was directly used without further purification for the ensuing reaction. 4-Dimethylaminopyridine (7.85 g, 3 eq) was added to a CH_2Cl_2 solution of the per-*O*-trimethylsilylated glucosamine **2** (10 g, 1 eq) and the TfN_3 solution in CH_2Cl_2 was added. The ice-bath was removed and the reaction was kept stirring at room temperature for another 12 h. The mixture was quenched with Glycine (2.42 g, 1.5 eq) then filtered through a short pad of silica gel, which was further washed with hexane. The filtrate was concentrated *in vacuo* to furnish **16** (10.3 g, 98%) as a colorless syrup. $[\alpha]^{25}_D$ 77.8 (*c* 0.5, $CHCl_3$); IR ($CHCl_3$) ν 2956, 2103, 1249, 1148, 1110, 837 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 5.21 (d, *J* = 3.2 Hz, 1H, H-1), 3.86 (dd, *J* = 9.2, 8.2 Hz, 1H, H-3), 3.72 (ddd, *J* = 11.8, 9.2, 4.5 Hz, 1H, H-5), 3.68-3.63 (m, 2H, H-6a, H-6b), 3.53 (t, *J* = 9.2 Hz, 1H, H-4), 2.88 (dd, *J* = 9.2, 3.2 Hz, 1H, H-2), 0.20 (s, 9H, TMS), 0.16 (s, 9H, TMS), 0.15 (s, 9H, TMS), 0.07(s, 9H, TMS); ^{13}C NMR (100 MHz, $CDCl_3$) δ 93.2 (CH), 73.3 (CH), 72.4 (CH), 72.3 (CH), 65.2 (CH), 61.9 (CH_2),1.2 (CH_3), 0.9 (CH_3), 0.0 (CH_3), -0.1 (CH_3); HRMS (ESI) calcd for $C_{18}H_{43}N_3O_5Si_4Na$ $[M+Na]^+$ 516.2178, found 516.2179.



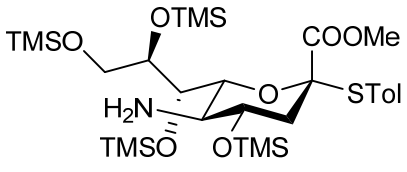
18

2-Azido-2-deoxy-1,3,4,6-tetra-*O*-trimethylsilyl-α-D-galactopyranose (18). Procedures were as shown for compound **16** to afford **18** as a colourless syrup (97 mg, 91%). $[\alpha]^{27}_{D}$ +85.6 (*c* 0.45, $CHCl_3$); IR ($CHCl_3$) ν 2956, 2922, 2107, 1472, 1249, 1164, 1028, 877 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 5.23 (d, *J* = 3.2 Hz, 1H, H-1), 3.95 (dd, *J* = 10.1, 2.8 Hz, 1H, H-3), 3.90 (d, *J* = 2.4 Hz, 1H, H-4), 3.81 (t, *J* = 6.4 Hz, 1H, H-5), 3.57 (dd, *J* = 9.7, 7.9 Hz, 1H, H-6a), 3.51-3.46 (m, 2H, H-2, H-6b), 0.19 (s, 9H, TMS), 0.16 (s, 9H, TMS), 0.11 (s, 9H, TMS), 0.09 (s, 9H, TMS); ^{13}C NMR (100 MHz, $CDCl_3$) δ 93.4 (CH), 71.6 (CH), 71.4 (CH), 70.0 (CH), 61.8 (CH), 61.1 (CH_2),0.8 (CH_3), 0.4 (CH_3), 0.0 (CH_3), -0.3 (CH_3); HRMS (ESI) calcd for $C_{18}H_{43}N_3O_5Si_4Na$ $[M+Na]^+$ 516.2178, found 516.2175.



20

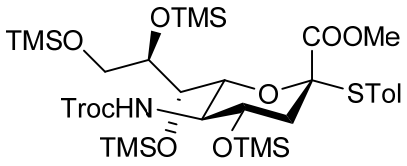
2-Azido-2-deoxy-1,3,4,6-tetra-*O*-trimethylsilyl-α-D-mannopyranose (20). Procedures were as shown for compound **16** to afford **20** as a colourless syrup (95 mg, 90%). $[\alpha]^{25}_{D}$ 34.4 (*c* 0.5, $CHCl_3$); IR ($CHCl_3$) ν 2956, 2922, 2103, 1249, 1148, 1049, 837 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 4.99 (s, 1H, H-1), 4.06 (dd, *J* = 3.6, 8.7 Hz, 1H, H-3), 3.78-3.59 (m, 5H, H-2, H-4, H-5, H-6a, H-6b), 0.16 (s, 9H, TMS), 0.14 (s, 9H, TMS), 0.13 (s, 9H, TMS), 0.08 (s, 9H, TMS); ^{13}C NMR (100 MHz, $CDCl_3$) δ 93.3 (CH), 74.0 (CH), 72.9 (CH), 69.0 (CH), 66.3 (CH), 62.3 (CH_2), 0.8 (CH_3), 0.5 (CH_3), 0.07 (CH_3), -0.1 (CH_3); HRMS (ESI) calcd for $C_{18}H_{43}N_3O_5Si_4Na$ $[M+Na]^+$ 516.2178, found 516.2175.



22

Methyl (4-methylphenyl 5-amino-3,5-dideoxy-2-thio-4,7,8,9-tetra-*O*-trimethylsilyl-D-glycero-α-D-galacto-2-nonulopyranoside)onate (22). To a suspension of **21**[1] (100 mg) in

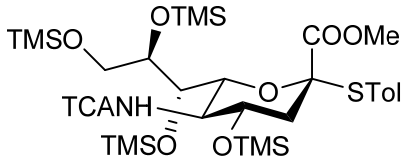
CH_3CN (1 mL) was added HMDS (130 μL, 2.5 eq) at rt under N_2 atmosphere, and the reaction was monitored by TLC or ^1H NMR. After 3 h, the mixture was evaporated *in vacuo* at rt to furnish the desired product **22** as a colorless syrup quantitatively. $[\alpha]^{26}_D$ 2.3 (*c* 0.5, $CHCl_3$); IR ($CHCl_3$) ν 2953, 1748, 1731, 1249, 1128, 1087, 837 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.39 (d, *J* = 8.2 Hz, 2H, Ar), 7.05 (d, *J* = 8.0 Hz, 2H, Ar), 4.06 (dd, *J* = 10.9, 1.9 Hz, 1H, H-9a), 3.90-3.86 (m, 2H, H-7, H-8), 3.53 (dd, *J* = 7.9, 1.8 Hz, 1H, H-9b), 3.50 (s, 3H), 3.31-3.24 (m, 2H, H-6, H-6), 2.67 (t, *J* = 9.5 Hz, 1H, H-5), 2.54 (dd, *J* = 12.8, 4.2 Hz, 1H, H-3eq), 2.30 (s, 3H), 1.68 (t, *J* = 12.0 Hz, 1H, H-3ax), 0.13 (s, 9H, TMS), 0.11 (s, 9H, TMS), 0.09 (s, 9H, TMS), 0.06 (s, 9H, TMS); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.3 (C), 139.6 (CH), 136.5 (CH), 126.3 (CH), 87.7 (CH), 79.8 (CH), 76.1 (CH), 75.0 (CH), 73.2 (CH), 64.8 (CH_2), 53.7 (CH), 52.3 (CH), 40.9 (CH_2), 21.4 (CH), 0.8 (CH_3), 0.6 (CH_3), 0.4 (CH_3), -0.2 (CH_3); HRMS (ESI) calcd for $C_{29}H_{58}NO_7Si_4S$ $[M+H]^+$ 676.3011, found 676.3015.



23

Methyl (4-methylphenyl 3,5-dideoxy-5-(2,2,2-trichloroethoxycarbonylamino)-2-thio-4,7,8,9-tetra-*O*-trimethylsilyl-D-glycero-α-D-galacto-2-nonulopyranoside)onate (23). To a suspension of **21**[1] (100 mg) in CH_3CN (1 mL) was added HMDS (130 μL, 2.5 eq) at rt under N_2 atmosphere, and the reaction was monitored by TLC or ^1H NMR. After 3 h, the mixture was added CH_2Cl_2/Py (7/3) (1 mL) and 2,2,2-trichloroethoxycarbonyl chloride (40 μL, 1.1 eq) was added at 0°C and the mixture was allowed to stir for another 2 h. The mixture was filtered through a pad of silica gel, which was washed with hexane. The filtrate was concentrated *in vacuo* to furnish **23** as a colourless syrup (184 mg, 84%). $[\alpha]^{25}_D$ 22.6 (*c* 0.5, $CHCl_3$); IR ($CHCl_3$) ν 2922, 2852, 1868, 1844, 1540, 1250, 1101 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.41 (d, *J* = 8.0 Hz, 2H, Ar), 7.08 (d, *J* = 8.0 Hz, 2H, Ar), 4.87 (d, *J* = 8.7 Hz, 1H, NH), 4.66 (d, *J* = 12.2 Hz, 1H, Troc-CH_2), 4.56 (d, *J* = 11.6 Hz, 1H, Troc-CH_2), 4.04 (dd, *J* = 10.1, 2.1 Hz, 1H, H-9a), 3.85-3.73 (m, 4H, H-4, H-6, H-7, H-8), 3.58 (s, 3H), 3.79 (dd, *J* = 10.3, 7.5 Hz, 1H, H-9b), 3.25 (q, *J* = 9.8 Hz, 1H, H-5), 2.61 (dd, *J* = 12.9, 4.3 Hz, 1H, H-3eq), 3.32 (s, 3H), 1.73 (dd, *J* = 12.5, 11.5 Hz, 1H, H-3ax), 0.14 (s, 9H, TMS), 0.12 (s, 9H, TMS), 0.06 (s, 9H, TMS), 0.06 (s, 9H, TMS); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.2 (C), 153.9 (C), 139.9 (CH_2), 136.7 (CH), 129.6 (CH), 126.1 (CH), 95.5 (CH), 87.5 (CH), 75.9 (CH), 75.8

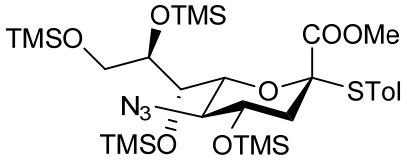
(CH), 75.8 (CH), 74.8 (CH_2), 74.6 (CH), 69.3 (CH), 64.5 (CH_2), 55.6 (CH), 52.6 (CH), 41.9 (CH_2), 29.9 (CH), 21.5 (CH_3), 0.8 (CH_3), 0.7(CH_3), 0.2 (CH_3), 0.1 (CH_3); HRMS (ESI) calcd for $C_{32}H_{58}NO_9Si_4NaSCl_3$ $[M+Na]^+$ 872.1873, found 872.1877.



24

Methyl (4-methylphenyl 3,5-dideoxy-2-thio-5-trichloroacetamido-4,7,8,9-tetra-*O*-trimethylsilyl-D-glycero-α-D-galacto-2-nonulopyranoside)onate (24).

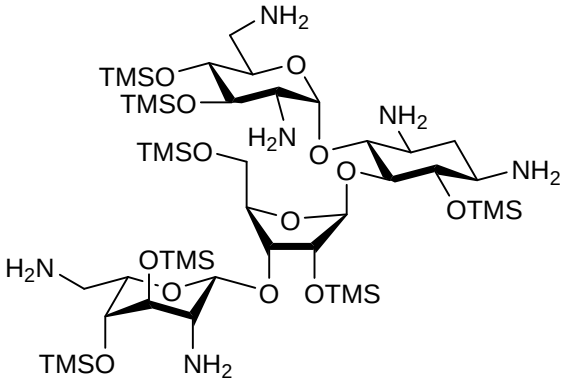
To a suspension of **21**[1] (100 mg) in CH_3CN (1 mL) was added HMDS (130 μL, 2.5 eq) at rt under N_2 atmosphere, and the reaction was monitored by TLC or ^1H NMR. After 3 h, the mixture was added CH_2Cl_2/Py (7/3) (1 mL) and trichloro acetyl chloride (31 μL, 1.1 eq) was added at 0°C and allowed to stir for 2 h. The mixture was filtered through a pad of silica gel, which was washed with hexane. The filtrate was concentrated *in vacuo* to furnish **24** as a yellowish syrup (195 mg, 92%). $[\alpha]^{25}_D$ 15.8 (*c* 0.5, $CHCl_3$); IR ($CHCl_3$) ν 2917, 2850, 1567, 1557, 1497, 1042 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.42 (d, *J* = 8.0 Hz, 2H, Ar), 7.09 (d, *J* = 8.0 Hz, 2H, Ar), 6.66 (d, *J* = 8.1 Hz, 1H, NH), 4.03 (dd, *J* = 10.5, 3.2 Hz, 1H, H-9a), 3.92-3.86 (m, 2H, H-4, H-6), 3.82 (dt, *J* = 6.9, 3.4 Hz, 1H, H-7), 3.75 (dd, *J* = 3.2, 2.3 Hz, 1H, H-8), 3.61 (s, 3H), 3.47 (dt, *J* = 16.2, 9.0 Hz, 2H, H-5, H-9b), 2.65 (dd, *J* = 12.8, 4.4 Hz, 1H, H-3eq), 2.33 (s, 3H), 1.73 (dd, *J* = 12.9, 11.3 Hz, 1H, H-3ax), 0.14 (s, 9H, TMS), 0.11 (s, 9H, TMS), 0.07 (s, 9H, TMS), 0.06 (s, 9H, TMS); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.2 (C), 161.4 (C), 140.0 (CH_2), 136.7 (CH), 129.7 (CH), 125.9 (CH), 92.9 (CH), 87.5 (CH), 75.6 (CH), 75.4 (CH), 75.3 (CH), 68.9 (CH), 64.1 (CH_2), 55.8 (CH), 52.7 (CH), 41.8 (CH_2), 21.5 (CH_3), 21.2 (CH_3), 0.8 (CH_3), 0.7(CH_3), 0.4 (CH_3), 0.06 (CH_3), -0.2 (CH_3); HRMS (ESI) calcd for $C_{31}H_{56}NO_8Si_4NaSCl_3$ $[M+Na]^+$ 842.1767, found 842.1761.



25

Methyl (4-methylphenyl 5-azido-2-thio-4,7,8,9-tetra-*O*-trimethylsilyl-3,5-dideoxy-D-

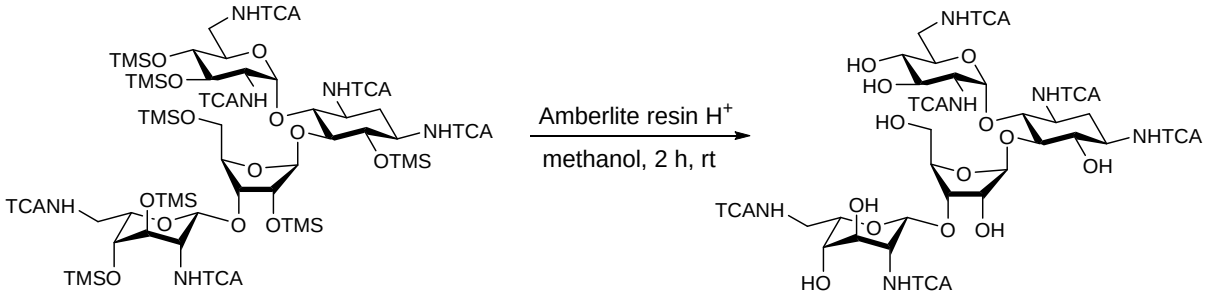
glycero-α-D-galacto-2-nonulopyranoside)onate (25). To a suspension of **21**[1] (100 mg) in CH_3CN (1 mL) was added HMDS (130 μL, 2.5 eq) at rt under N_2 atmosphere, and the reaction was monitored by TLC or ^1H NMR. After 3 h, the mixture was added 4-dimethylaminopyridine (94 mg, 3 eq) and a solution of TfN_3 (1.1 eq) in DCM (3 mL) at 0 °C. The ice-bath was then removed, and the reaction was kept stirring at room temperature for another 12 h. The mixture was filtered through a pad of silica gel, which was washed with hexane. The filtrate was concentrated *in vacuo* to furnish **25** (164 mg, 91%) as colorless syrup. $[\alpha]^{25}_D$ 13.2 (*c* 0.5, $CHCl_3$); IR ($CHCl_3$) 2924, 2112, 1733, 1507, 1456, 1037 cm^{-1} ; ^1H NMR (400 MHz, $CDCl_3$) δ 7.40 (d, *J* = 8.0 Hz, 2H, Ar), 7.08 (d, *J* = 8.0 Hz, 2H, Ar), 4.09 (dd, *J* = 10.2, 2.0 Hz, 1H, H-9a), 3.85-3.80 (m, 2H, H-6, H-8), 3.61 (ddd, *J* = 14.9, 7.8, 5.7 Hz, 1H, H-4), 3.55 (s, 3H), 3.49 (dd, *J* = 10.7, 8.1 Hz, 1H, H-9b), 3.27-3.23 (m, 2H, H-5, H-7), 2.59 (dd, *J* = 12.9, 4.6 Hz, 1H, H-3eq), 2.32 (s, 3H), 1.73 (dd, *J* = 12.9, 11.5 Hz, 1H, H-3ax), 0.18 (s, 9H, TMS), 0.14 (s, 9H, TMS), 0.13 (s, 9H, TMS), 0.05 (s, 9H, TMS); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.9 (C), 140.0 (CH), 136.7 (CH), 129.7 (CH), 126.0 (CH), 87.6 (CH), 76.7 (CH), 75.5 (CH), 75.1 (CH), 71.9 (CH), 64.9 (CH_2), 62.8 (CH), 52.6 (CH), 41.2 (CH_2), 21.5 (CH), 0.81 (CH_3), 0.13 (CH_3), -0.10 (CH_3); HRMS (ESI) calcd for $C_{29}H_{55}N_3O_7Si_4SNa$ $[M+Na]^+$ 724.2736, found 724.2729.



27

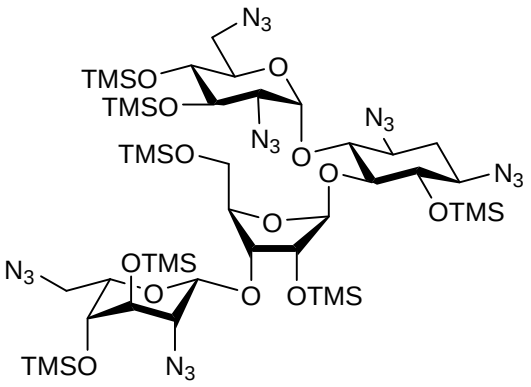
Compound (27). To a suspension of commercially available neomycine sulfate **26** (100 mg) was added HMDS (160 μL, 7 eq) in CH_3CN (2 mL) at rt under N_2 atmosphere and the reaction was monitored by TLC or ^1H NMR. After 36 h, the mixture was filtered and the filtrate was evaporated *in vacuo* at rt to furnish the desired product **27** as a colorless solid form (101 mg, 82%). $[\alpha]^{25}_D$ 35.1 (*c* 0.5, $CHCl_3$); IR ($CHCl_3$) ν 3361, 3298, 2955, 2923, 1635, 1593, 1252, 1020, 842 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 5.38 (d, *J* = 3.7 Hz, 1H), 5.25 (d, *J* = 3.7 Hz, 1H), 4.76 (d, *J* = 1.3 Hz, 1H), 4.16 (t, *J* = 4.6 Hz, 1H), 4.00-3.95 (m, 2H), 3.86 (t, *J* = 2.9 Hz, 1H), 3.71 (t, *J* = 3.87 Hz, 2H), 3.67-3.61 (m, 3H), 3.51-3.44 (m, 2H), 3.39-3.37

(m, 1H), 3.29-3.19 (m, 3H), 2.96-2.91 (m, 2H), 2.82 (s, 1H), 2.70-2.55 (m, 5H), 1.87-1.76 (m, 2H), 1.21-1.12 (m, 2H), 0.18 (s, 9H, TMS), 0.15 (s, 9H, TMS), 0.11 (s, 18H, 2 x TMS), 0.10 (s, 9H, TMS), 0.08 (s, 9H, TMS), 0.07 (s, 9H, TMS); ^{13}C NMR (100 MHz, $CDCl_3$) δ 107.2 (CH), 99.9 (CH), 99.4 (CH), 83.8 (CH), 81.8 (CH), 81.4 (CH), 80.6 (CH), 76.5 (CH), 76.3 (CH), 75.1 (CH), 74.3 (CH), 74.1 (CH), 72.7 (CH), 71.0 (CH), 62.6 (CH_2), 57.4 (CH), 55.0 (CH), 52.2 (CH), 51.4 (CH), 43.5 (CH_2), 43.3 (CH_2), 38.4 (CH_2), 1.5 (2 x CH_3), 1.2 (CH_3), 0.4 (CH_3), 0.1 (CH_3), -0.1 (CH_3); HRMS (ESI) calcd for $C_{44}H_{103}N_6O_{13}Si_7$ $[M+H]^+$ 1119.5968, found 1119.5986.



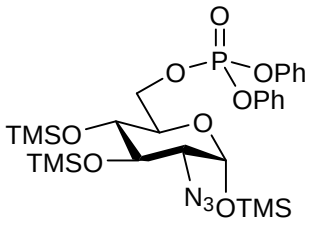
28 **28a**

Compound (28). To a solution of compound **27** (100 mg) in DCM/Pyr (7/3) (1 mL) was added trichloroacetyl chloride (70 μL, 7.0 eq) at 0 °C and the mixture was allowed to stir for 2 h. The mixture was filtered through a pad of silica gel, which was washed with hexane/EtOAc (2/1). The filtrate was concentrated *in vacuo* to furnish **28** as a colourless syrup (151 mg, 85%). *Note*: Because of the poor stability of the TMS groups of **28**, the TMS groups were further removed to collect the data by the treatment with Amberlite resin H^+ in methanol. Compound **28a** was furnished quantitatively. $[\alpha]^{25}_D$ 11.4 (*c* 0.5, MeOH); IR ($CHCl_3$) ν 3328, 2924, 1697, 1525, 1039, 822 cm^{-1}; ^{1}H NMR (400 MHz, MeOD) δ 8.81 (t, *J* = 5.5 Hz, NH), 8.73 (d, *J* = 9.3 Hz, NH), 8.73 (d, *J* = 8.2 Hz, NH), 8.66 (d, *J* = 7.5 Hz, NH), 8.62 (d, *J* = 9.3 Hz, NH), 8.44 (bs, NH), 8.14-8.12 (m, NH), 5.47 (s, 1H), 5.16 (d, *J* = 3.0 Hz, 1H), 5.04 (d, *J* = 1.0 Hz, 1H), 4.22 (t, *J* = 5.6 Hz, 1H), 4.09-4.01 (m, 4H), 3.93-3.79 (m, 9H), 3.73-3.62 (m, 6H), 3.59-3.48 (m, 9H), 1.91 (dt, *J* = 12.8, 3.7 Hz, 1H), 1.68 (q, *J* = 12.5 Hz, 1H); ^{13}C NMR (100 MHz, CD_3OD) δ 162.6 (C), 162.5 (C), 162.3 (C), 162.2 (C), 162.0 (C), 161.4 (C), 108.1 (CH), 97.0 (CH), 95.0 (CH), 91.9 (CH), 91.9 (CH), 91.8 (CH), 85.2 (CH), 81.5 (CH), 74.5 (CH), 73.5 (CH), 71.8 (CH), 71.1 (CH), 69.5 (CH), 69.3 (CH), 68.7 (CH), 67.5 (CH), 60.8 (CH_2), 55.2 (CH), 52.0 (CH), 51.0 (CH), 50.6 (CH), 41.3 (CH_2), 41.1 (CH_2), 30.2 (CH_2); HRMS (ESI) calcd for $C_{35}H_{41}N_6O_{19}Cl_{18}$ $[M+H]^+$ 1478.6820, found 1478.6780.



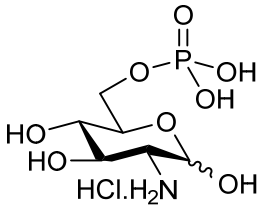
29

Compound (29). Trifluoromethanesulfonic anhydride (148 μL, 10 eq) was slowly added drop wise from an addition funnel to a solution of sodium azide (335 mg, 58 eq) in a mixture of water (1.5 mL) and CH_2Cl_2 (3 mL) at 0°C. After stirring at the same temperature for 2 h, the organic layer was separated, and the aqueous layer was extracted with CH_2Cl_2 (2 x 3 mL). The combined organic layers were neutralized with saturated $NaHCO_3$(aq). The generated trifluoromethanesulfonic azide (TfN_3) was directly used without further purification for the ensuing reaction. 4-Dimethylaminopyridine (200 mg, 18 eq) was added to a solution of the per-*O*-trimethylsilylated compound **27** (100 mg) in CH_2Cl_2 (1mL) and the TfN_3 solution in CH_2Cl_2 (9 mL) was added at 0 °C. The ice-bath was removed and the reaction was kept stirring at room temperature for another 12 h. The mixture was filtered through a pad of silica gel, which was washed with hexane. The filtrate was concentrated *in vacuo* to furnish **29** (103 mg, 91%) as a colorless syrup. $[\alpha]^{25}_D$ 81.8 (*c* 0.5, $CHCl_3$); IR ($CHCl_3$) ν 2954, 2922, 2101, 1251, 1148, 1089, 876 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 5.82 (d, *J* = 3.4 Hz, 1H), 5.54 (d, *J* = 7.0 Hz, 1H), 5.17 (s, 1H), 4.20 (d, *J* = 5.3 Hz, 1H), 4.09 (dd, *J* = 7.1, 14.5 Hz, 1H), 3.99 (m, 2H), 3.89 (dd, *J* = 4.1, 7.6 Hz, 1H), 3.82-3.76 (m, 3H), 3.68-3.52 (m, 6H), 3.42-3.36 (m, 5H), 3.31-3.18 (m, 5H), 3.02 (dd, *J* = 4.5, 12.7 Hz, 1H), 2.30 (dt, *J* = 4.4, 17.4 Hz, 1H), 0.22 (s, 9H, TMS), 0.14 (s, 18H, TMS), 0.13 (s, 9H, TMS), 0.12 (s, 9H, TMS), 0.11 (s, 9H, TMS); ^{13}C NMR (100 MHz, $CDCl_3$) δ 98.3 (CH), 83.7 (CH), 80.5 (CH), 74.4 (CH), 73.5 (CH), 72.6 (CH), 71.9 (CH), 69.1 (CH), 62.2 (CH), 61.2 (CH), 60.6 (CH), 59.6 (CH), 56.3 (CH), 51.5 (CH_2), 32.9 (CH_2), 31.8 (CH_2), 29.9 (CH_2), 29.2 (CH_2), 1.2 (CH_3), 1.1 (CH_3), 0.9 (CH_3), 0.3 (CH_3), 0.2 (CH_3), 0.1 (CH_3); HRMS (ESI) calcd for $C_{44}H_{90}N_{18}O_{13}Si_7Na$ $[M+Na]^+$ 1297.5217, found 1297.5211.



30

2-Azido-6-*O*-diphenylphosphoryl-2-deoxy-1,3,4-tri-*O*-trimethylsilyl-α-D-glucopyranose (30). Trifluoromethanesulfonic anhydride (194 μL, 1.2 eq) was added drop wise from an addition funnel to a solution of sodium azide (254 mg, 4 eq) in a mixture of water (2 mL) and CH_2Cl_2 (1.3 mL) at 0 ºC. After stirring at the same temperature for 2 h, the organic layer was separated, and the aqueous layer was extracted with CH_2Cl_2 (2 x 2 mL). The combined organic layers were neutralized with saturated $NaHCO_{3(aq)}$. The generated trifluoromethanesulfonic azide (TfN_3) was directly used without further purification for the ensuing reaction. 4-Dimethylaminopyridine (359 mg, 3 eq) was added to a CH_2Cl_2 solution of **2** (454 mg) and the previously produced TfN_3 solution in CH_2Cl_2 was added at 0 ºC. The ice-bath was then removed and the reaction was kept stirring at room temperatur for another12 h. Upon completion, pyridine (10 mL) and diphenylchlorophosphate (624 μL, 3.0 eq) were added with stirring at 0 ºC. The mixture was allowed to warm to rt and kept stirring for 6 h. The mixture was concentrated and purified using flash column chromatography on silica gel (Hex/EtOAc 20:1) to yield **30** (499 mg, 78%). $[\alpha]^{25}_D$ 59.8 (*c* 0.5, $CHCl_3$); IR ($CHCl_3$) ν 2957, 2108, 1591, 1489, 1293, 1071, 1026, 844 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.31 (t, *J* = 7.8 Hz, 4H, Ar), 4.21 (d, *J* = 8.1 Hz, 4H, Ar), 7.16 (t, *J* = 7.3 Hz, 2H, Ar), 5.06 (d, *J* = 3.0 Hz, 1H, H-1), 4.44 (td, *J* = 9.2, 1.8 Hz, 1H, H-6a), 4.29 (ddd, *J* = 11.5, 7.4, 3.8 Hz, 1H, H-6b), 3.85-3.81 (m, 2H, H-3, H-5), 3.49 (t, *J* = 9.0 Hz, 1H, H-4), 2.76 (dd, *J* = 9.8, 3.0 Hz, 1H, H-2), 0.19 (s, 9H, TMS), 0.14 (s, 9H, TMS), 0.12 (s, 9H, TMS); ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.04 (C), 150.97 (C), 150.85 (C), 150.78 (C), 129.94 (CH), 129.88 (CH), 125.49 (CH), 125.44 (CH), 120.30 (CH), 120.26 (CH), 92.9 (CH), 73.0 (CH), 71.1 (CH), 70.60 (CH), 70.54 (CH), 67.98 (CH_2), 67.93 (CH_2), 65.0 (CH), 1.0 (CH_3), 0.9 (CH_3), -0.03 (CH_3); ^{31}P NMR (162 MHz, $CDCl_3$) δ -12.04; HRMS (ESI) calcd for $C_{27}H_{44}O_8Si_3P$ $[M+Na]^+$ 676.2072, found 676.2065.



31

Glucosamine 6-phosphate (31). To a solution of compound **30** (100 mg) in 75% EtOH (5 mL) was added $Pd(OH)_2$ (300 mg) and the mixture was stirred overnight under H_2 atmosphere. Palladium hydroxide was removed by filtering through a pad of celite, and the amine group was neutralized with 1M HCl (240 μL, 1 eq). The mixture was stirred for 2 h, and in the same pot, PtO_2 (72 mg, 2 eq) was added. After stirring for another10 h at rt under H_2 atmosphere, the mixture was filtered through a celite pad. The filtrate was evaporated *in vacuo* at rt, and the residue was washed with EtOAc and extracted with water. The water layer was lyophilized to give glucosamine 6-phosphate (**31**) as a white powder (44 mg, 93%) without further purification. The physical data are in agreement with the literature.[2] $[\alpha]^{22}_D$ 275.5 (c 1.68, H_2O); ^{1}H NMR (400 MHz, D_2O) δ 5.40 (d, *J* = 3.5 Hz, 1H, $1H_\alpha$), 4.92 (d, *J* = 8.4 Hz, 1H, $1H_\beta$), 4.05-4.18 9 (m, 4H, H-6a$_\alpha$, H-6b$_\alpha$, H-6a$_\beta$, H-6b$_\beta$), 3.96 (d, *J* = 10.0 Hz, 1H, H5$_\beta$), 3.85 (t, *J* = 9.72 Hz, 2H, H3$_\alpha$, H5α), 3.59-3.74 (m, 2H, H3$_\alpha$, H3$_\beta$), 3.49-3.53 (td, *J* = 2.59 Hz, 9.54 Hz, 2H, H4$_\alpha$, H4$_\beta$), 3.64 (dd, *J* = 3.59 Hz, 10.6 Hz, 1H, H2$_\alpha$), 2.98 (dd, 1H, *J* = 8.58, 10.87 Hz, 1H, H2$_\beta$); ^{13}C NMR (100 MHz, D_2O) δ 92.8 (CH), 89.2 (CH), 74.7 (CH), 74.6 (CH), 71.8 (CH), 70.4 (CH), 70.3 (CH), 69.4 (CH), 69.2 (CH), 69.1 (CH), 67.1 (CH_2), 64.5 (CH_2),64.5 (CH_2), 64.4 (CH_2), 56.6 (CH), 54.2 (CH); ^{31}P NMR (162 MHz, D_2O) δ 1.52, 1.05; HRMS (ESI) calcd for $C_6H_{14}NO_8PCl$ $[M-H]^-$ 294.0146, found 294.0139.

Reference

1. K.-C. Lu, S.-Y. Tseng and C.-C. Lin, *Carbohydr. Res.*, 2002, **337**, 755–760.
2. J.- K. Park, L.-X. Wang and S. Roseman. *J. Biol. Chem.* 2002, **277**, 15573–15578.

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PROCNO 1

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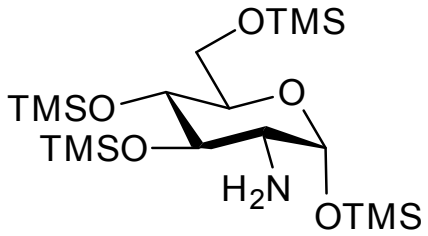

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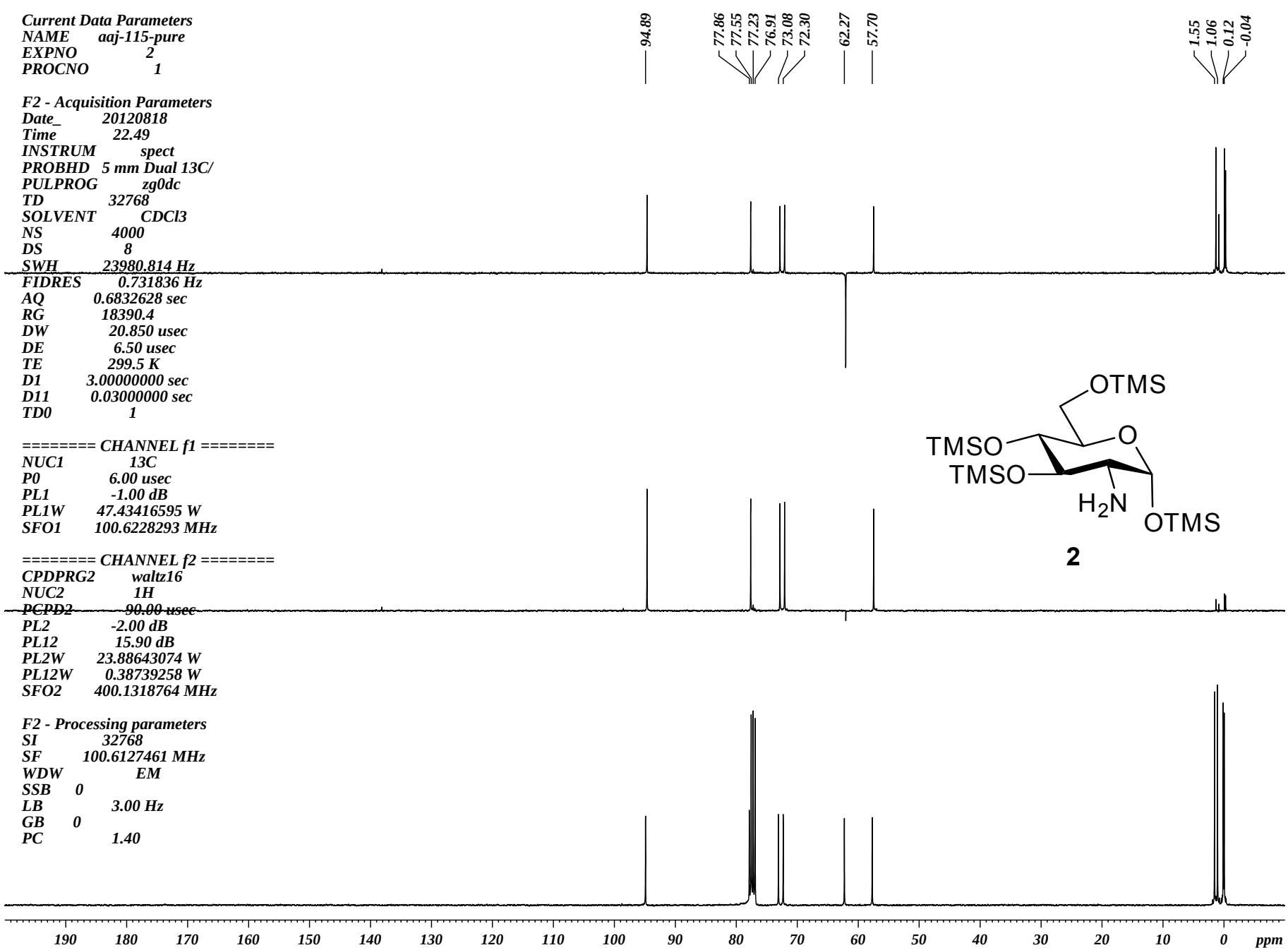

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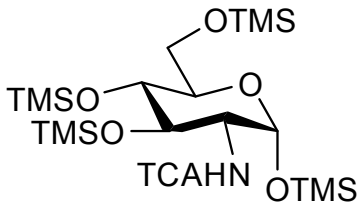

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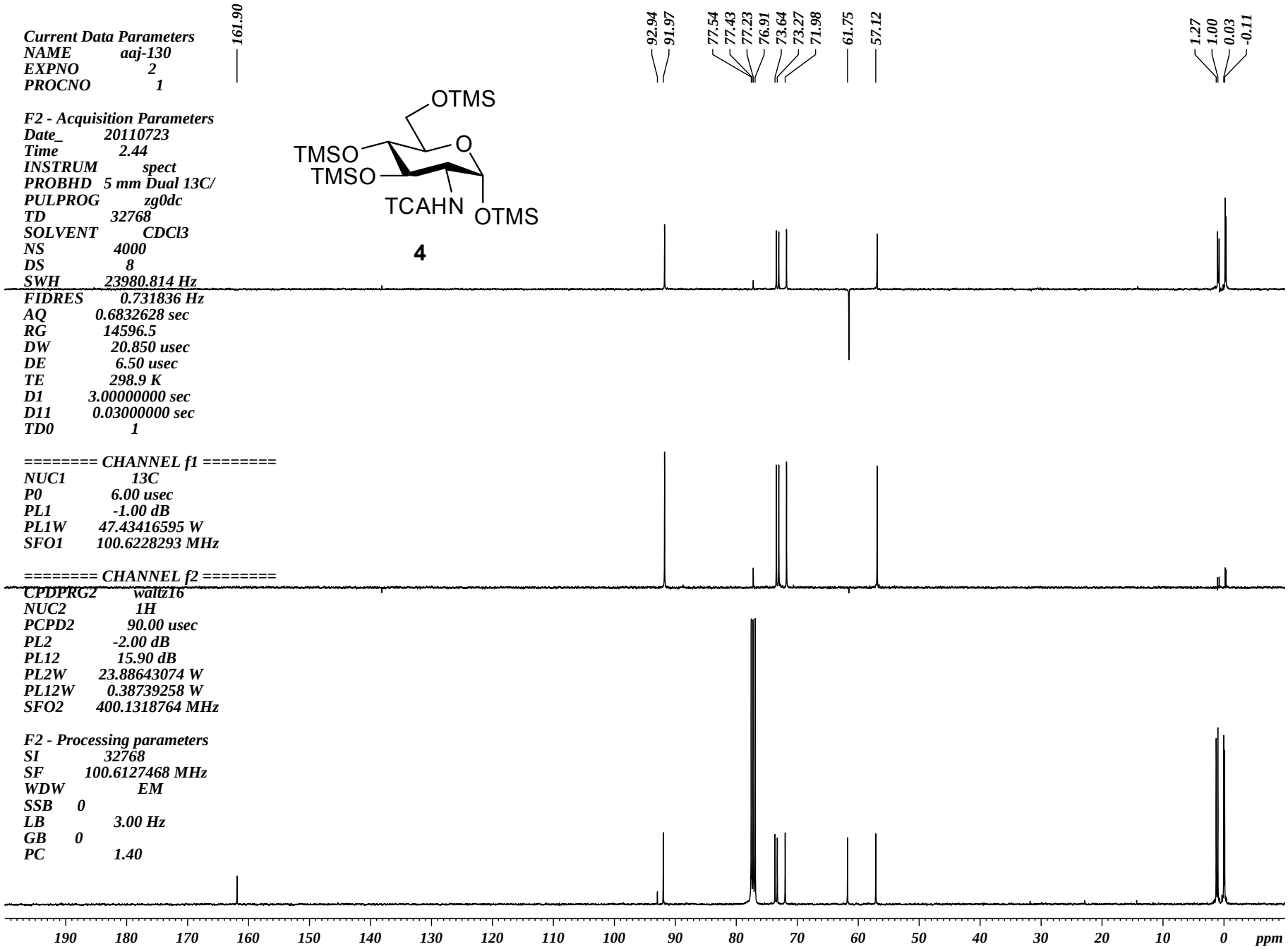
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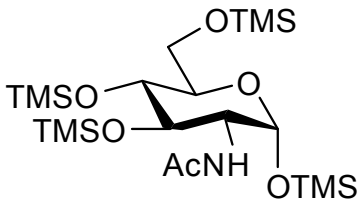

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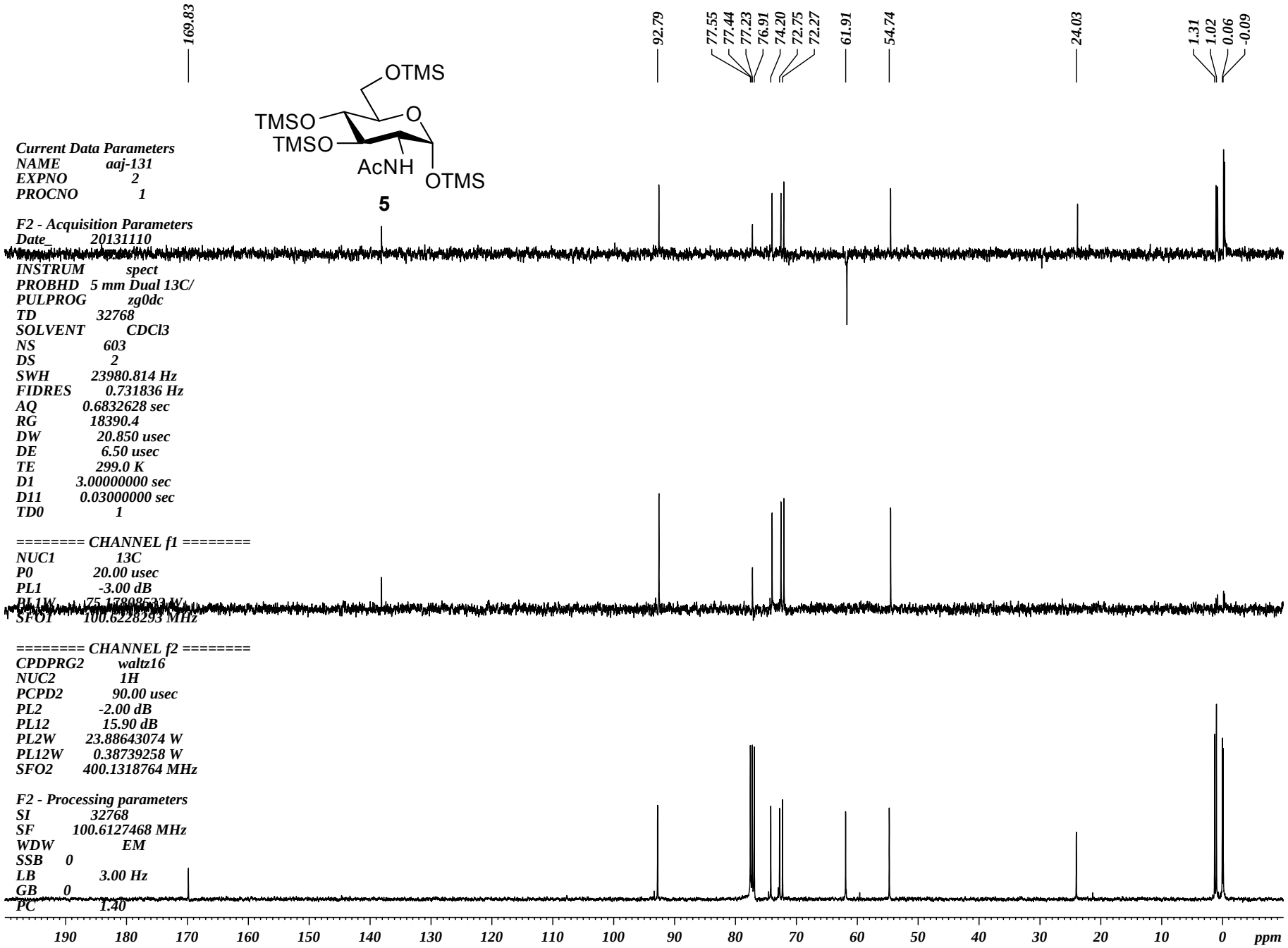
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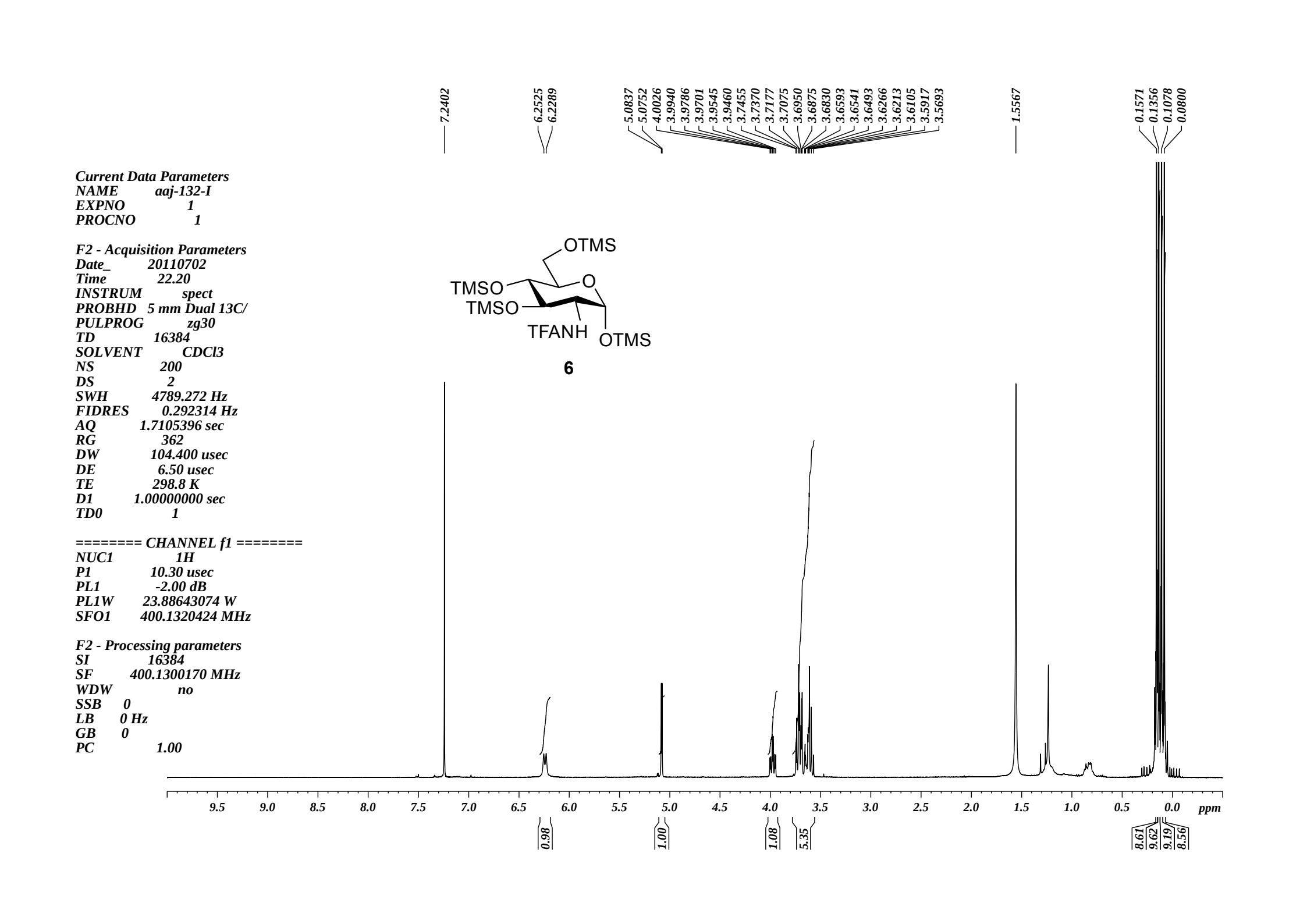
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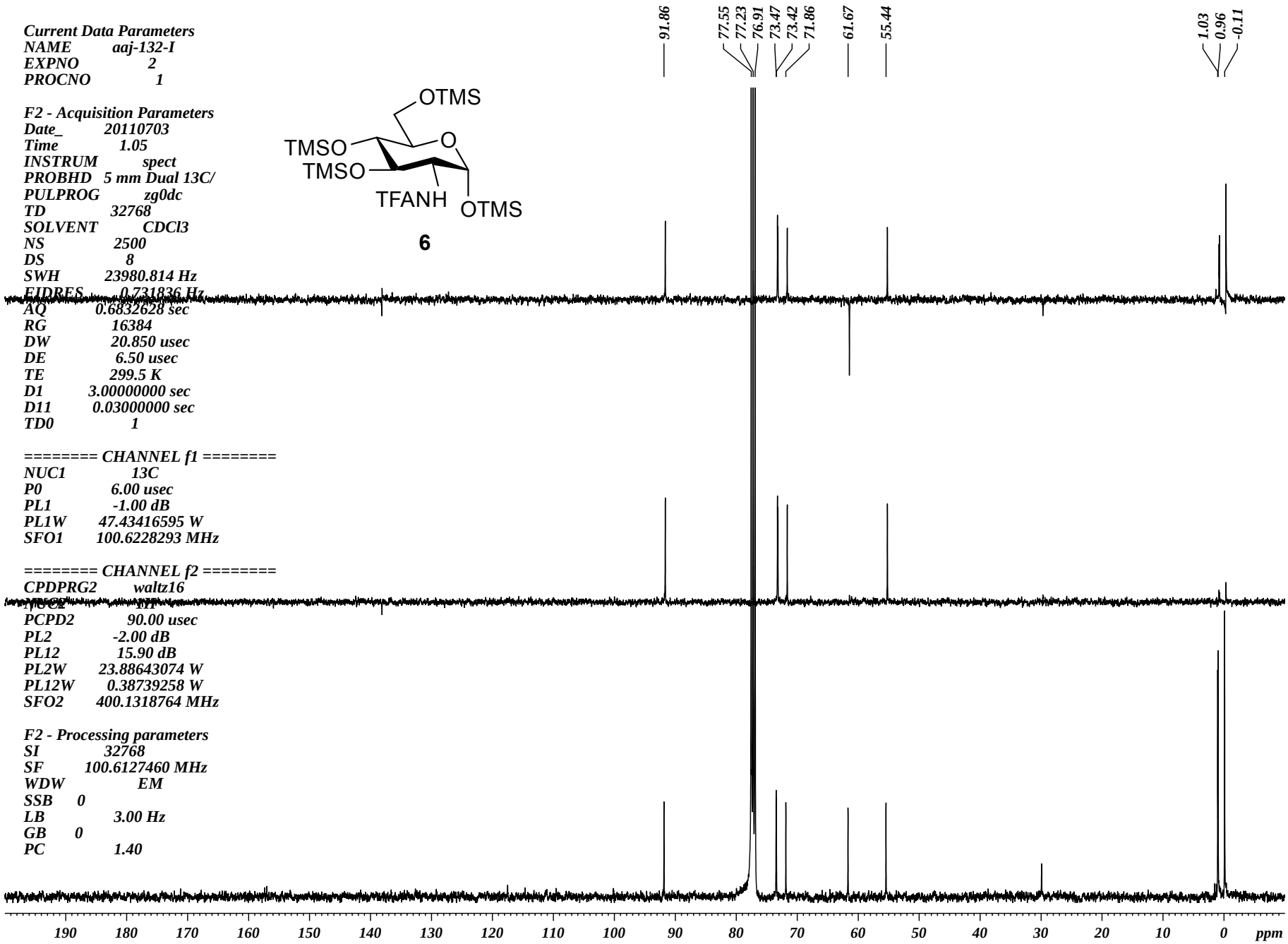
6
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.98
1.00
1.08
5.35
8.61
9.62
9.19
8.56

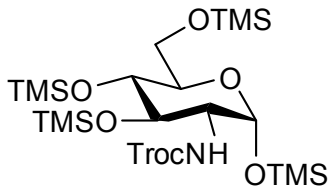

Current Data Parameters
NAME aaj-132-I
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110703
Time 1.05
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg0dc
TD 32768
SOLVENT CDCl3
NS 2500
DS 8
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 0.6832628 sec
RG 16384
DW 20.850 usec
DE 6.50 usec
TE 299.5 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P0 6.00 usec
PL1 -1.00 dB
PL1W 47.43416595 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 15.90 dB
PL2W 23.88643074 W
PL12W 0.38739258 W
SFO2 400.1318764 MHz
F2 - Processing parameters
SI 32768
SF 100.6127460 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
OTMS
TMSO
TMSO
O
TFANH
OTMS
6
91.86
77.55
77.23
76.91
73.47
73.42
71.86
61.67
55.44
1.03
0.96
-0.11
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

Current Data Parameters
NAME aaj-135
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110706
Time 22.55
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 200
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7105396 sec
RG 228.1
DW 104.400 usec
DE 6.50 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.30 usec
PL1 -2.00 dB
PL1W 23.88643074 W
SFO1 400.1320424 MHz

F2 - Processing parameters
SI 16384
SF 400.1300173 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00



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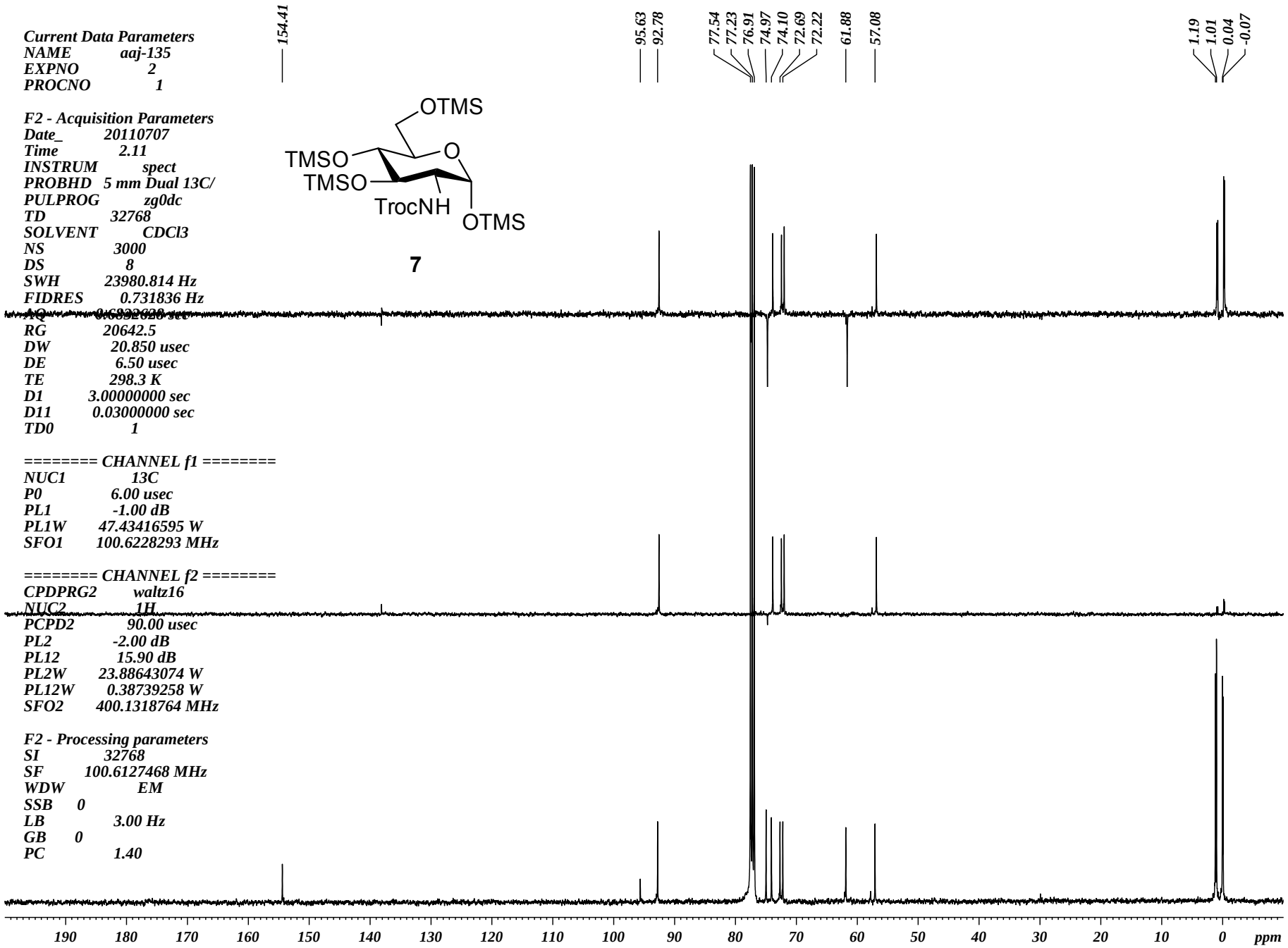
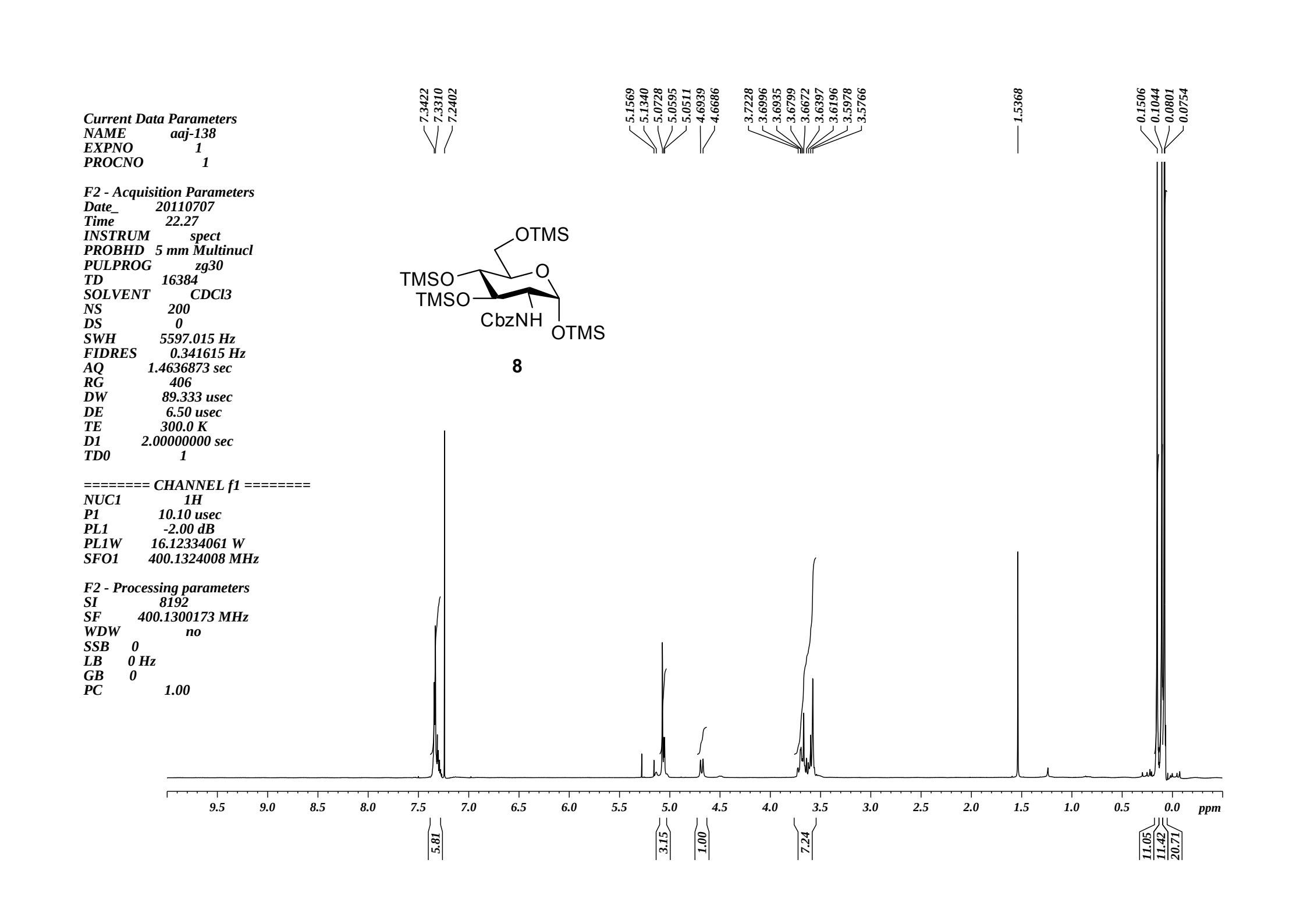
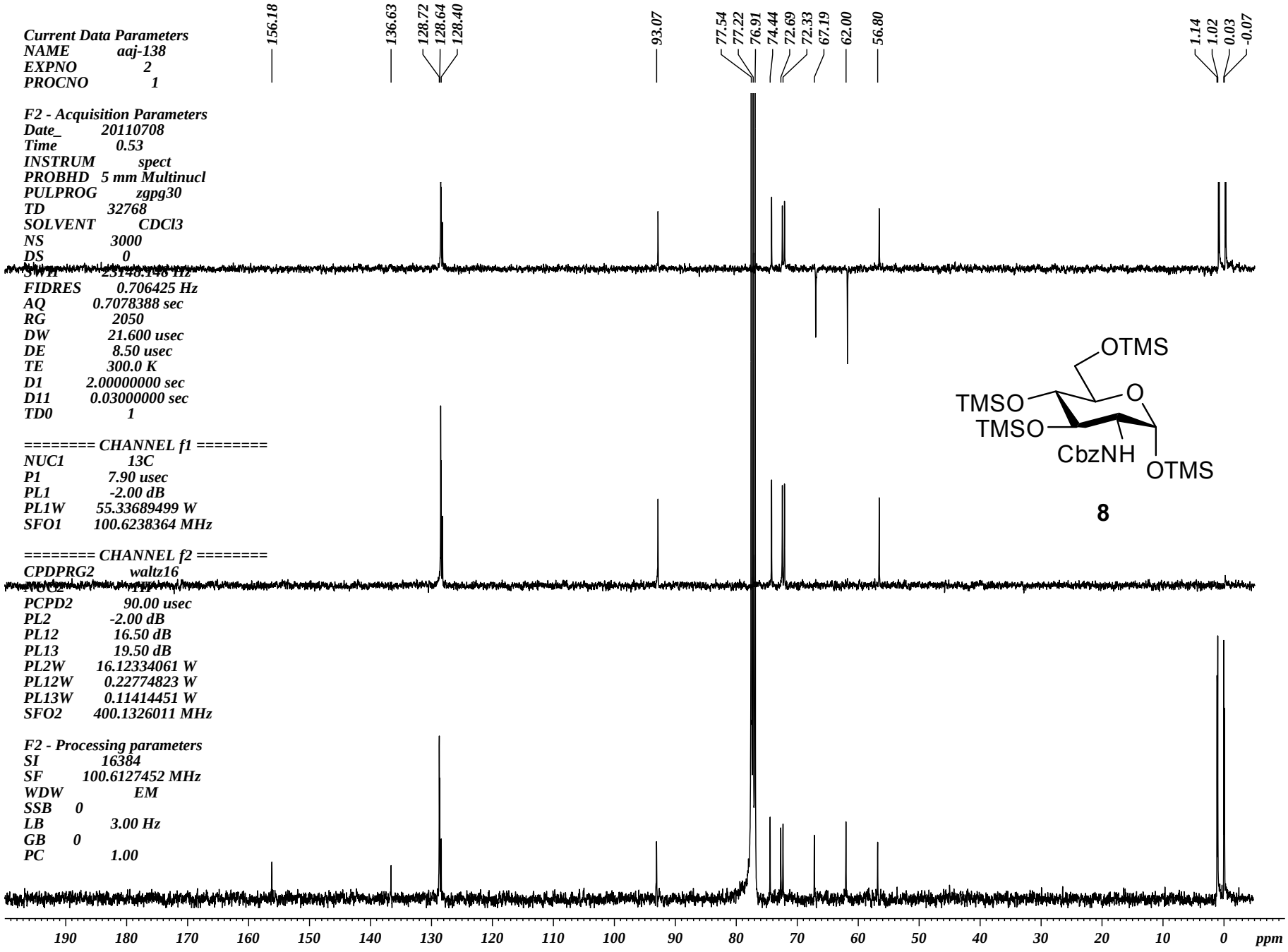
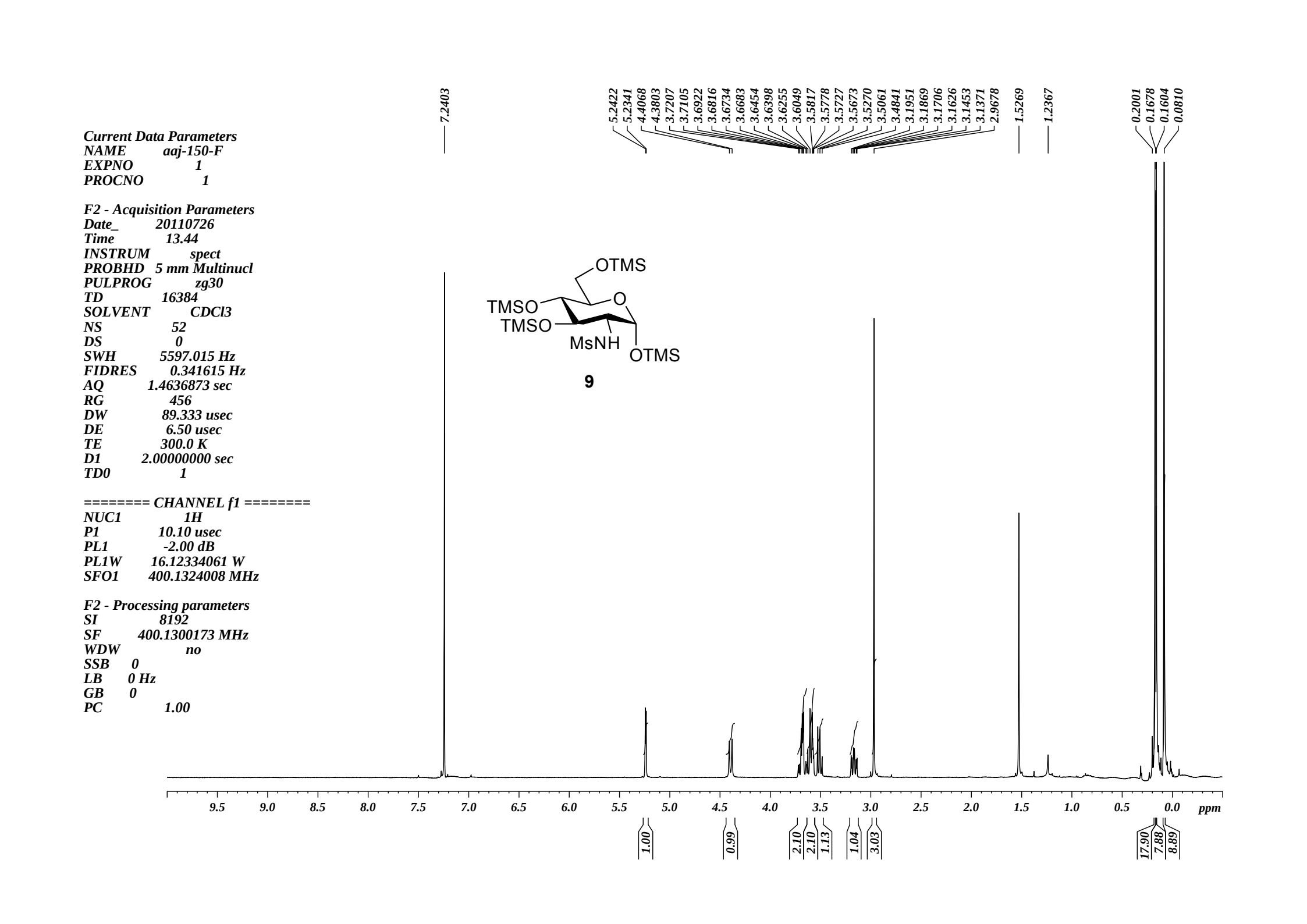
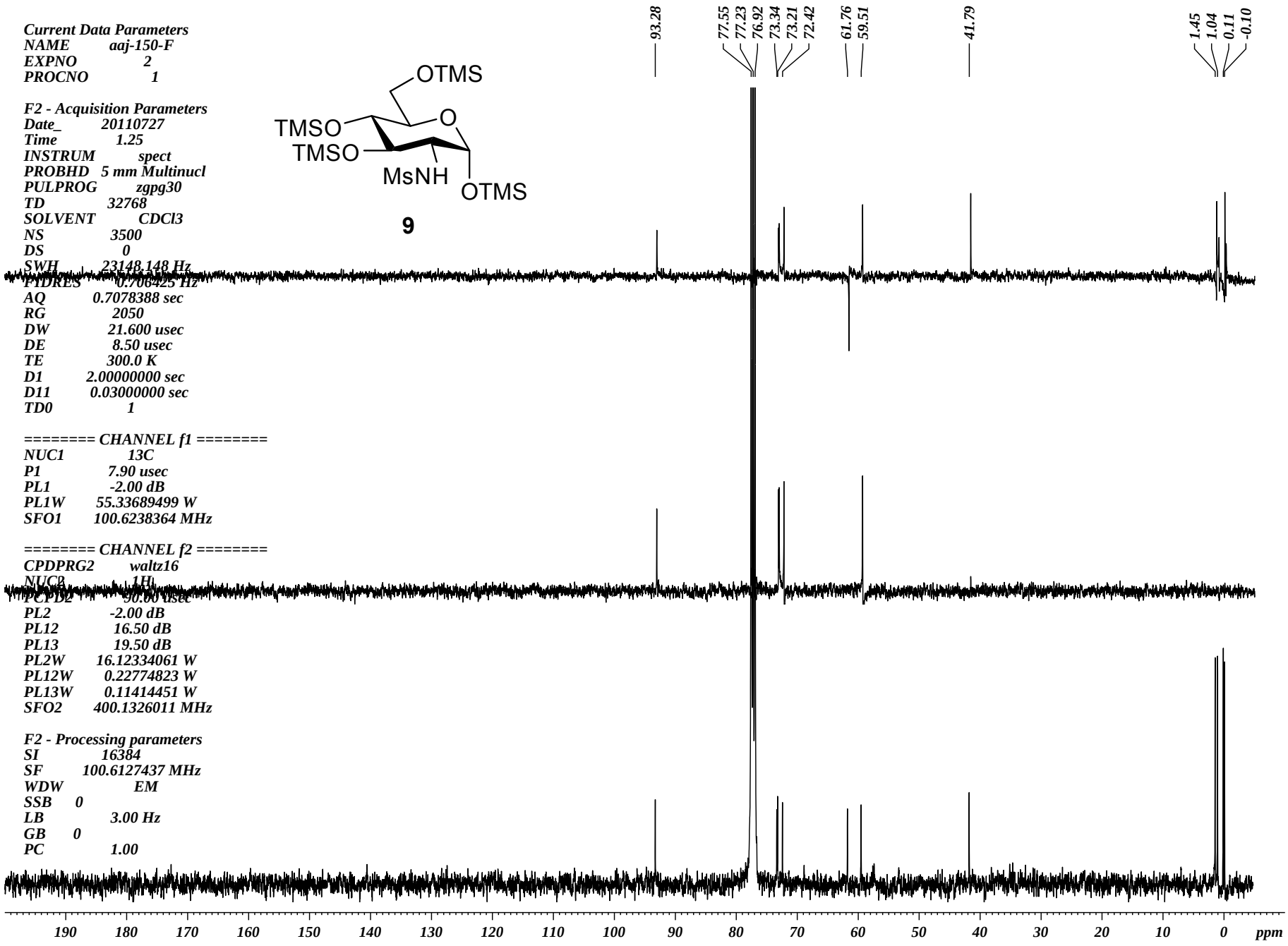

154.41
95.63
92.78
77.54
77.23
76.91
74.97
74.10
72.69
72.22
61.88
57.08
1.19
1.01
0.04
-0.07
OTMS
TMSO
O
TMSO
TrocNH
OTMS
7
Current Data Parameters
NAME aaj-135
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110707
Time 2.11
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg0dc
TD 32768
SOLVENT CDCl3
NS 3000
DS 8
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 0.6832820 sec
RG 20642.5
DW 20.850 usec
DE 6.50 usec
TE 298.3 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P0 6.00 usec
PL1 -1.00 dB
PL1W 47.43416595 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 15.90 dB
PL2W 23.88643074 W
PL12W 0.38739258 W
SFO2 400.1318764 MHz
F2 - Processing parameters
SI 32768
SF 100.6127468 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


Current Data Parameters
NAME aaj-138
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110707
Time 22.27
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 200
DS 0
SWH 5597.015 Hz
FIDRES 0.341615 Hz
AQ 1.4636873 sec
RG 406
DW 89.333 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.10 usec
PL1 -2.00 dB
PL1W 16.12334061 W
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 8192
SF 400.1300173 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
7.3422
7.3310
7.2402
5.1569
5.1340
5.0728
5.0595
5.0511
4.6939
4.6686
3.7228
3.6996
3.6935
3.6799
3.6672
3.6397
3.6196
3.5978
3.5766
1.5368
0.1506
0.1044
0.0801
0.0754
OTMS
O
TMSO
TMSO
CbzNH
OTMS
8
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
2.0
1.5
1.0
0.5
0.0
ppm
5.81
3.15
1.00
7.24
11.05
11.42
20.71


156.18
136.63
128.72
128.64
128.40
93.07
77.54
77.22
76.91
74.44
72.69
72.33
67.19
62.00
56.80
1.14
1.02
0.03
-0.07
Current Data Parameters
NAME aaj-138
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110708
Time 0.53
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 3000
DS 0
SWH 25148.148 Hz
FIDRES 0.706425 Hz
AQ 0.7078388 sec
RG 2050
DW 21.600 usec
DE 8.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.90 usec
PL1 -2.00 dB
PL1W 55.33689499 W
SFO1 100.6238364 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 16.50 dB
PL13 19.50 dB
PL2W 16.12334061 W
PL12W 0.22774823 W
PL13W 0.11414451 W
SFO2 400.1326011 MHz
F2 - Processing parameters
SI 16384
SF 100.6127452 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
OTMS
O
TMSO
TMSO
CbzNH
OTMS
8
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


Current Data Parameters
NAME aaj-150-F
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110726
Time 13.44
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 52
DS 0
SWH 5597.015 Hz
FIDRES 0.341615 Hz
AQ 1.4636873 sec
RG 456
DW 89.333 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.10 usec
PL1 -2.00 dB
PL1W 16.12334061 W
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 8192
SF 400.1300173 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
7.2403
5.2422
5.2341
4.4068
4.3803
3.7207
3.7105
3.6922
3.6816
3.6734
3.6683
3.6454
3.6398
3.6255
3.6049
3.5817
3.5778
3.5727
3.5673
3.5270
3.5061
3.4841
3.1951
3.1869
3.1706
3.1626
3.1453
3.1371
2.9678
1.5269
1.2367
0.2001
0.1678
0.1604
0.0810
OTMS
TMSO
TMSO
O
MsNH
OTMS
9
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 2.0 1.5 1.0 0.5 0.0 ppm
1.00
0.99
2.10
2.10
1.13
1.04
3.03
17.90
7.88
8.89

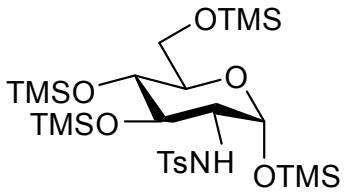

Current Data Parameters
NAME aaj-150-F
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110727
Time 1.25
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 3500
DS 0
SWH 23148.148 Hz
FIDRES 0.706425 Hz
AQ 0.7078388 sec
RG 2050
DW 21.600 usec
DE 8.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.90 usec
PL1 -2.00 dB
PL1W 55.33689499 W
SFO1 100.6238364 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 16.50 dB
PL13 19.50 dB
PL2W 16.12334061 W
PL12W 0.22774823 W
PL13W 0.11414451 W
SFO2 400.1326011 MHz
F2 - Processing parameters
SI 16384
SF 100.6127437 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
OTMS
TMSO
TMSO
O
MsNH
OTMS
9
93.28
77.55
77.23
76.92
73.34
73.21
72.42
61.76
59.51
41.79
1.45
1.04
0.11
-0.10
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

Current Data Parameters
NAME aaj-148
EXPNO 1
PROCNO 1

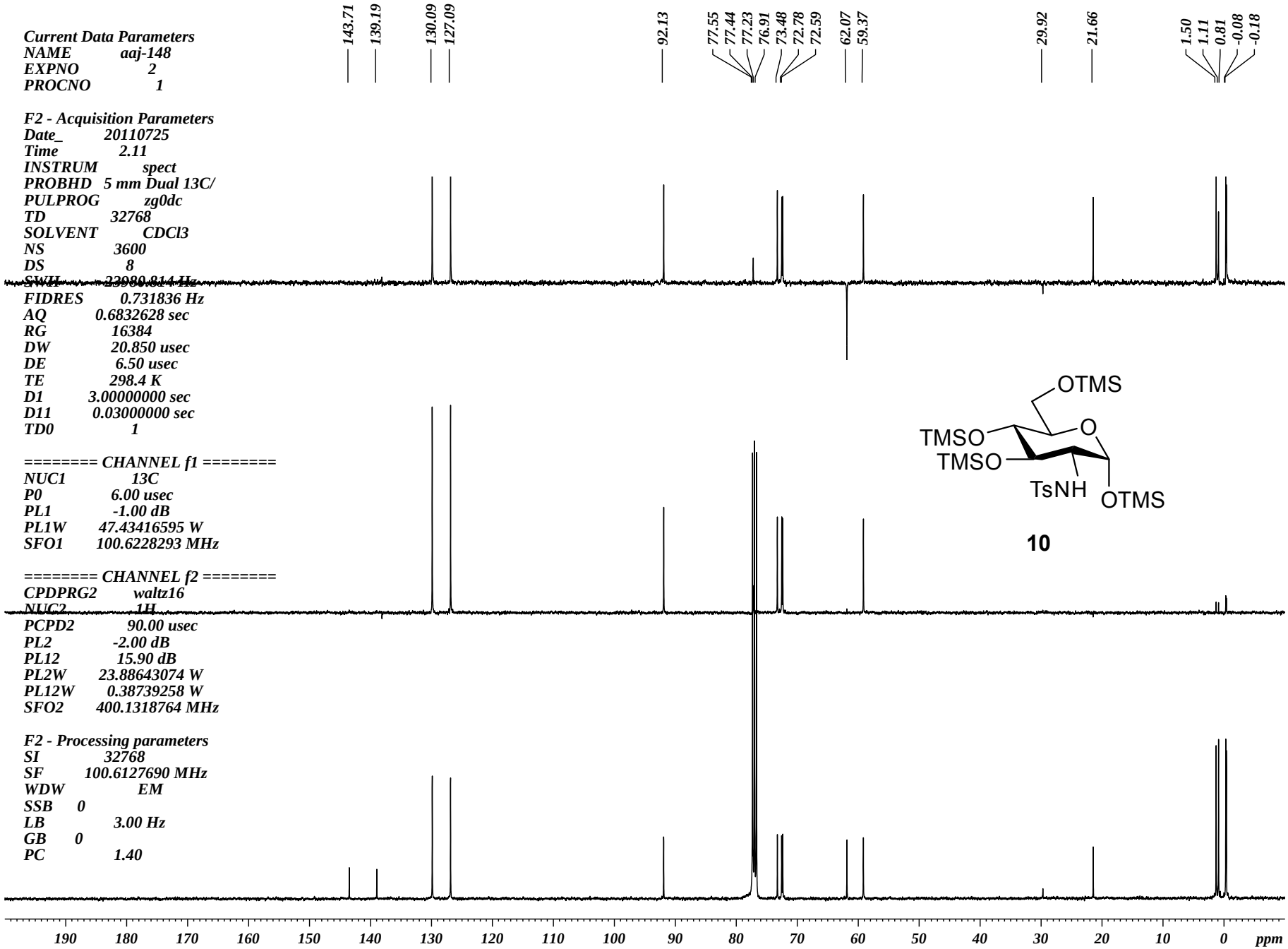
F2 - Acquisition Parameters
Date_ 20110724
Time 22.22
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 100
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7105396 sec
RG 228.1
DW 104.400 usec
DE 6.50 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1

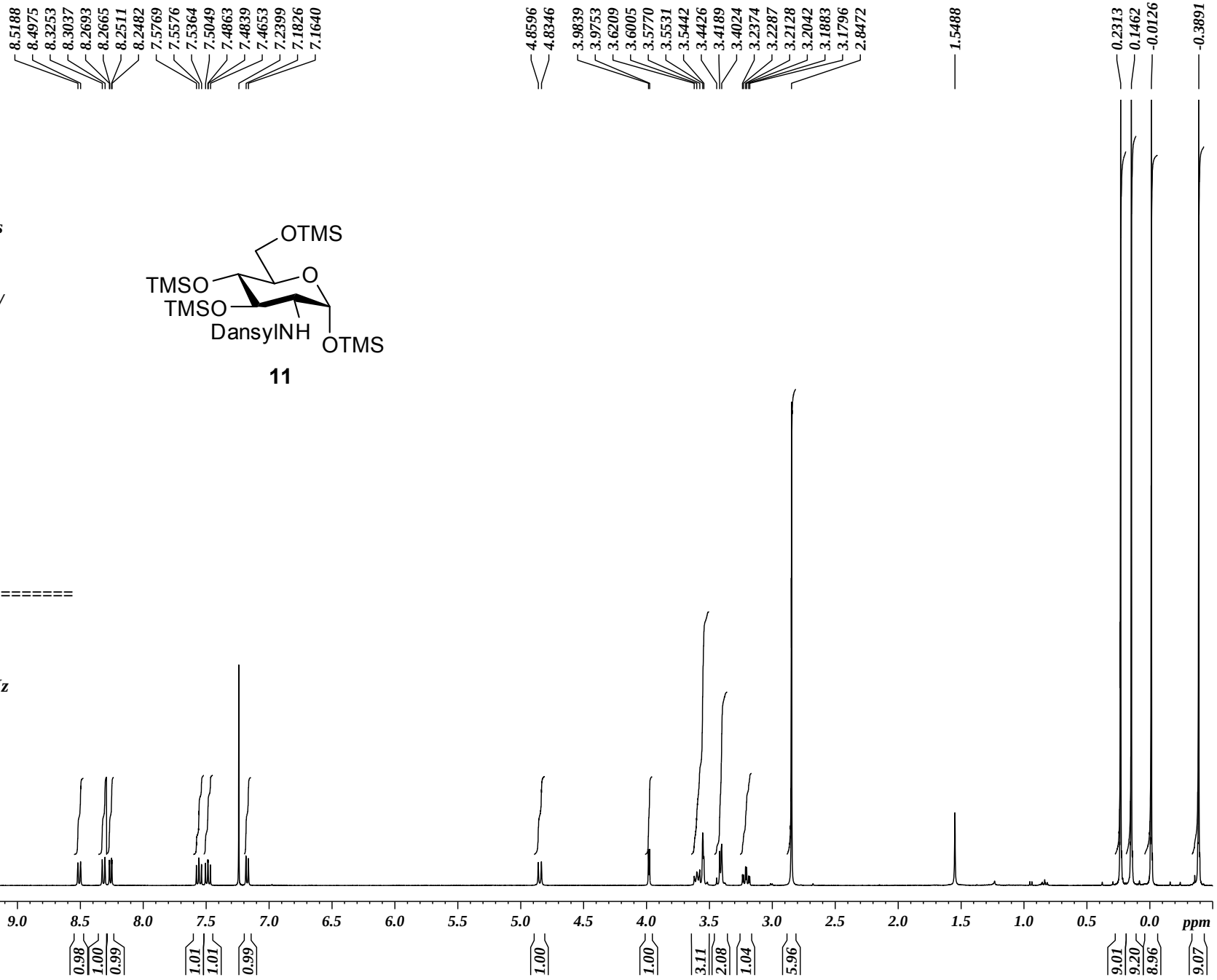
======== CHANNEL f1 ========
NUC1 1H
P1 10.30 usec
PL1 -2.00 dB
PL1W 23.88643074 W
SFO1 400.1320424 MHz

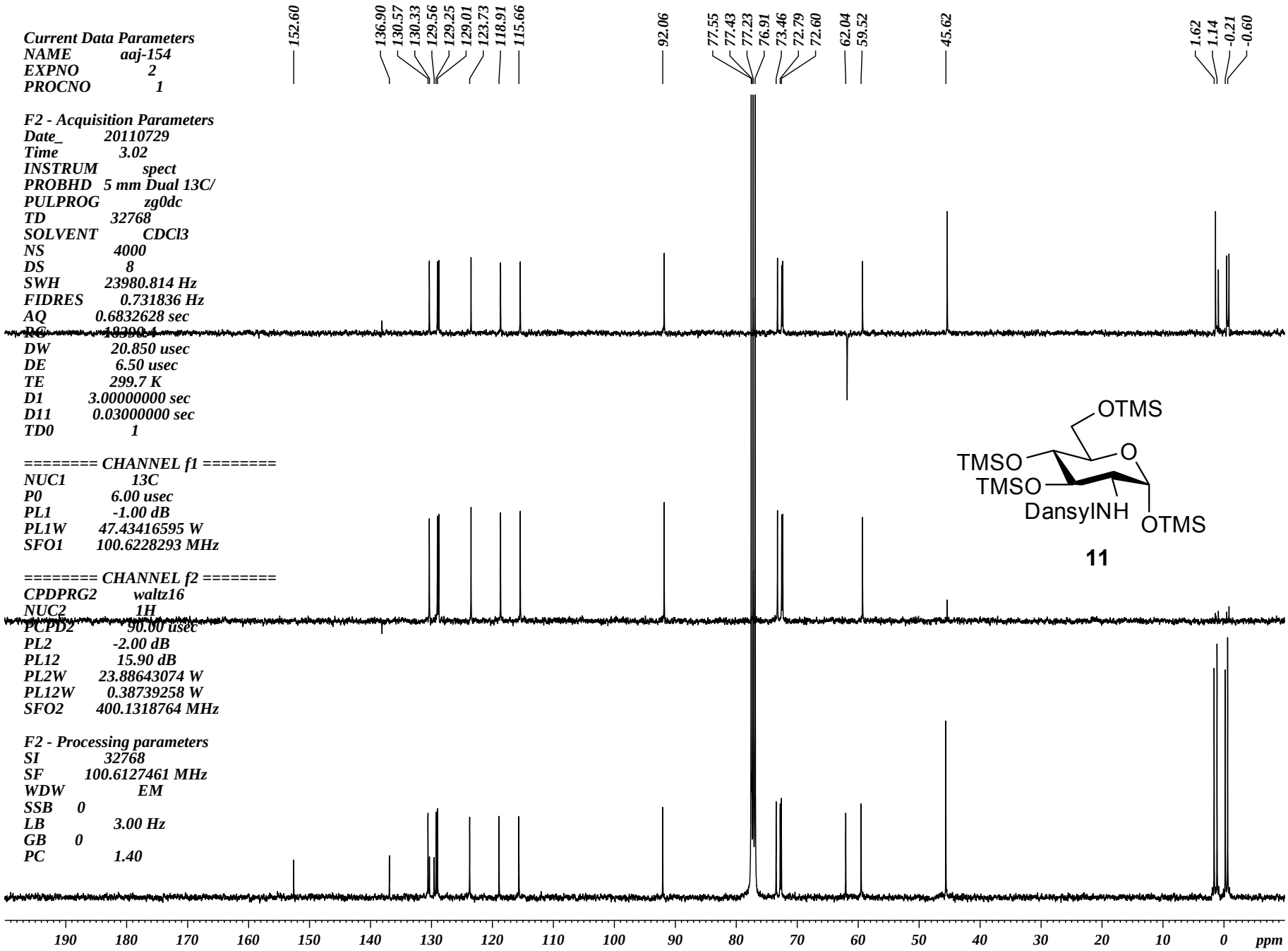
F2 - Processing parameters
SI 16384
SF 400.1300173 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

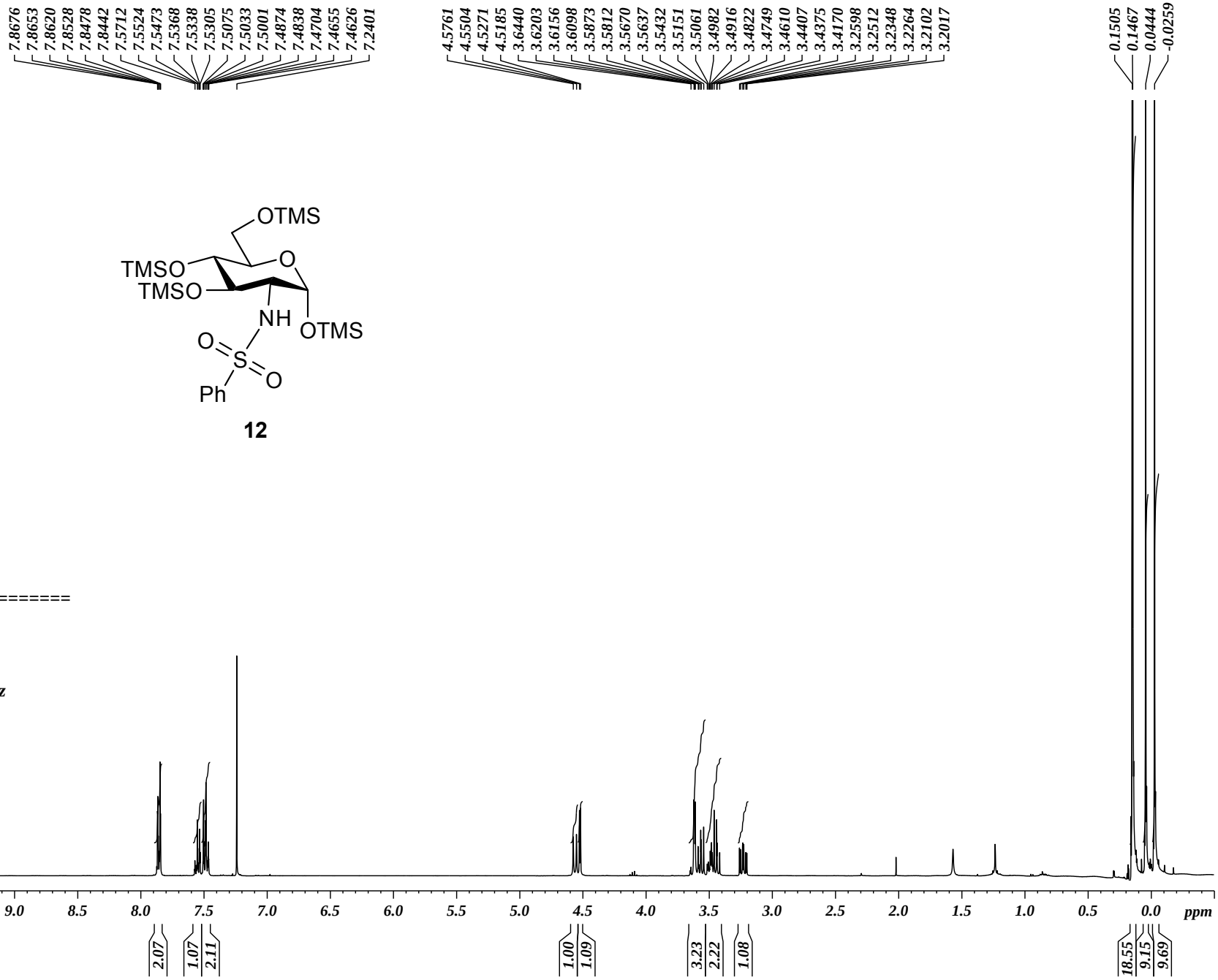


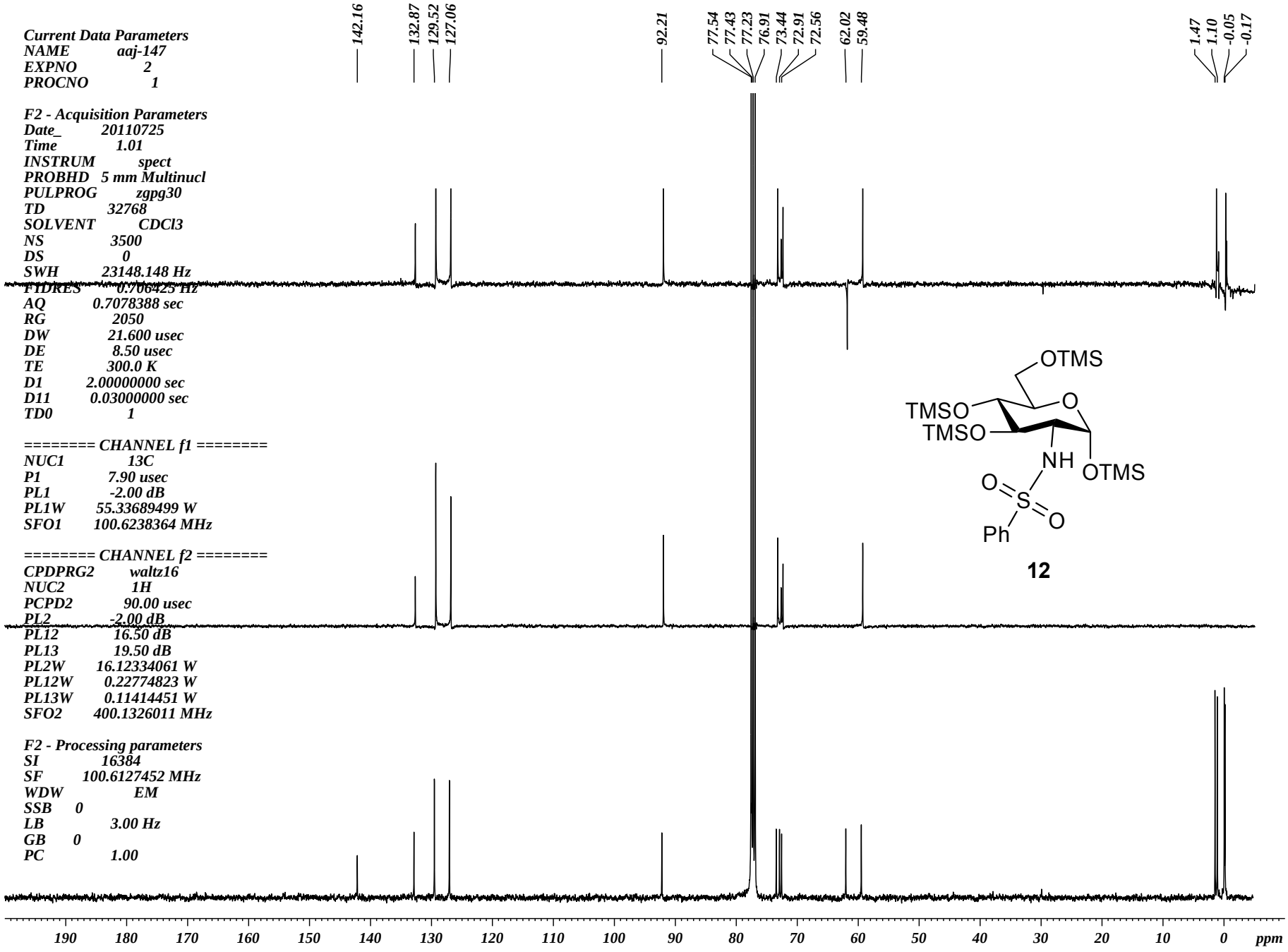
10

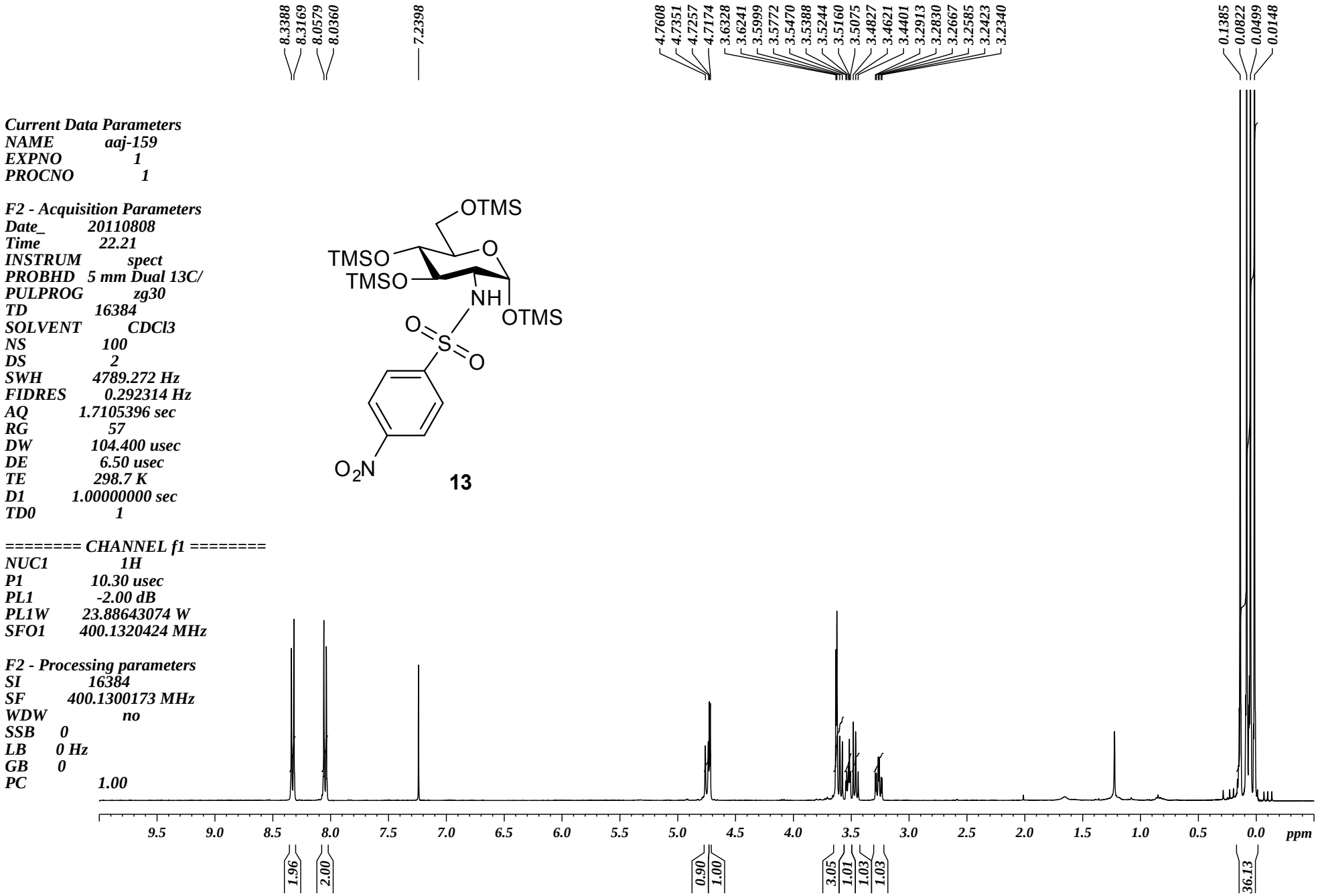

143.71
139.19
130.09
127.09
92.13
77.55
77.44
77.23
76.91
73.48
72.78
72.59
62.07
59.37
29.92
21.66
1.50
1.11
0.81
-0.08
-0.18
Current Data Parameters
NAME aaj-148
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110725
Time 2.11
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg0dc
TD 32768
SOLVENT CDCl3
NS 3600
DS 8
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 0.6832628 sec
RG 16384
DW 20.850 usec
DE 6.50 usec
TE 298.4 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P0 6.00 usec
PL1 -1.00 dB
PL1W 47.43416595 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 15.90 dB
PL2W 23.88643074 W
PL12W 0.38739258 W
SFO2 400.1318764 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
OTMS
O
TMSO
TMSO
TsNH
OTMS
10
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

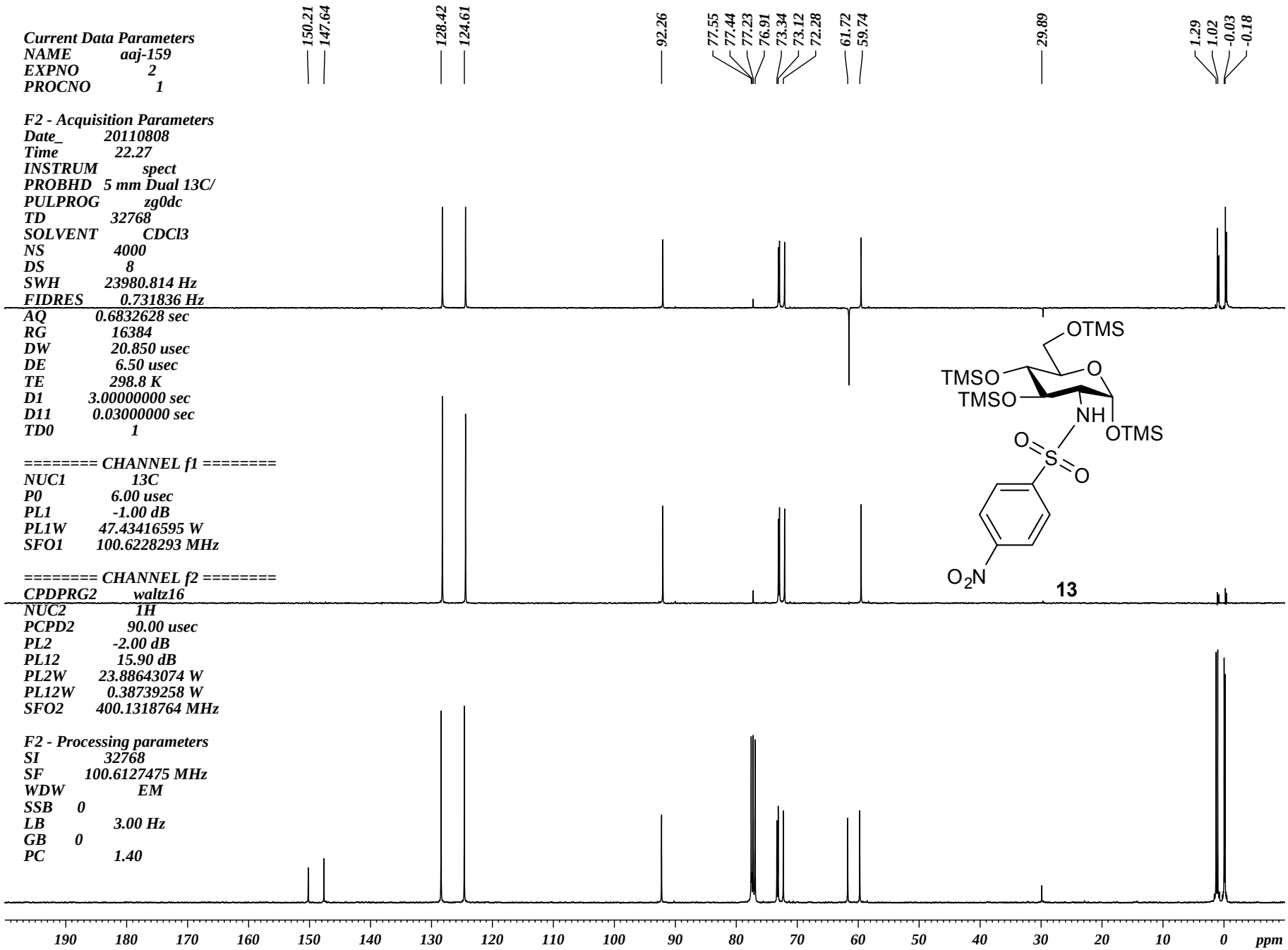

8.5188
8.4975
8.3253
8.3037
8.2693
8.2665
8.2511
8.2482
7.5769
7.5576
7.5364
7.5049
7.4863
7.4839
7.4653
7.2399
7.1826
7.1640
4.8596
4.8346
3.9839
3.9753
3.6209
3.6005
3.5770
3.5531
3.5442
3.4426
3.4189
3.4024
3.2374
3.2287
3.2128
3.2042
3.1883
3.1796
2.8472
1.5488
0.2313
0.1462
-0.0126
-0.3891
OTMS
TMSO
TMSO
O
DansylNH
OTMS
11
Current Data Parameters
NAME aaj-154
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110728
Time 22.52
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 200
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7105396 sec
RG 256
DW 104.400 usec
DE 6.50 usec
TE 299.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.30 usec
PL1 -2.00 dB
PL1W 23.88643074 W
SFO1 400.1320424 MHz
F2 - Processing parameters
SI 16384
SF 400.1300173 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
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
2.0
1.5
1.0
0.5
0.0
ppm
0.98
1.00
0.99
1.01
1.01
0.99
1.00
1.00
3.11
2.08
1.04
5.96
9.01
9.20
8.96
9.07

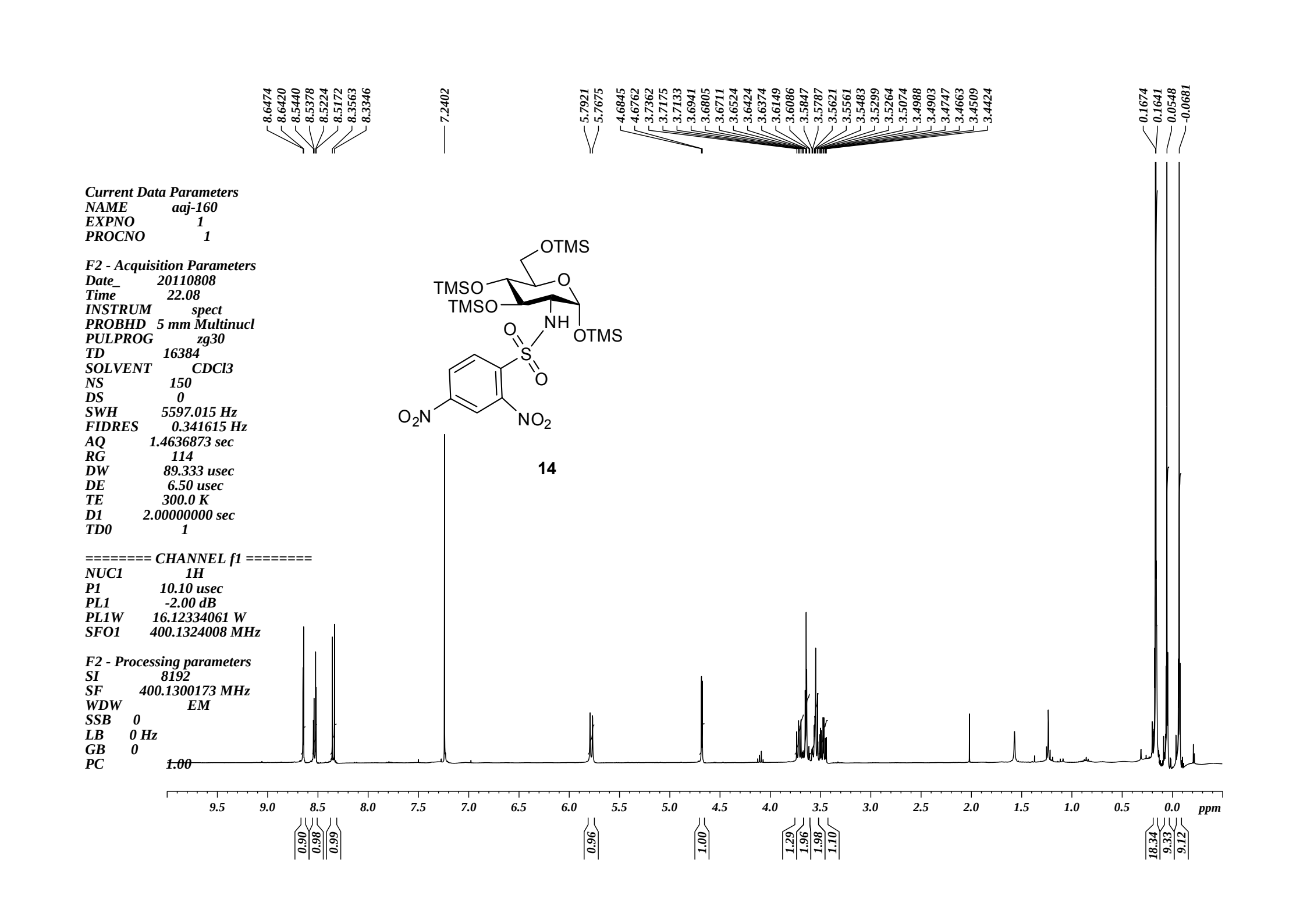

152.60
136.90
130.57
130.33
129.56
129.25
129.01
123.73
118.91
115.66
92.06
77.55
77.43
77.23
76.91
73.46
72.79
72.60
62.04
59.52
45.62
1.62
1.14
-0.21
-0.60
Current Data Parameters
NAME aaj-154
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110729
Time 3.02
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg0dc
TD 32768
SOLVENT CDCl3
NS 4000
DS 8
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 0.6832628 sec
RG 18390.4
DW 20.850 usec
DE 6.50 usec
TE 299.7 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P0 6.00 usec
PL1 -1.00 dB
PL1W 47.43416595 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 15.90 dB
PL2W 23.88643074 W
PL12W 0.38739258 W
SFO2 400.1318764 MHz
F2 - Processing parameters
SI 32768
SF 100.6127461 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
OTMS
O
TMSO
TMSO
DansylNH
OTMS
11
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

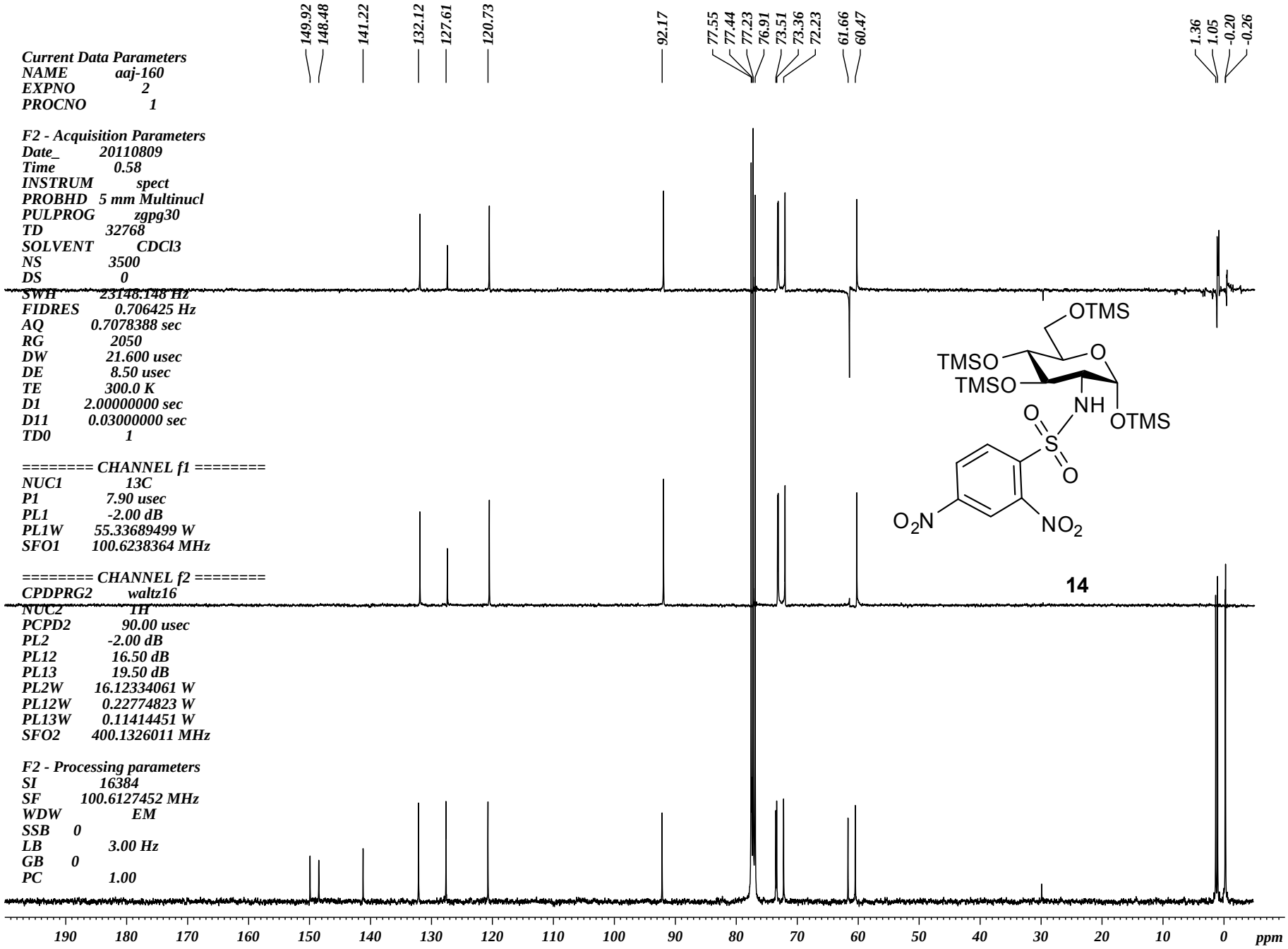

7.8676
7.8653
7.8620
7.8528
7.8478
7.8442
7.5712
7.5524
7.5473
7.5368
7.5338
7.5305
7.5075
7.5033
7.5001
7.4874
7.4838
7.4704
7.4655
7.4626
7.2401
4.5761
4.5504
4.5271
4.5185
3.6440
3.6203
3.6156
3.6098
3.5873
3.5812
3.5670
3.5637
3.5432
3.5151
3.5061
3.4982
3.4916
3.4822
3.4749
3.4610
3.4407
3.4375
3.4170
3.2598
3.2512
3.2348
3.2264
3.2102
3.2017
0.1505
0.1467
0.0444
-0.0259
OTMS
O
TMSO
TMSO
NH
OTMS
O
S
O
Ph
12
Current Data Parameters
NAME aaj-147
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110724
Time 22.14
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 100
DS 0
SWH 5597.015 Hz
FIDRES 0.341615 Hz
AQ 1.4636873 sec
RG 181
DW 89.333 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.10 usec
PL1 -2.00 dB
PL1W 16.12334061 W
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 8192
SF 400.1300173 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
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
2.0
1.5
1.0
0.5
0.0
ppm
2.07
1.07
2.11
1.00
1.09
3.23
2.22
1.08
18.55
9.15
9.69

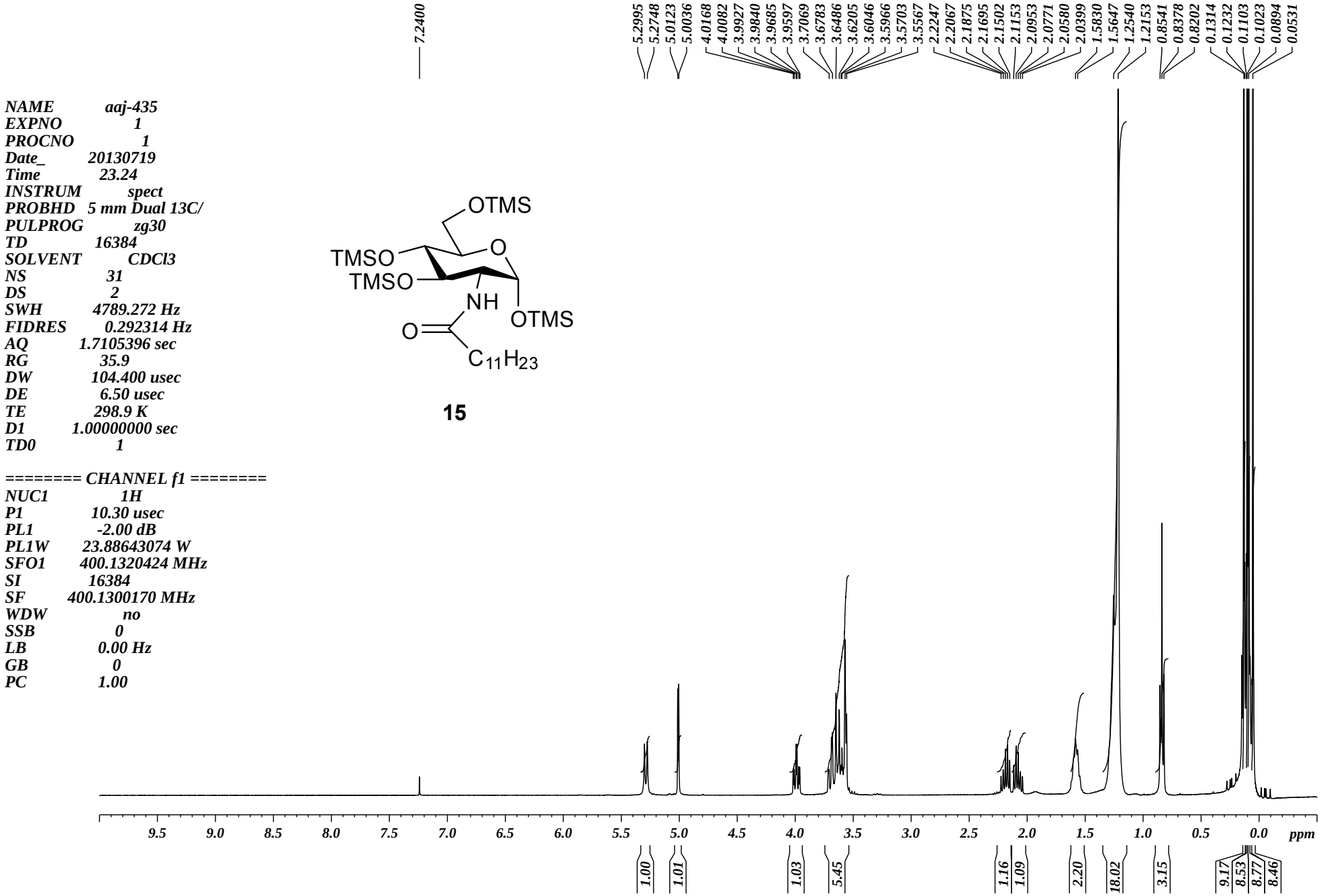

142.16
132.87
129.52
127.06
92.21
77.54
77.43
77.23
76.91
73.44
72.91
72.56
62.02
59.48
1.47
1.10
-0.05
-0.17
Current Data Parameters
NAME aaj-147
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110725
Time 1.01
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 3500
DS 0
SWH 23148.148 Hz
FIDRES 0.706425 Hz
AQ 0.7078388 sec
RG 2050
DW 21.600 usec
DE 8.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.90 usec
PL1 -2.00 dB
PL1W 55.33689499 W
SFO1 100.6238364 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 16.50 dB
PL13 19.50 dB
PL2W 16.12334061 W
PL12W 0.22774823 W
PL13W 0.11414451 W
SFO2 400.1326011 MHz
F2 - Processing parameters
SI 16384
SF 100.6127452 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
OTMS
TMSO
TMSO
O
NH
OTMS
O
S
O
Ph
12
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

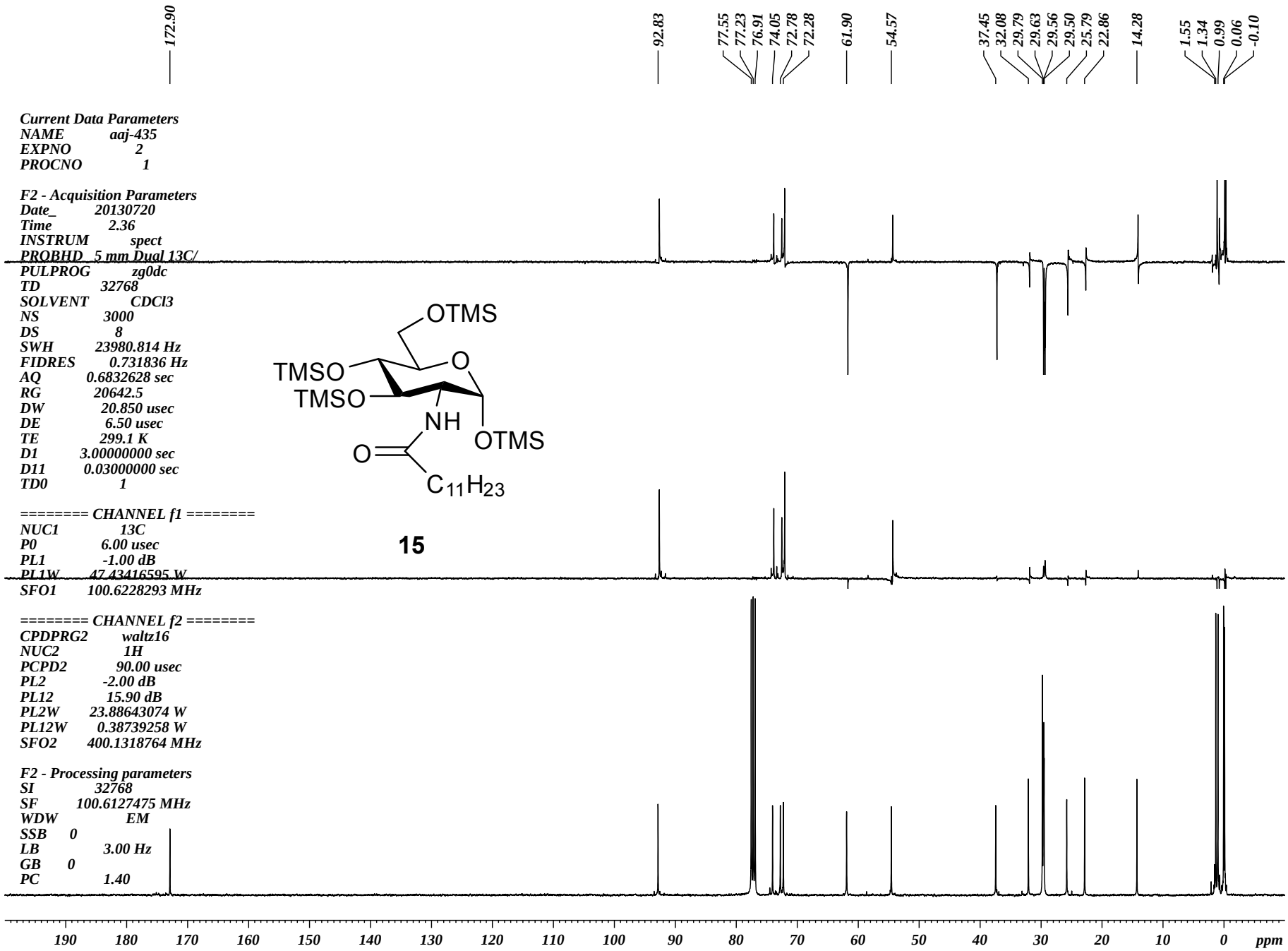

8.3388
8.3169
8.0579
8.0360
7.2398
4.7608
4.7351
4.7257
4.7174
3.6328
3.6241
3.5999
3.5772
3.5470
3.5388
3.5244
3.5160
3.5075
3.4827
3.4621
3.4401
3.2913
3.2830
3.2667
3.2585
3.2423
3.2340
0.1385
0.0822
0.0499
0.0148
Current Data Parameters
NAME aaj-159
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110808
Time 22.21
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 100
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7105396 sec
RG 57
DW 104.400 usec
DE 6.50 usec
TE 298.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.30 usec
PL1 -2.00 dB
PL1W 23.88643074 W
SFO1 400.1320424 MHz
F2 - Processing parameters
SI 16384
SF 400.1300173 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
OTMS
TMSO
TMSO
O
NH
OTMS
O
S
O
O2N
13
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
2.0
1.5
1.0
0.5
0.0
ppm
1.96
2.00
0.90
1.00
3.05
1.01
1.03
1.03
36.13


150.21
147.64
128.42
124.61
92.26
77.55
77.44
77.23
76.91
73.34
73.12
72.28
61.72
59.74
29.89
1.29
1.02
-0.03
-0.18
Current Data Parameters
NAME aaj-159
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110808
Time 22.27
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg0dc
TD 32768
SOLVENT CDCl3
NS 4000
DS 8
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 0.6832628 sec
RG 16384
DW 20.850 usec
DE 6.50 usec
TE 298.8 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P0 6.00 usec
PL1 -1.00 dB
PL1W 47.43416595 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 15.90 dB
PL2W 23.88643074 W
PL12W 0.38739258 W
SFO2 400.1318764 MHz
F2 - Processing parameters
SI 32768
SF 100.6127475 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
OTMS
TMSO
TMSO
O
NH
OTMS
O
S
O
O2N
13
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


8.6474
8.6420
8.5440
8.5378
8.5224
8.5172
8.3563
8.3346
7.2402
5.7921
5.7675
4.6845
4.6762
3.7362
3.7175
3.7133
3.6941
3.6805
3.6711
3.6524
3.6424
3.6374
3.6149
3.6086
3.5847
3.5787
3.5621
3.5561
3.5483
3.5299
3.5264
3.5074
3.4988
3.4903
3.4747
3.4663
3.4509
3.4424
0.1674
0.1641
0.0548
-0.0681
Current Data Parameters
NAME aaj-160
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110808
Time 22.08
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 150
DS 0
SWH 5597.015 Hz
FIDRES 0.341615 Hz
AQ 1.4636873 sec
RG 114
DW 89.333 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.10 usec
PL1 -2.00 dB
PL1W 16.12334061 W
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 8192
SF 400.1300173 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
OTMS
TMSO
TMSO
O
NH
OTMS
O
S
O
O2N
NO2
14
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
2.0
1.5
1.0
0.5
0.0
ppm
0.90
0.98
0.99
0.96
1.00
1.29
1.96
1.98
1.10
18.34
9.33
9.12


149.92
148.48
141.22
132.12
127.61
120.73
92.17
77.55
77.44
77.23
76.91
73.51
73.36
72.23
61.66
60.47
1.36
1.05
-0.20
-0.26
Current Data Parameters
NAME aaj-160
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110809
Time 0.58
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 3500
DS 0
SWH 23148.148 Hz
FIDRES 0.706425 Hz
AQ 0.7078388 sec
RG 2050
DW 21.600 usec
DE 8.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.90 usec
PL1 -2.00 dB
PL1W 55.33689499 W
SFO1 100.6238364 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 16.50 dB
PL13 19.50 dB
PL2W 16.12334061 W
PL12W 0.22774823 W
PL13W 0.11414451 W
SFO2 400.1326011 MHz
F2 - Processing parameters
SI 16384
SF 100.6127452 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
OTMS
TMSO
TMSO
O
NH
OTMS
O
S
O
O2N
NO2
14
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


7.2400
5.2995
5.2748
5.0123
5.0036
4.0168
4.0082
3.9927
3.9840
3.9685
3.9597
3.7069
3.6783
3.6486
3.6205
3.6046
3.5966
3.5703
3.5567
2.2247
2.2067
2.1875
2.1695
2.1502
2.1153
2.0953
2.0771
2.0580
2.0399
1.5830
1.5647
1.2540
1.2153
0.8541
0.8378
0.8202
0.1314
0.1232
0.1103
0.1023
0.0894
0.0531
NAME aaj-435
EXPNO 1
PROCNO 1
Date_ 20130719
Time 23.24
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 31
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7105396 sec
RG 35.9
DW 104.400 usec
DE 6.50 usec
TE 298.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.30 usec
PL1 -2.00 dB
PL1W 23.88643074 W
SFO1 400.1320424 MHz
SI 16384
SF 400.1300170 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
OTMS
TMSO
TMSO
O
NH
OTMS
O
C11H23
15
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
2.0
1.5
1.0
0.5
0.0
ppm
1.00
1.01
1.03
5.45
1.16
1.09
2.20
18.02
3.15
9.17
8.53
8.77
8.46

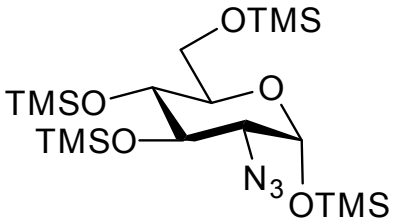

172.90
92.83
77.55
77.23
76.91
74.05
72.78
72.28
61.90
54.57
37.45
32.08
29.79
29.63
29.56
29.50
25.79
22.86
14.28
1.55
1.34
0.99
0.06
-0.10
Current Data Parameters
NAME aaj-435
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130720
Time 2.36
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg0dc
TD 32768
SOLVENT CDCl3
NS 3000
DS 8
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 0.6832628 sec
RG 20642.5
DW 20.850 usec
DE 6.50 usec
TE 299.1 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P0 6.00 usec
PL1 -1.00 dB
PL1W 47.43416595 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 15.90 dB
PL2W 23.88643074 W
PL12W 0.38739258 W
SFO2 400.1318764 MHz
F2 - Processing parameters
SI 32768
SF 100.6127475 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
OTMS
TMSO
TMSO
O
NH
OTMS
O
C11H23
15
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

Current Data Parameters
NAME aaj-164-pdt
EXPNO 1
PROCNO 1

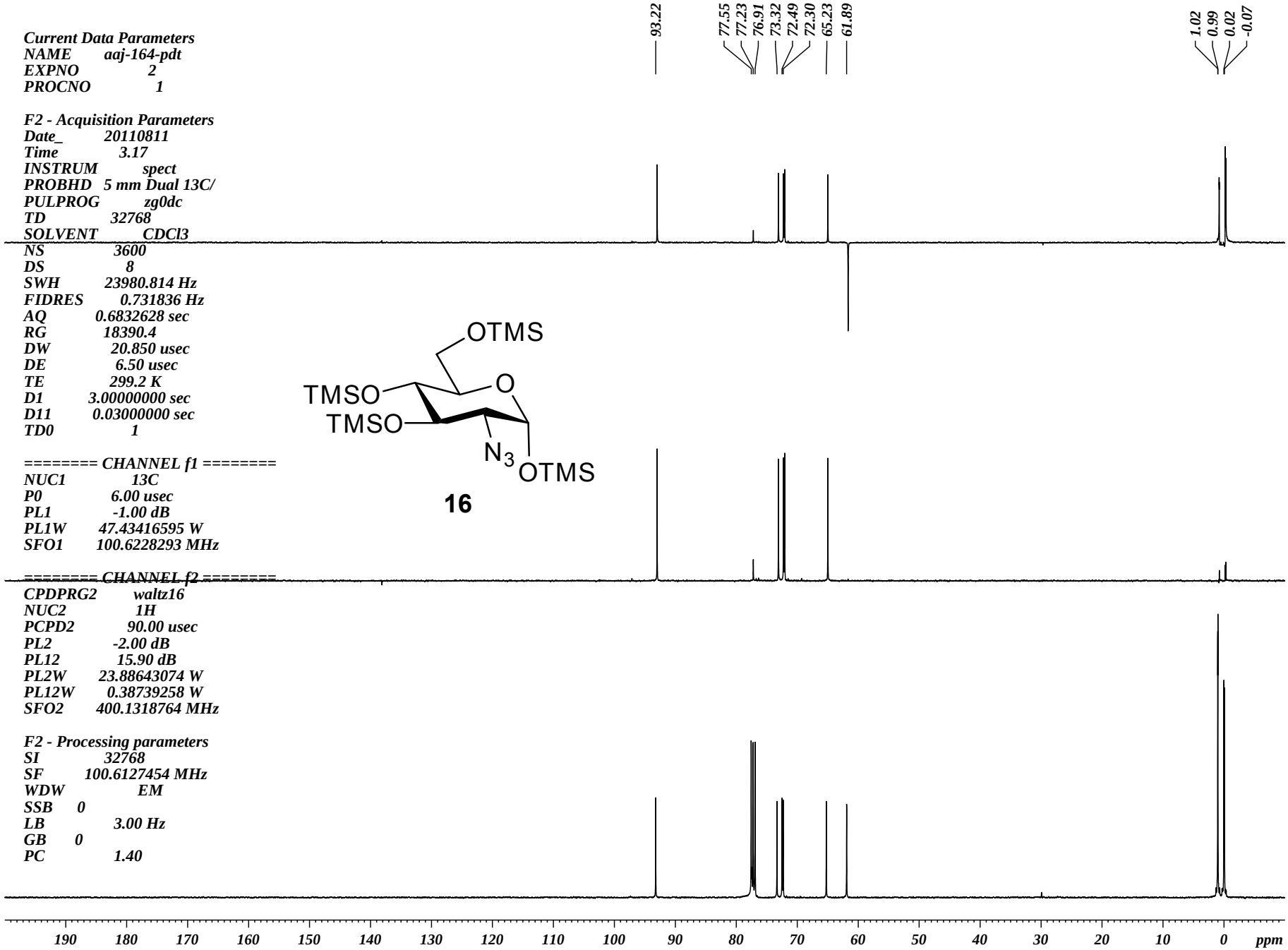
F2 - Acquisition Parameters
Date_ 20110810
Time 23.15
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 24
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7105396 sec
RG 64
DW 104.400 usec
DE 6.50 usec
TE 299.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.30 usec
PL1 -2.00 dB
PL1W 23.88643074 W
SFO1 400.1320424 MHz

F2 - Processing parameters
SI 16384
SF 400.1300173 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00



16

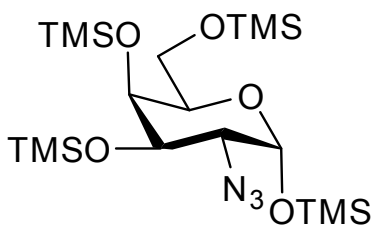

93.22
77.55
77.23
76.91
73.32
72.49
72.30
65.23
61.89
1.02
0.99
0.02
-0.07
Current Data Parameters
NAME aaj-164-pdt
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110811
Time 3.17
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg0dc
TD 32768
SOLVENT CDCl3
NS 3600
DS 8
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 0.6832628 sec
RG 18390.4
DW 20.850 usec
DE 6.50 usec
TE 299.2 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P0 6.00 usec
PL1 -1.00 dB
PL1W 47.43416595 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 15.90 dB
PL2W 23.88643074 W
PL12W 0.38739258 W
SFO2 400.1318764 MHz
F2 - Processing parameters
SI 32768
SF 100.6127454 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
OTMS
TMSO
TMSO
O
N3
OTMS
16
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

Current Data Parameters
NAME vpv-373 A
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120901
Time 7.42
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 100
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7105396 sec
RG 228.1
DW 104.400 usec
DE 6.50 usec
TE 299.1 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.30 usec
PL1 -2.00 dB
PL1W 23.88643074 W
SFO1 400.1320424 MHz

F2 - Processing parameters
SI 16384
SF 400.1300172 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



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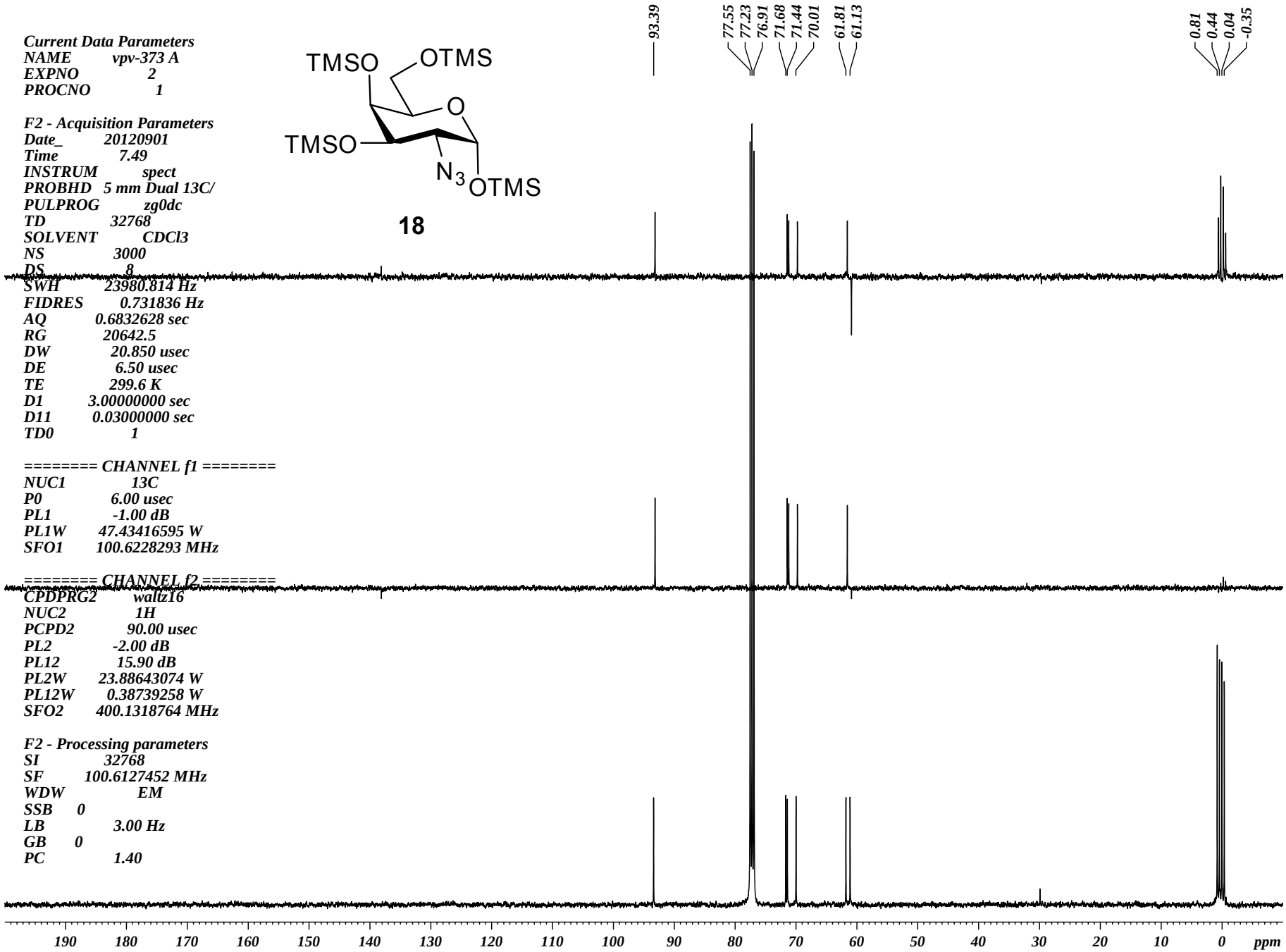
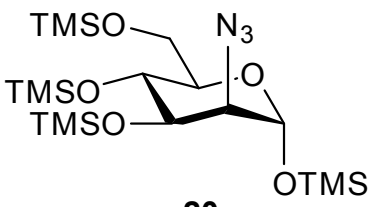

93.39
77.55
77.23
76.91
71.68
71.44
70.01
61.81
61.13
0.81
0.44
0.04
-0.35
TMSO
OTMS
O
TMSO
N3
OTMS
18
Current Data Parameters
NAME vpv-373 A
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120901
Time 7.49
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg0dc
TD 32768
SOLVENT CDCl3
NS 3000
DS 8
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 0.6832628 sec
RG 20642.5
DW 20.850 usec
DE 6.50 usec
TE 299.6 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P0 6.00 usec
PL1 -1.00 dB
PL1W 47.43416595 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 15.90 dB
PL2W 23.88643074 W
PL12W 0.38739258 W
SFO2 400.1318764 MHz
F2 - Processing parameters
SI 32768
SF 100.6127452 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

Current Data Parameters
NAME aaj-mannose-azaide all data
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20121011
Time 23.05
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 200
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7105396 sec
RG 71.8
DW 104.400 usec
DE 6.50 usec
TE 299.4 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.30 usec
PL1 -2.00 dB
PL1W 23.88643074 W
SFO1 400.1320424 MHz

F2 - Processing parameters
SI 16384
SF 400.1300170 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00



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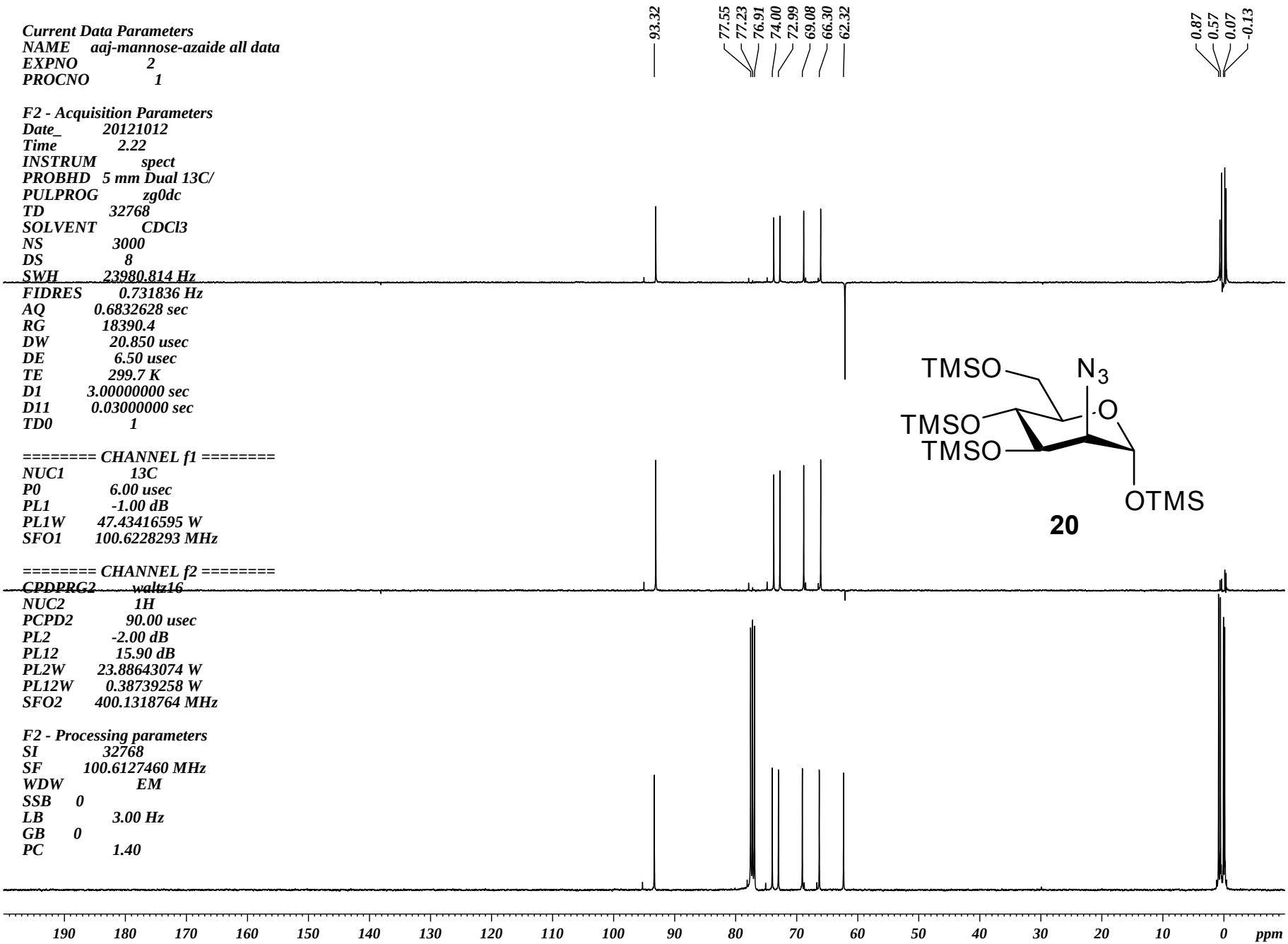
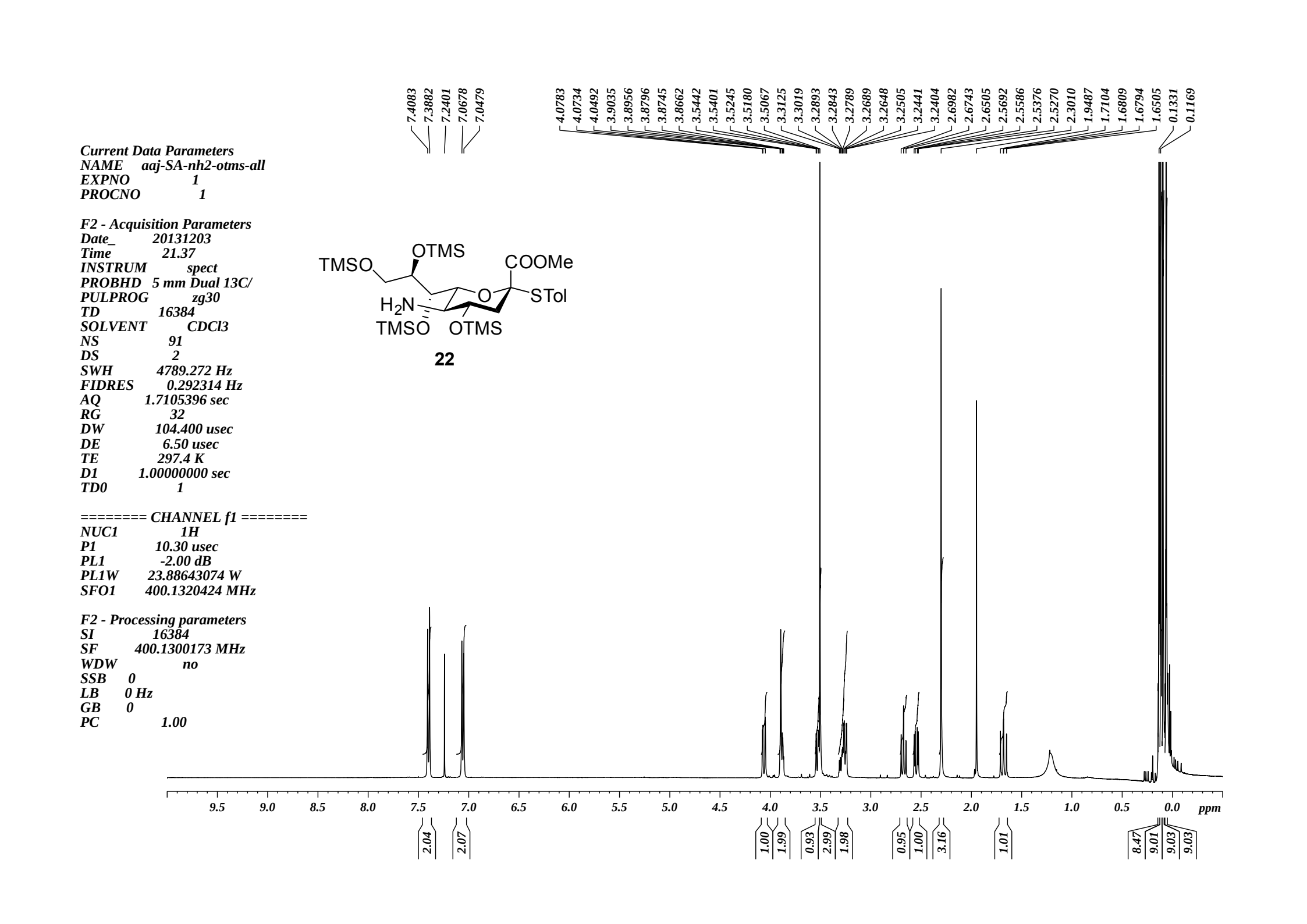
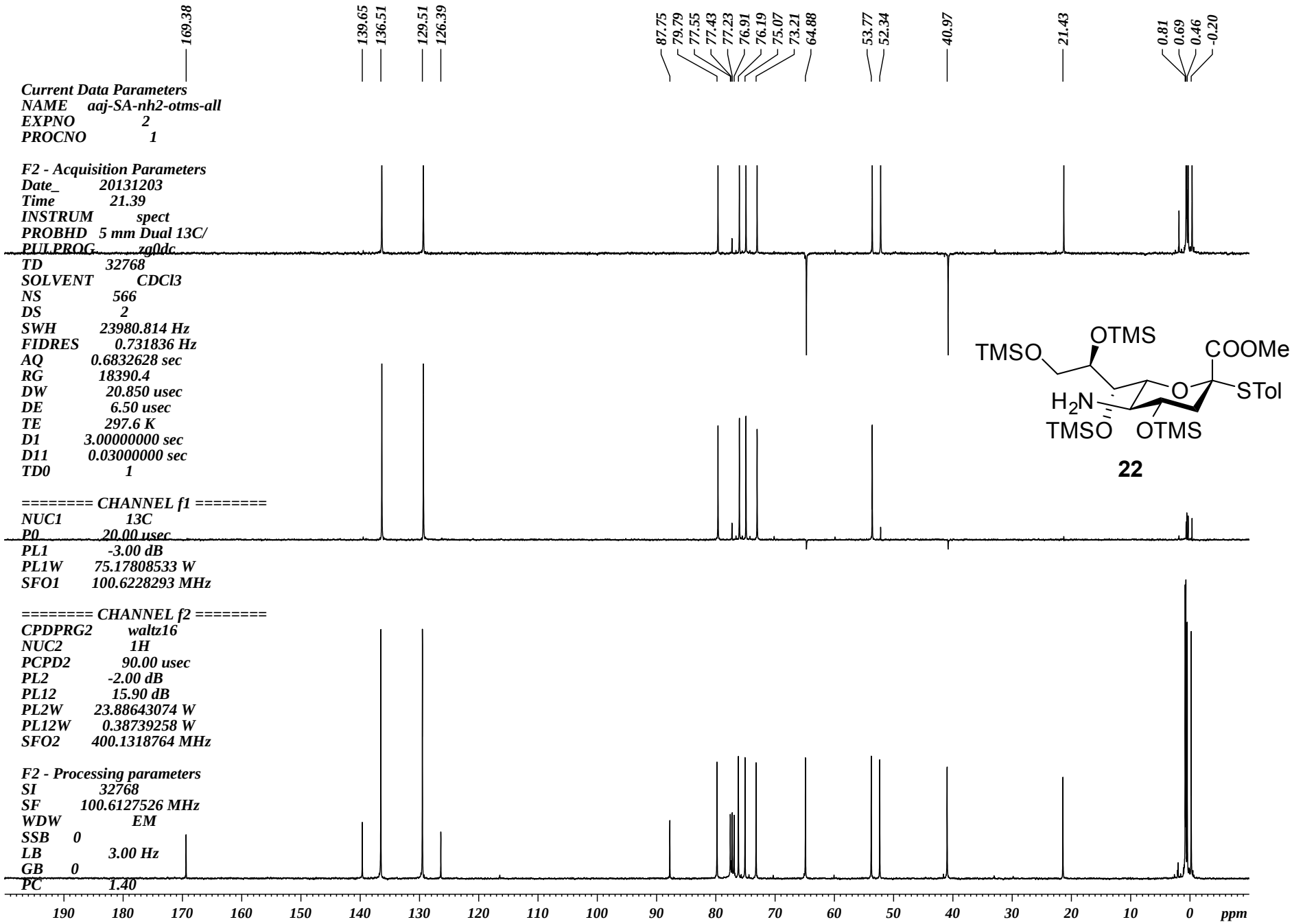
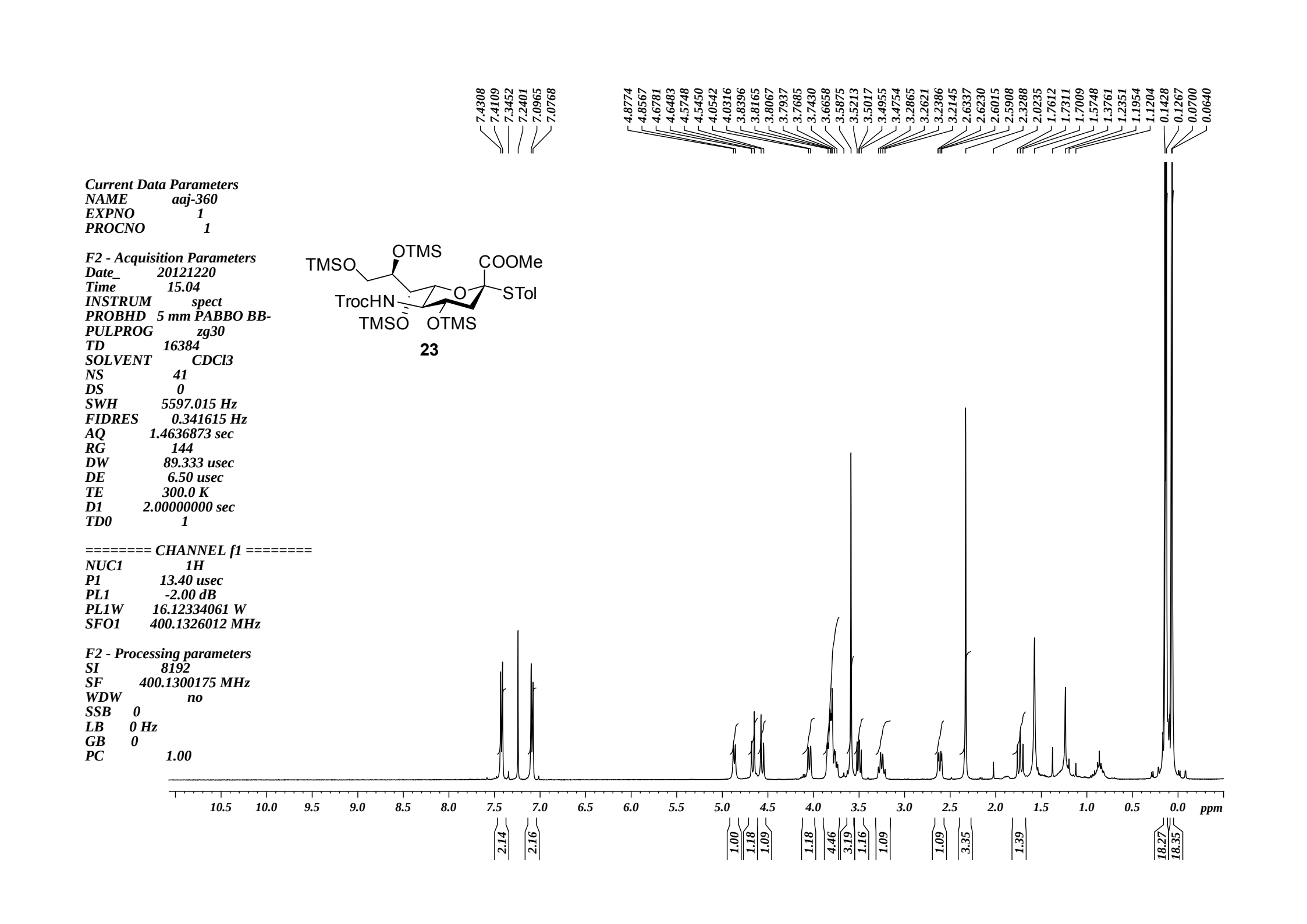
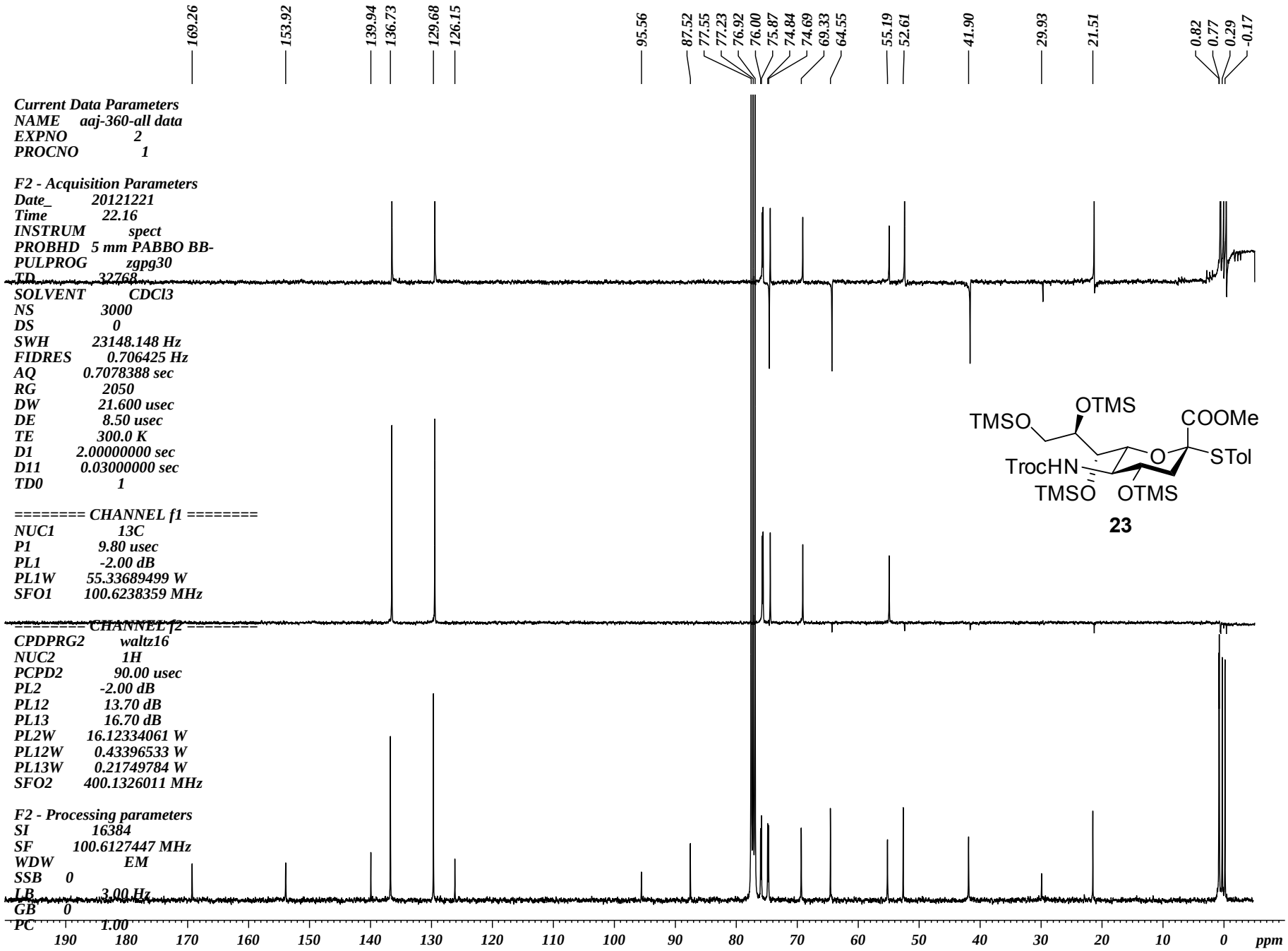
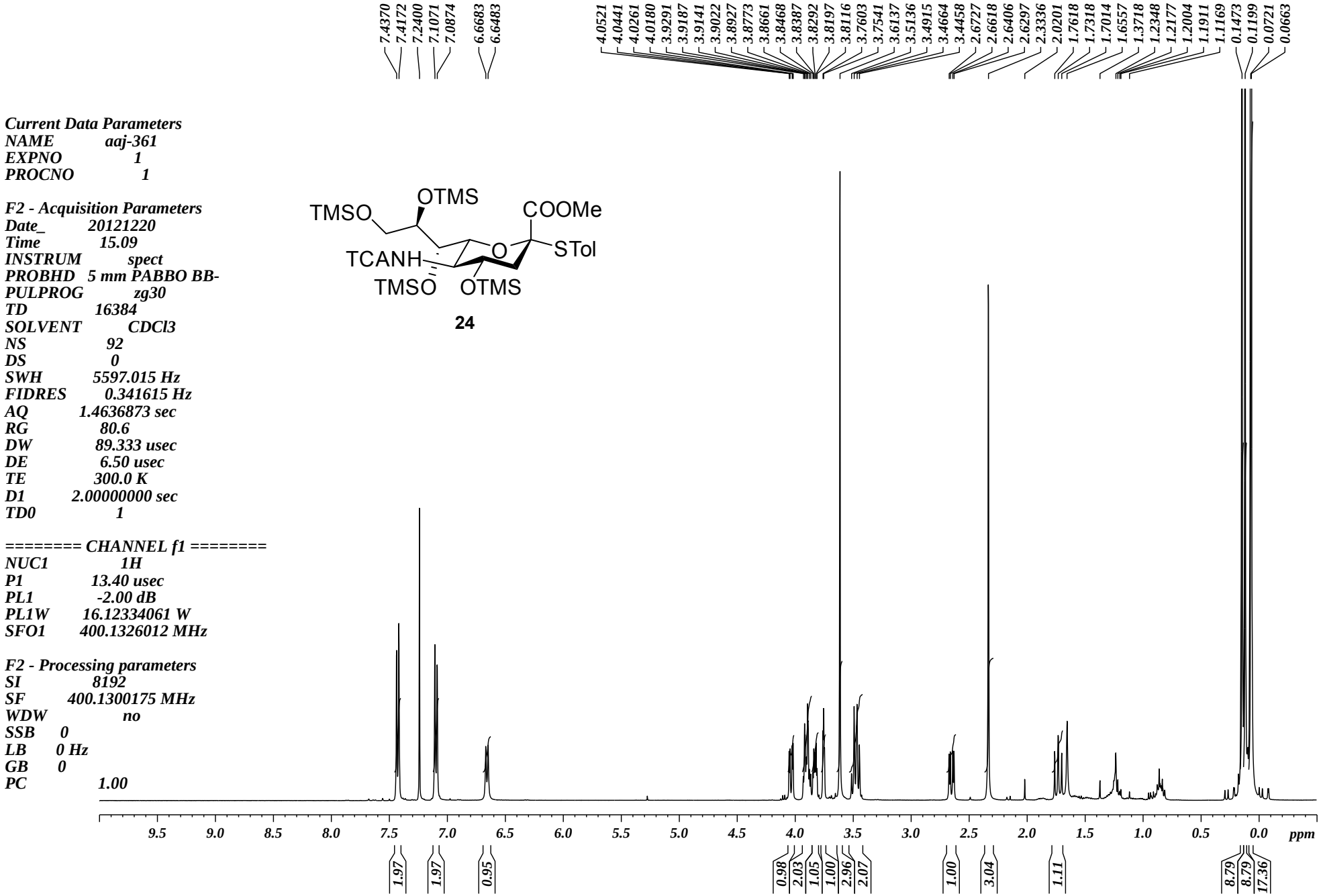

93.32
77.55
77.23
76.91
74.00
72.99
69.08
66.30
62.32
0.87
0.57
0.07
-0.13
Current Data Parameters
NAME aaj-mannose-azaide all data
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121012
Time 2.22
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg0dc
TD 32768
SOLVENT CDCl3
NS 3000
DS 8
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 0.6832628 sec
RG 18390.4
DW 20.850 usec
DE 6.50 usec
TE 299.7 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P0 6.00 usec
PL1 -1.00 dB
PL1W 47.43416595 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 15.90 dB
PL2W 23.88643074 W
PL12W 0.38739258 W
SFO2 400.1318764 MHz
F2 - Processing parameters
SI 32768
SF 100.6127460 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
TMSO
N3
O
TMSO
TMSO
OTMS
20
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

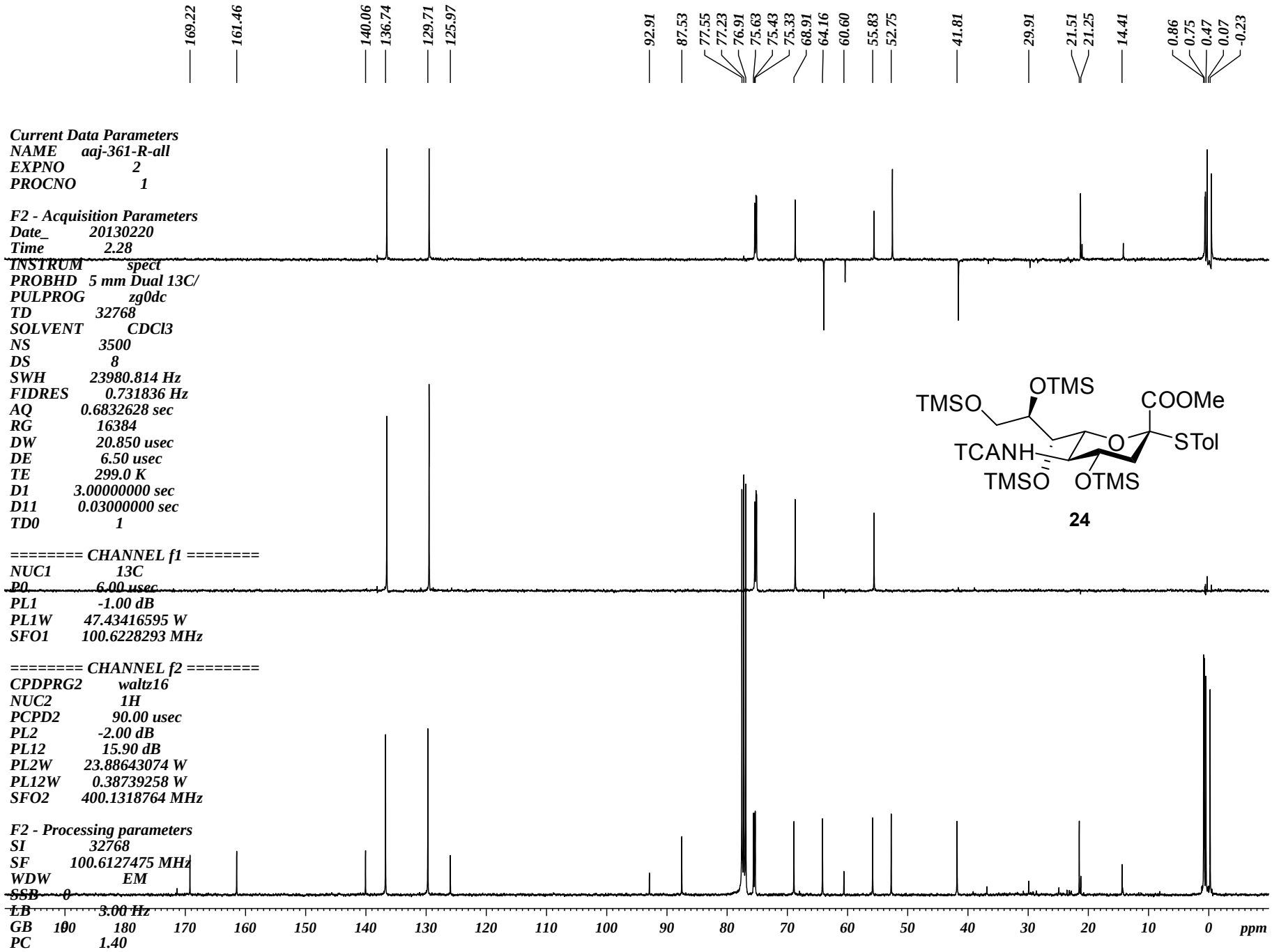

7.4083
7.3882
7.2401
7.0678
7.0479
4.0783
4.0734
4.0492
3.9035
3.8956
3.8796
3.8745
3.8662
3.5442
3.5401
3.5245
3.5180
3.5067
3.3125
3.3019
3.2893
3.2843
3.2789
3.2689
3.2648
3.2505
3.2441
3.2404
2.6982
2.6743
2.6505
2.5692
2.5586
2.5376
2.5270
2.3010
1.9487
1.7104
1.6809
1.6794
1.6505
0.1331
0.1169
Current Data Parameters
NAME aaj-SA-nh2-otms-all
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131203
Time 21.37
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 91
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7105396 sec
RG 32
DW 104.400 usec
DE 6.50 usec
TE 297.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.30 usec
PL1 -2.00 dB
PL1W 23.88643074 W
SFO1 400.1320424 MHz
F2 - Processing parameters
SI 16384
SF 400.1300173 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
TMSO
OTMS
COOMe
O
STol
H2N
TMSO
OTMS
22
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
2.0
1.5
1.0
0.5
0.0
ppm
2.04
2.07
1.00
1.99
0.93
2.99
1.98
0.95
1.00
3.16
1.01
8.47
9.01
9.03
9.03

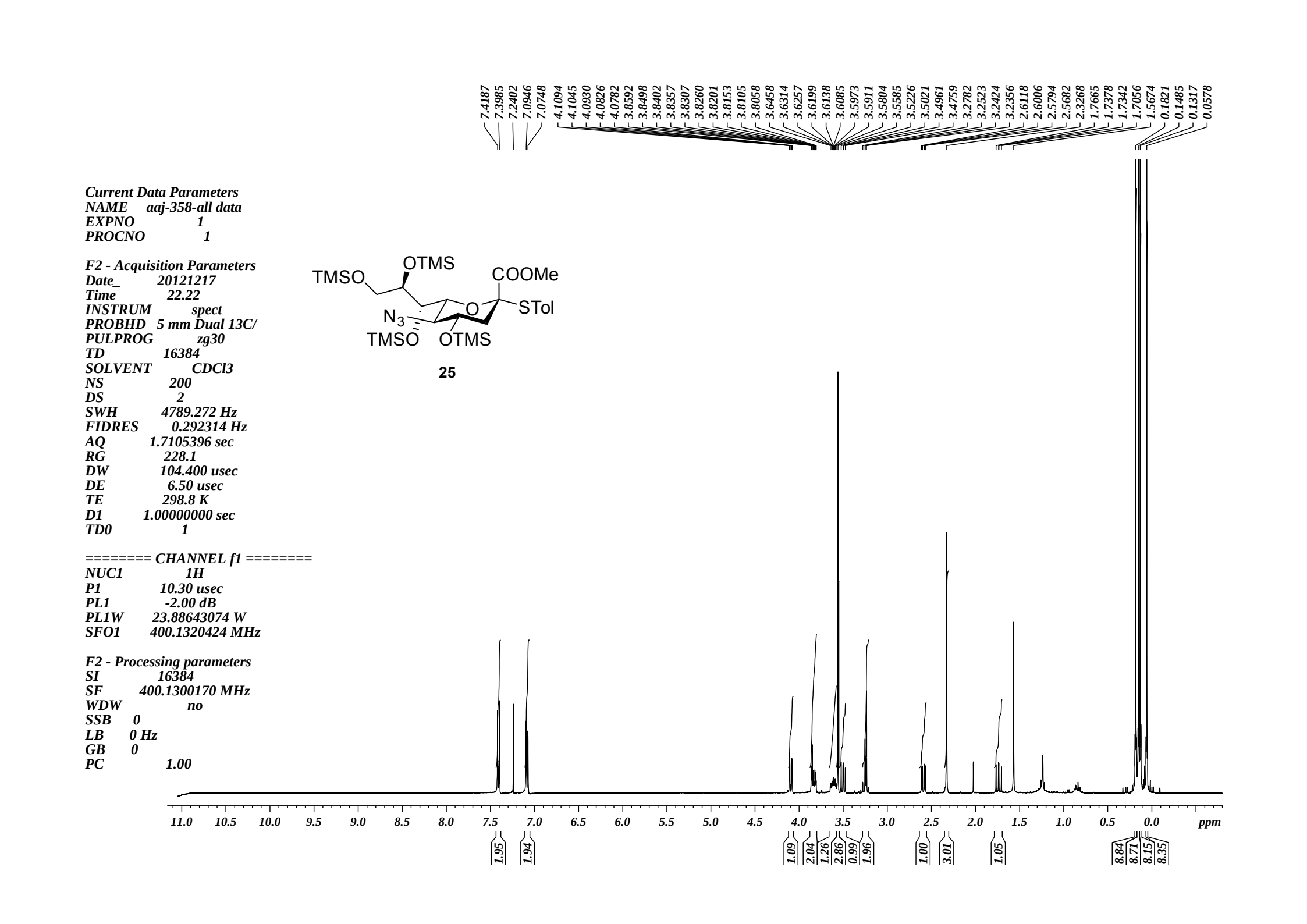

169.38
139.65
136.51
129.51
126.39
87.75
79.79
77.55
77.43
77.23
76.91
76.19
75.07
73.21
64.88
53.77
52.34
40.97
21.43
0.81
0.69
0.46
-0.20
Current Data Parameters
NAME aaj-SA-nh2-otms-all
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131203
Time 21.39
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg0dc
TD 32768
SOLVENT CDCl3
NS 566
DS 2
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 0.6832628 sec
RG 18390.4
DW 20.850 usec
DE 6.50 usec
TE 297.6 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P0 20.00 usec
PL1 -3.00 dB
PL1W 75.17808533 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 15.90 dB
PL2W 23.88643074 W
PL12W 0.38739258 W
SFO2 400.1318764 MHz
F2 - Processing parameters
SI 32768
SF 100.6127526 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
TMSO
OTMS
COOMe
O
STol
H2N
TMSO
OTMS
22
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

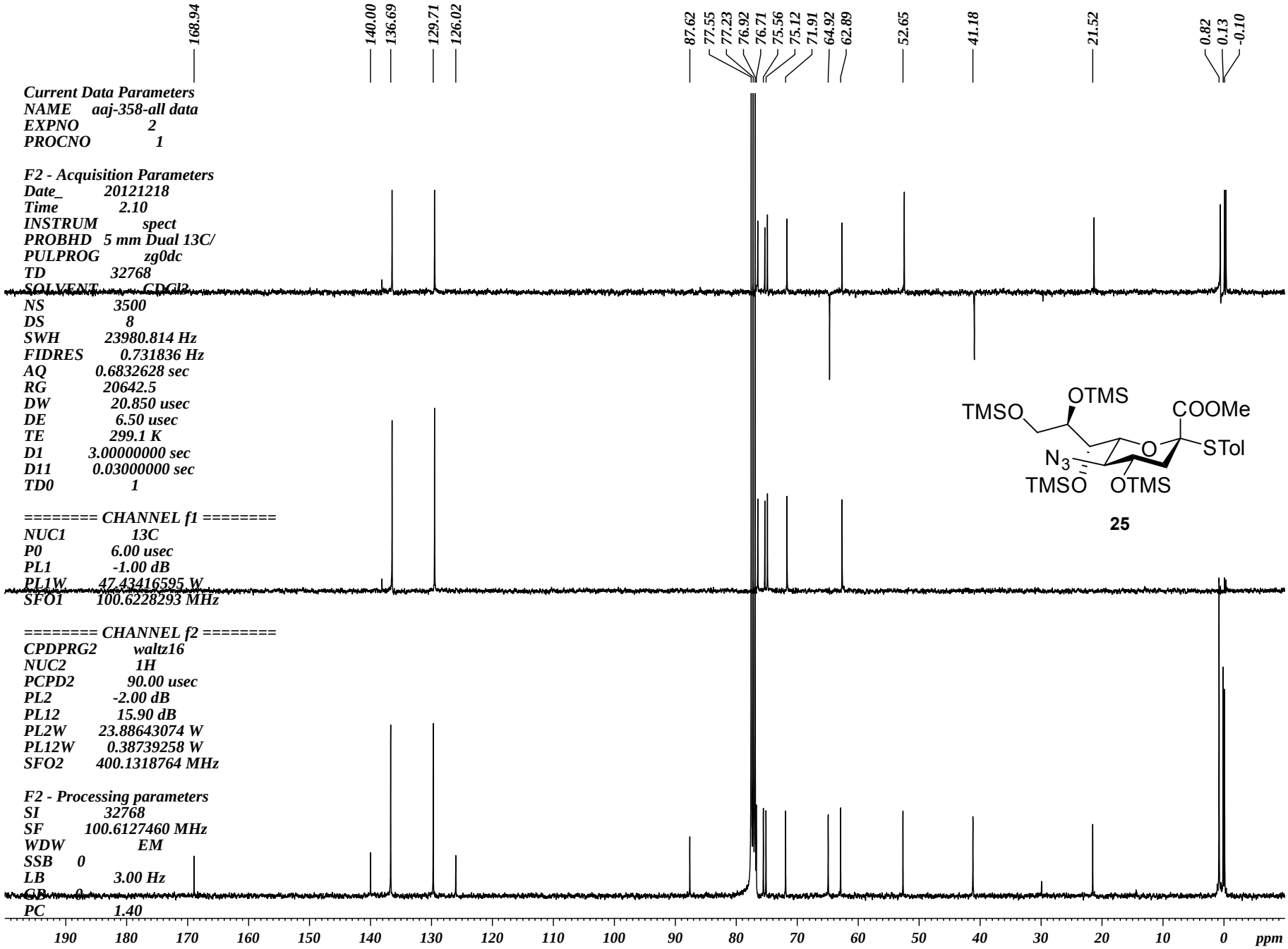

7.4308
7.4109
7.3452
7.2401
7.0965
7.0768
4.8774
4.8567
4.6781
4.6483
4.5748
4.5450
4.0542
4.0316
3.8396
3.8165
3.8067
3.7937
3.7685
3.7430
3.6658
3.5875
3.5213
3.5017
3.4955
3.4754
3.2865
3.2621
3.2386
3.2145
2.6337
2.6230
2.6015
2.5908
2.3288
2.0235
1.7612
1.7311
1.7009
1.5748
1.3761
1.2351
1.1954
1.1204
0.1428
0.1267
0.0700
0.0640
Current Data Parameters
NAME aaj-360
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121220
Time 15.04
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 41
DS 0
SWH 5597.015 Hz
FIDRES 0.341615 Hz
AQ 1.4636873 sec
RG 144
DW 89.333 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.40 usec
PL1 -2.00 dB
PL1W 16.12334061 W
SFO1 400.1326012 MHz
F2 - Processing parameters
SI 8192
SF 400.1300175 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
TMSO
OTMS
COOMe
O
STol
TrocHN
TMSO
OTMS
23
10.5
10.0
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
2.0
1.5
1.0
0.5
0.0
ppm
2.14
2.16
1.00
1.18
1.09
1.18
4.46
3.19
1.16
1.09
1.09
3.35
1.39
18.27
18.35

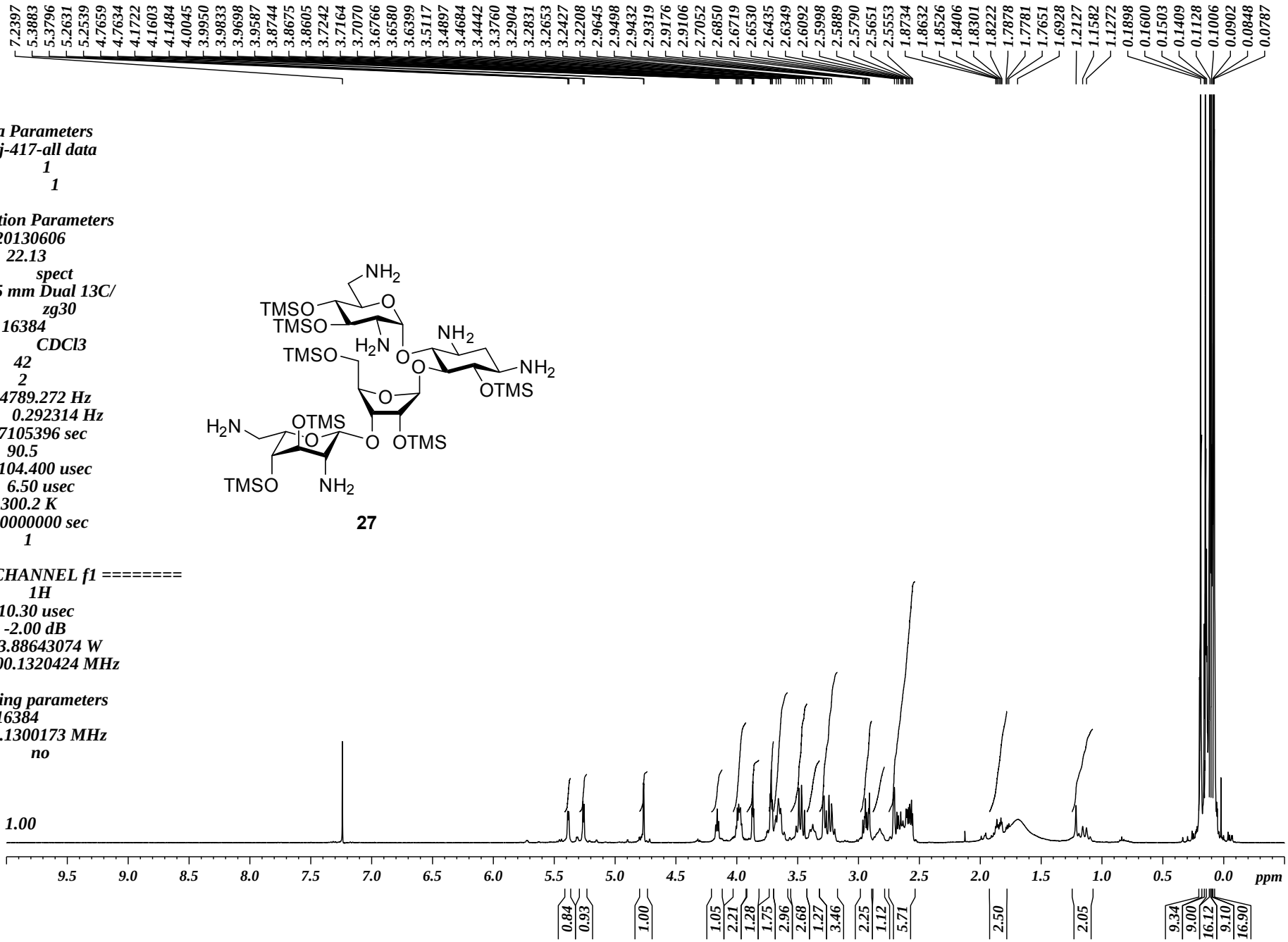

169.26
153.92
139.94
136.73
129.68
126.15
95.56
87.52
77.55
77.23
76.92
76.00
75.87
74.84
74.69
69.33
64.55
55.19
52.61
41.90
29.93
21.51
0.82
0.77
0.29
-0.17
Current Data Parameters
NAME aaj-360-all data
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121221
Time 22.16
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 3000
DS 0
SWH 23148.148 Hz
FIDRES 0.706425 Hz
AQ 0.7078388 sec
RG 2050
DW 21.600 usec
DE 8.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.80 usec
PL1 -2.00 dB
PL1W 55.33689499 W
SFO1 100.6238359 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 13.70 dB
PL13 16.70 dB
PL2W 16.12334061 W
PL12W 0.43396533 W
PL13W 0.21749784 W
SFO2 400.1326011 MHz
F2 - Processing parameters
SI 16384
SF 100.6127447 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
TMSO
OTMS
COOMe
O
STol
TrocHN
TMSO
OTMS
23
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

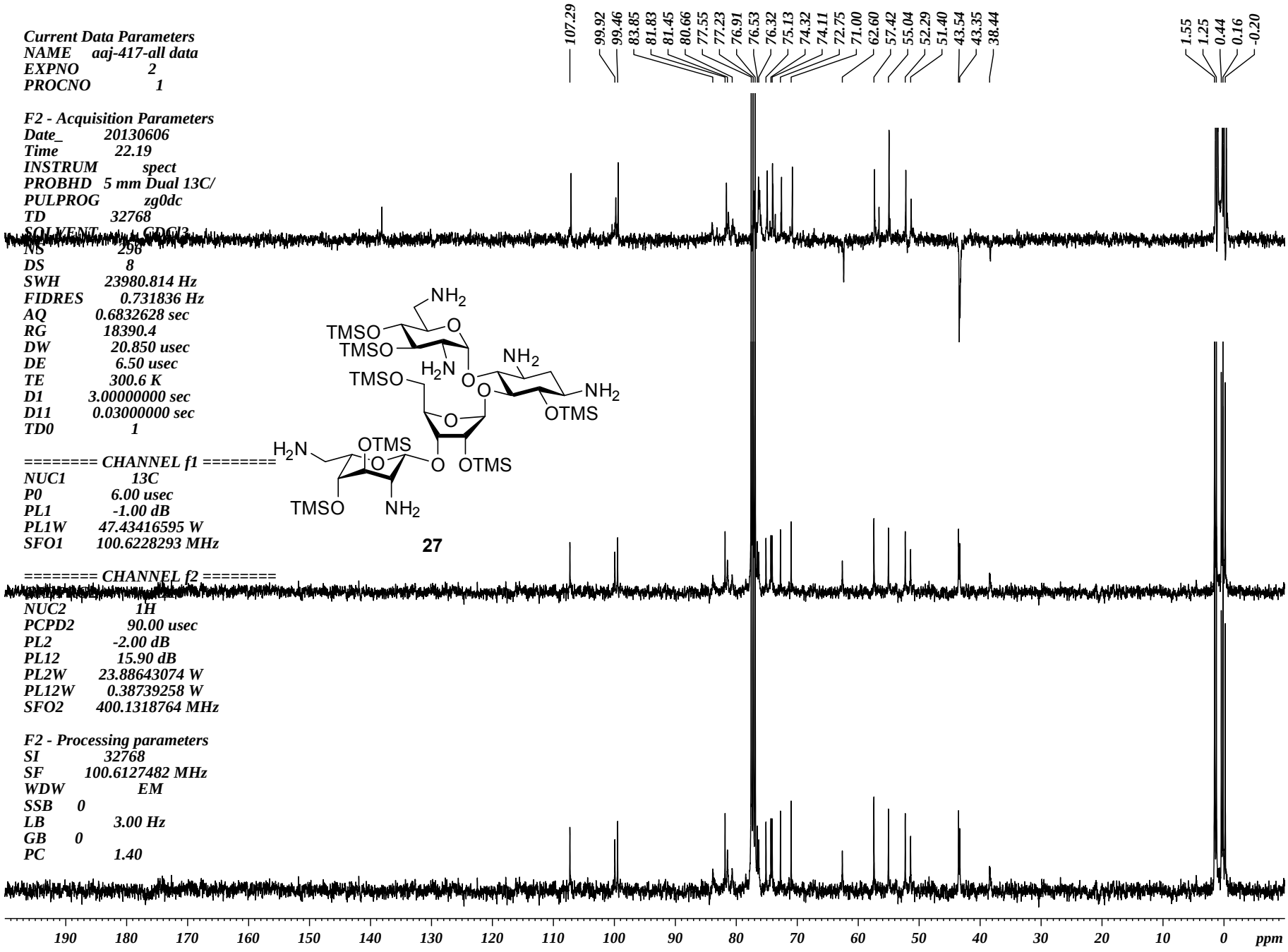

7.4370
7.4172
7.2400
7.1071
7.0874
6.6683
6.6483
4.0521
4.0441
4.0261
4.0180
3.9291
3.9187
3.9141
3.9022
3.8927
3.8773
3.8661
3.8468
3.8387
3.8292
3.8197
3.8116
3.7603
3.7541
3.6137
3.5136
3.4915
3.4664
3.4458
2.6727
2.6618
2.6406
2.6297
2.3336
2.0201
1.7618
1.7318
1.7014
1.6557
1.3718
1.2348
1.2177
1.2004
1.1911
1.1169
0.1473
0.1199
0.0721
0.0663
Current Data Parameters
NAME aaj-361
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121220
Time 15.09
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 92
DS 0
SWH 5597.015 Hz
FIDRES 0.341615 Hz
AQ 1.4636873 sec
RG 80.6
DW 89.333 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.40 usec
PL1 -2.00 dB
PL1W 16.12334061 W
SFO1 400.1326012 MHz
F2 - Processing parameters
SI 8192
SF 400.1300175 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
TMSO
OTMS
COOMe
TCANH
O
STol
TMSO
OTMS
24
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
2.0
1.5
1.0
0.5
0.0
ppm
1.97
1.97
0.95
0.98
2.03
1.05
1.00
2.96
2.07
1.00
3.04
1.11
8.79
8.79
17.36


169.22
161.46
140.06
136.74
129.71
125.97
92.91
87.53
77.55
77.23
76.91
75.63
75.43
75.33
68.91
64.16
60.60
55.83
52.75
41.81
29.91
21.51
21.25
14.41
0.86
0.75
0.47
0.07
-0.23
Current Data Parameters
NAME aaj-361-R-all
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130220
Time 2.28
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg0dc
TD 32768
SOLVENT CDCl3
NS 3500
DS 8
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 0.6832628 sec
RG 16384
DW 20.850 usec
DE 6.50 usec
TE 299.0 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P0 6.00 usec
PL1 -1.00 dB
PL1W 47.43416595 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 15.90 dB
PL2W 23.88643074 W
PL12W 0.38739258 W
SFO2 400.1318764 MHz
F2 - Processing parameters
SI 32768
SF 100.6127475 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
TMSO
OTMS
COOMe
TCANH
O
STol
TMSO
OTMS
24
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.4187 7.3985 7.2402 7.0946 7.0748 4.1094 4.1045 4.0930 4.0826 4.0782 3.8592 3.8498 3.8402 3.8357 3.8307 3.8260 3.8201 3.8153 3.8105 3.8058 3.6458 3.6314 3.6257 3.6199 3.6138 3.6085 3.5973 3.5911 3.5804 3.5585 3.5226 3.5021 3.4961 3.4759 3.2782 3.2523 3.2424 3.2356 2.6118 2.6006 2.5794 2.5682 2.3268 1.7665 1.7378 1.7342 1.7056 1.5674 0.1821 0.1485 0.1317 0.0578
Current Data Parameters
NAME aaj-358-all data
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121217
Time 22.22
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 200
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7105396 sec
RG 228.1
DW 104.400 usec
DE 6.50 usec
TE 298.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.30 usec
PL1 -2.00 dB
PL1W 23.88643074 W
SFO1 400.1320424 MHz
F2 - Processing parameters
SI 16384
SF 400.1300170 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
TMSO
OTMS
COOMe
STol
N3
TMSO
OTMS
25
11.0 10.5 10.0 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 2.0 1.5 1.0 0.5 0.0 ppm
1.95 1.94 1.09 2.04 1.26 2.86 0.99 1.96 1.00 3.01 1.05 8.84 8.71 8.15 8.35


168.94
140.00
136.69
129.71
126.02
87.62
77.55
77.23
76.92
76.71
75.56
75.12
71.91
64.92
62.89
52.65
41.18
21.52
0.82
0.13
-0.10
Current Data Parameters
NAME aaj-358-all data
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121218
Time 2.10
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg0dc
TD 32768
SOLVENT CDCl3
NS 3500
DS 8
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 0.6832628 sec
RG 20642.5
DW 20.850 usec
DE 6.50 usec
TE 299.1 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P0 6.00 usec
PL1 -1.00 dB
PL1W 47.43416595 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 15.90 dB
PL2W 23.88643074 W
PL12W 0.38739258 W
SFO2 400.1318764 MHz
F2 - Processing parameters
SI 32768
SF 100.6127460 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
TMSO
OTMS
COOMe
O
STol
N3
TMSO
OTMS
25
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


7.2397
5.3883
5.3796
5.2631
5.2539
4.7659
4.7634
4.1722
4.1603
4.1484
4.0045
3.9950
3.9833
3.9698
3.9587
3.8744
3.8675
3.8605
3.7242
3.7164
3.7070
3.6766
3.6580
3.6399
3.5117
3.4897
3.4684
3.4442
3.3760
3.2904
3.2831
3.2653
3.2427
3.2208
2.9645
2.9498
2.9432
2.9319
2.9176
2.9106
2.7052
2.6850
2.6719
2.6530
2.6435
2.6349
2.6092
2.5998
2.5889
2.5790
2.5651
2.5553
1.8734
1.8632
1.8526
1.8406
1.8301
1.8222
1.7878
1.7781
1.7651
1.6928
1.2127
1.1582
1.1272
0.1898
0.1600
0.1503
0.1409
0.1128
0.1006
0.0902
0.0848
0.0787
Current Data Parameters
NAME aaj-417-all data
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130606
Time 22.13
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 42
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7105396 sec
RG 90.5
DW 104.400 usec
DE 6.50 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.30 usec
PL1 -2.00 dB
PL1W 23.88643074 W
SFO1 400.1320424 MHz
F2 - Processing parameters
SI 16384
SF 400.1300173 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
NH2
TMSO
TMSO
O
H2N
NH2
TMSO
O
O
NH2
OTMS
O
H2N
OTMS
O
O
OTMS
TMSO
NH2
27
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
2.0
1.5
1.0
0.5
0.0
ppm
0.84
0.93
1.00
1.05
2.21
1.28
1.75
2.96
2.68
1.27
3.46
2.25
1.12
5.71
2.50
2.05
9.34
9.00
16.12
9.10
16.90


107.29
99.92
99.46
83.85
81.83
81.45
80.66
77.55
77.23
76.91
76.53
76.32
75.13
74.32
74.11
72.75
71.00
62.60
57.42
55.04
52.29
51.40
43.54
43.35
38.44
1.55
1.25
0.44
0.16
-0.20
Current Data Parameters
NAME aaj-417-all data
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130606
Time 22.19
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg0dc
TD 32768
SOLVENT CDCl3
NS 256
DS 8
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 0.6832628 sec
RG 18390.4
DW 20.850 usec
DE 6.50 usec
TE 300.6 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P0 6.00 usec
PL1 -1.00 dB
PL1W 47.43416595 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
SFO2 400.1318764 MHz
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 15.90 dB
PL2W 23.88643074 W
PL12W 0.38739258 W
SFO2 400.1318764 MHz
F2 - Processing parameters
SI 32768
SF 100.6127482 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
NH2
TMSO
TMSO
O
H2N
O
NH2
TMSO
O
O
NH2
OTMS
O
H2N
OTMS
O
O
OTMS
TMSO
NH2
27
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

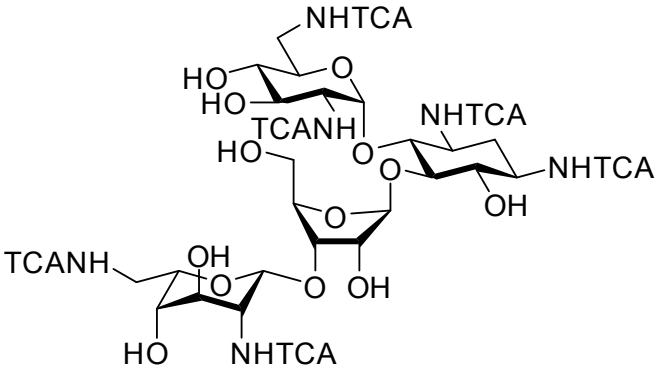
8.8114 8.7969 8.7450 8.7245 8.6754 8.6568 8.6402 8.6170 8.4402 8.1489 8.1403 8.1276 5.4754 5.1656 5.1580 5.0450 5.0420 4.7803 4.2370 4.2231 4.2089 4.0964 4.0799 4.0660 4.0404 4.0329 4.0275 4.0192 3.9356 3.9293 3.9162 3.9088 3.9011 3.8844 3.8764 3.8648 3.8535 3.8397 3.8162 3.7918 3.7343 3.7275 3.7036 3.6975 3.6867 3.6714 3.6497 3.6241 3.5973 3.5838 3.5759 3.5646 3.5533 3.5445 3.5244 3.5111 3.5026 3.2752 3.2443 3.2402 3.2364 3.2322 3.2283 3.2104 3.2024 1.9335 1.9111 1.9015 1.7019 1.6716 1.2150 0.1480 0.1398 0.0359

Current Data Parameters
NAME aaj-428-all
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130622
Time 23.12
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT MeOD
NS 200
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7105396 sec
RG 181
DW 104.400 usec
DE 6.50 usec
TE 298.5 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.30 usec
PL1 -2.00 dB
PL1W 23.88643074 W
SFO1 400.1320424 MHz

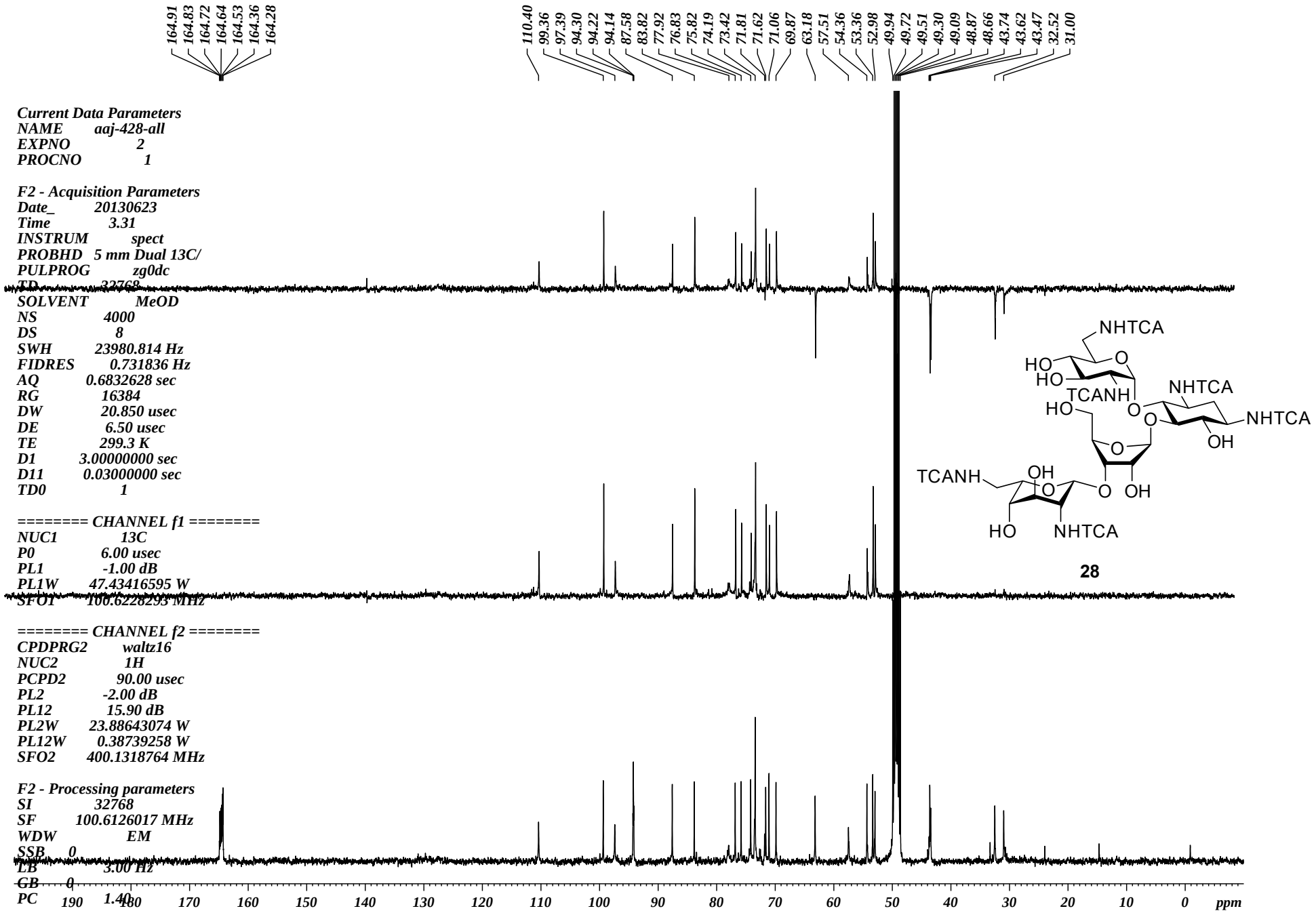
F2 - Processing parameters
SI 16384
SF 400.1300364 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

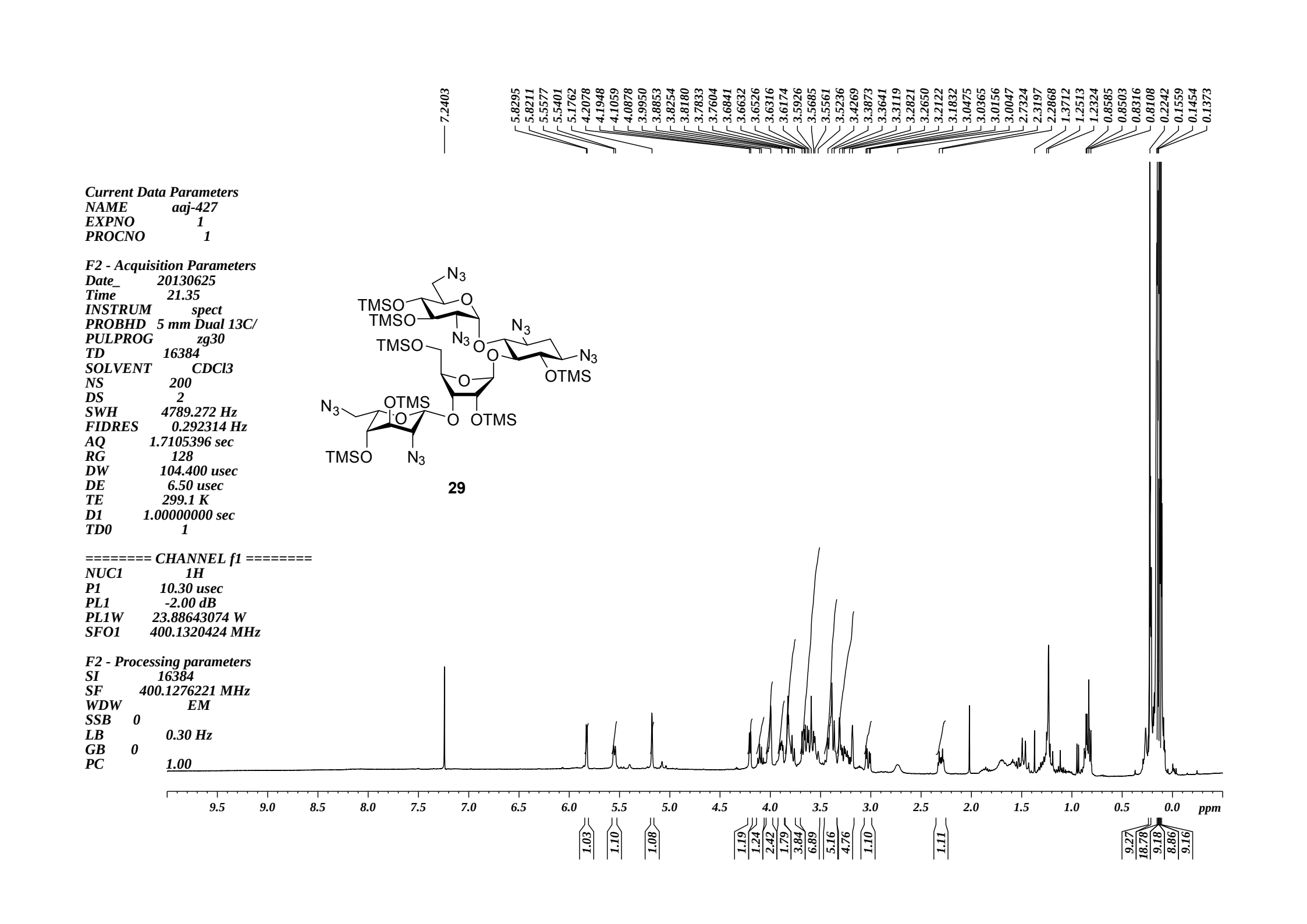


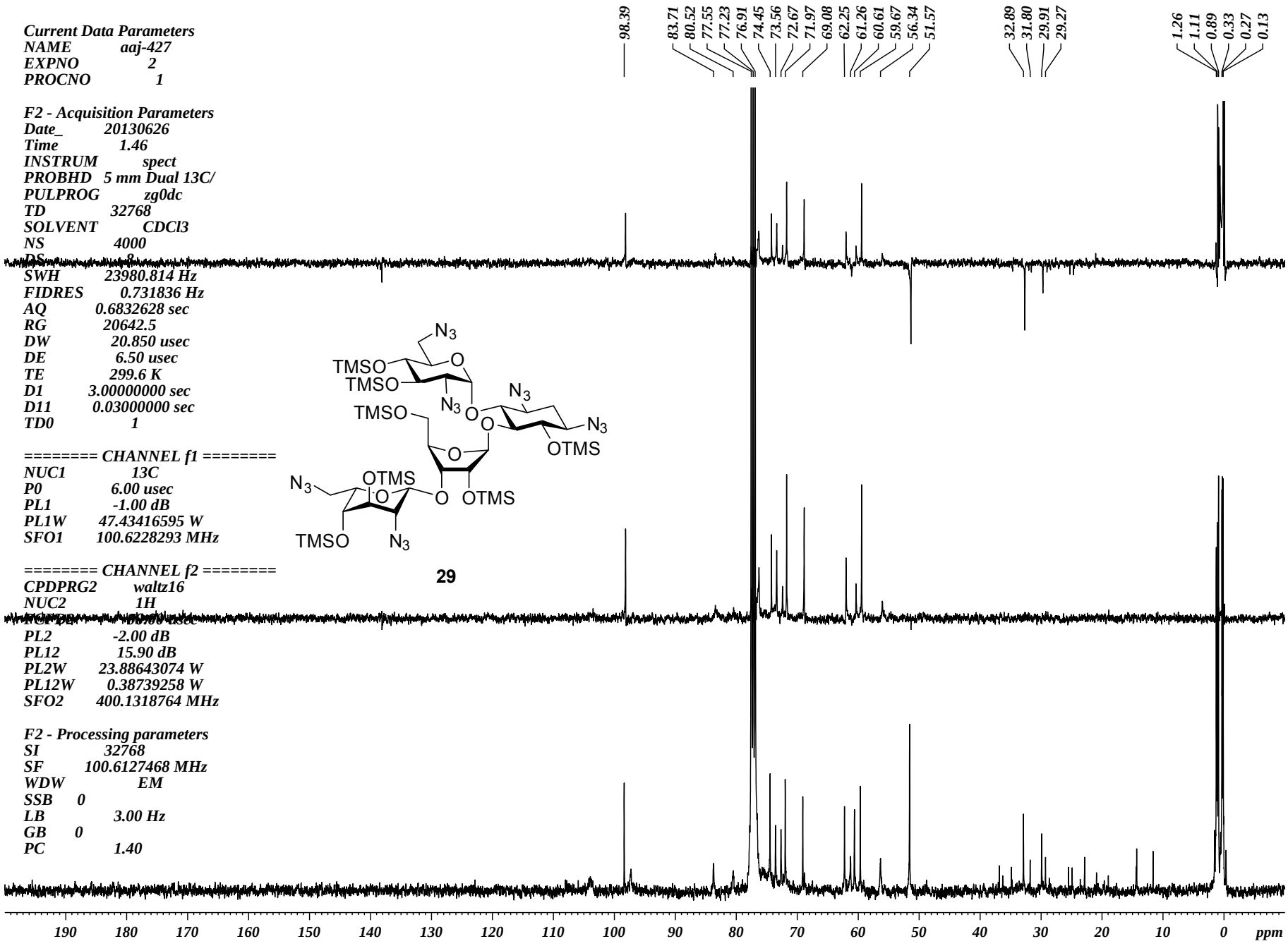
28

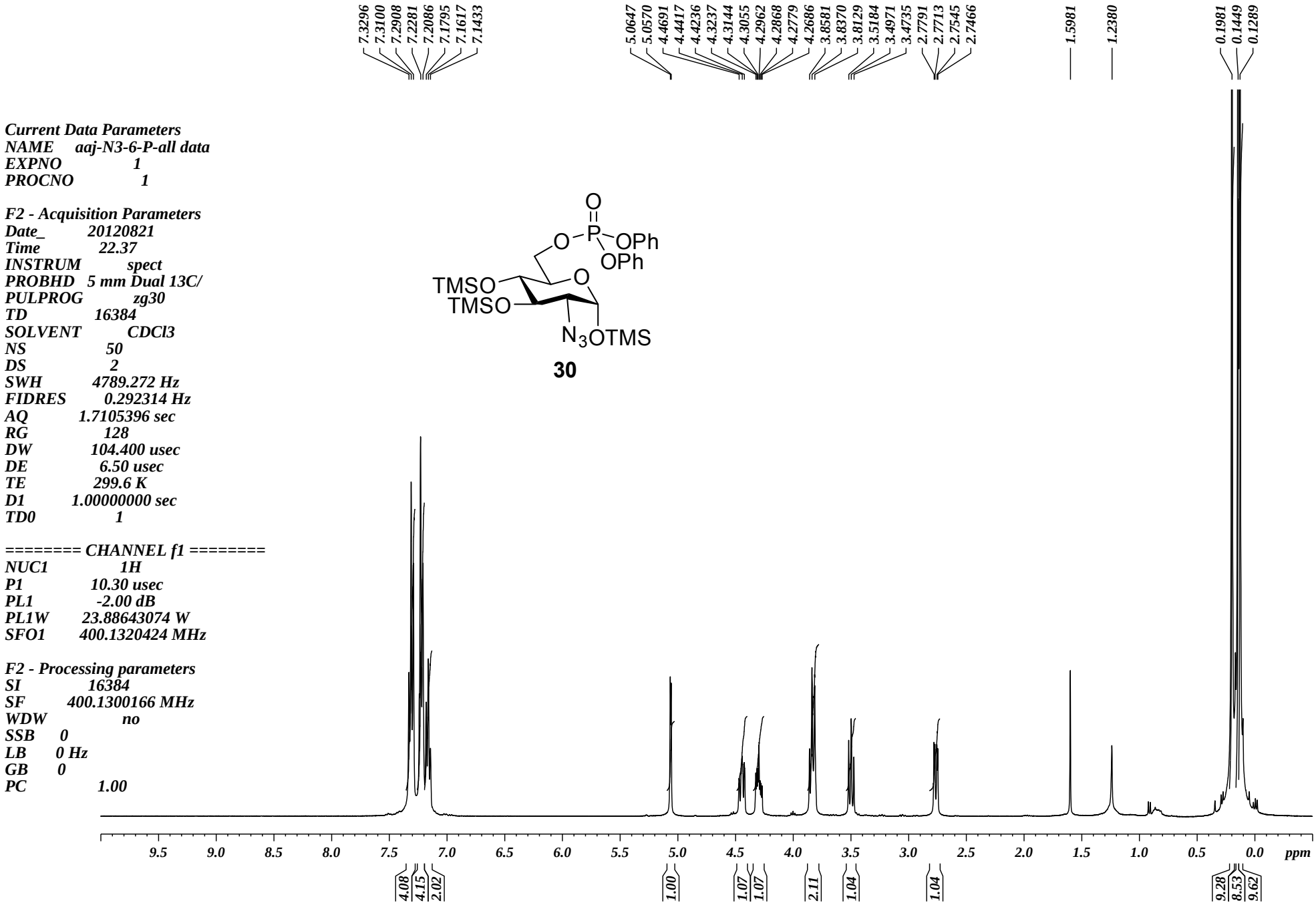
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 *ppm*

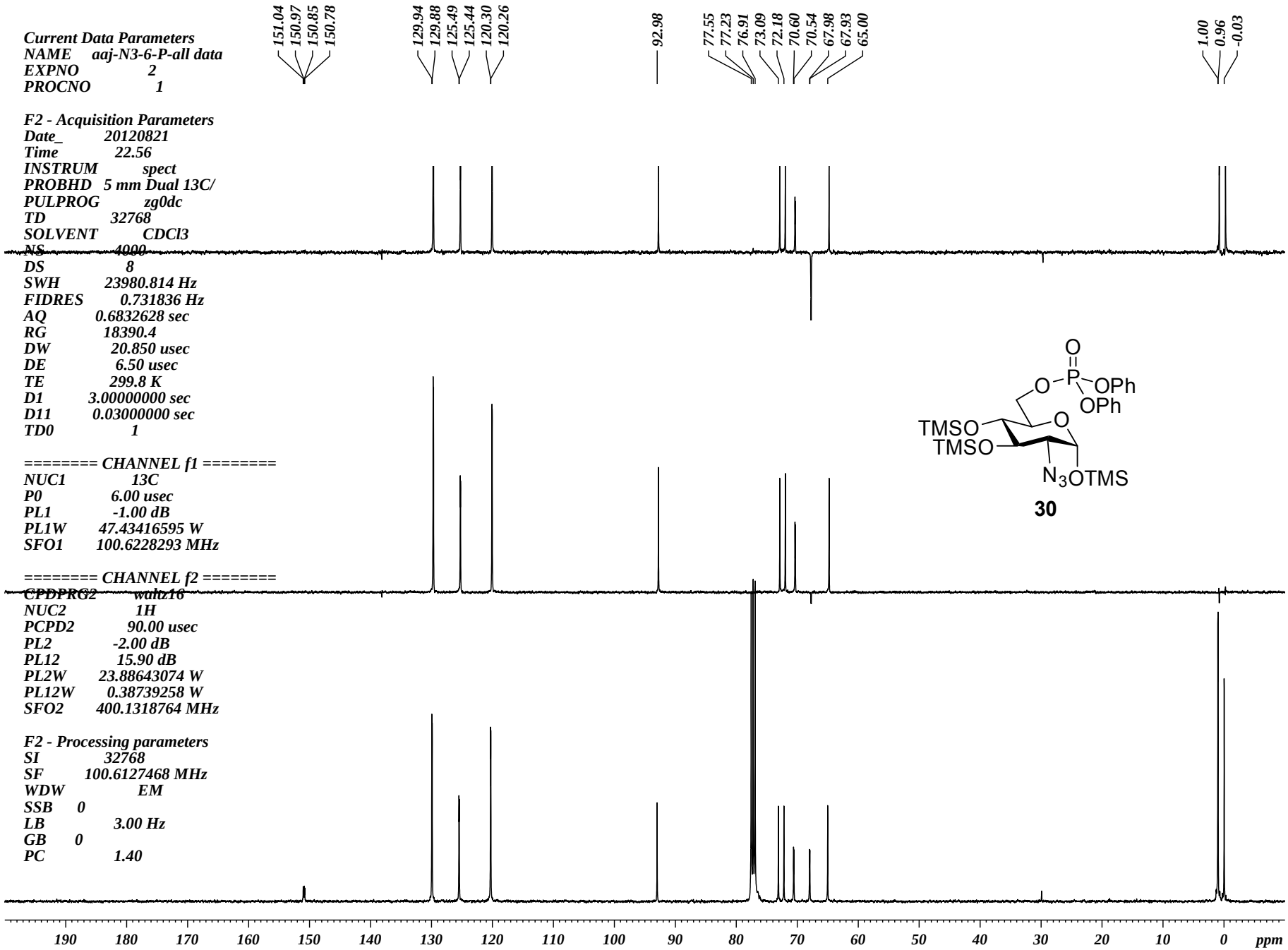
1.14 1.08 2.10 1.14 1.35 1.22 1.48 1.31 1.37 4.70 10.70 6.77 10.14 0.74 1.59 1.71


164.91
164.83
164.72
164.64
164.53
164.36
164.28
110.40
99.36
97.39
94.30
94.22
94.14
87.58
83.82
77.92
76.83
75.82
74.19
73.42
71.81
71.62
71.06
69.87
63.18
57.51
54.36
53.36
52.98
49.94
49.72
49.51
49.30
49.09
48.87
48.66
43.74
43.62
43.47
32.52
31.00
Current Data Parameters
NAME aaj-428-all
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130623
Time 3.31
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg0dc
TD 32768
SOLVENT MeOD
NS 4000
DS 8
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 0.6832628 sec
RG 16384
DW 20.850 usec
DE 6.50 usec
TE 299.3 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P0 6.00 usec
PL1 -1.00 dB
PL1W 47.43416595 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 15.90 dB
PL2W 23.88643074 W
PL12W 0.38739258 W
SFO2 400.1318764 MHz
F2 - Processing parameters
SI 32768
SF 100.6126017 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
NHTCA
HO
HO
TCANH
O
NHTCA
HO
O
O
NHTCA
OH
O
TCANH
OH
O
O
OH
HO
NHTCA
28
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


7.2403
5.8295
5.8211
5.5577
5.5401
5.1762
4.2078
4.1948
4.1059
4.0878
3.9950
3.8853
3.8254
3.8180
3.7833
3.7604
3.6841
3.6632
3.6526
3.6316
3.6174
3.5926
3.5685
3.5561
3.5236
3.4269
3.3873
3.3641
3.3119
3.2821
3.2650
3.2122
3.1832
3.0475
3.0365
3.0156
3.0047
2.7324
2.3197
2.2868
1.3712
1.2513
1.2324
0.8585
0.8503
0.8316
0.8108
0.2242
0.1559
0.1454
0.1373
Current Data Parameters
NAME aaj-427
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130625
Time 21.35
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 200
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7105396 sec
RG 128
DW 104.400 usec
DE 6.50 usec
TE 299.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.30 usec
PL1 -2.00 dB
PL1W 23.88643074 W
SFO1 400.1320424 MHz
F2 - Processing parameters
SI 16384
SF 400.1276221 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
N3
TMSO
TMSO
O
N3
O
N3
TMSO
O
OTMS
N3
OTMS
O
O
OTMS
TMSO
N3
29
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
2.0
1.5
1.0
0.5
0.0
ppm
1.03
1.10
1.08
1.19
1.24
2.42
1.79
3.84
6.89
5.16
4.76
1.10
1.11
9.27
18.78
9.18
8.86
9.16


98.39
83.71
80.52
77.55
77.23
76.91
74.45
73.56
72.67
71.97
69.08
62.25
61.26
60.61
59.67
56.34
51.57
32.89
31.80
29.91
29.27
1.26
1.11
0.89
0.33
0.27
0.13
Current Data Parameters
NAME aaj-427
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130626
Time 1.46
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg0dc
TD 32768
SOLVENT CDCl3
NS 4000
DS 8
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 0.6832628 sec
RG 20642.5
DW 20.850 usec
DE 6.50 usec
TE 299.6 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P0 6.00 usec
PL1 -1.00 dB
PL1W 47.43416595 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 15.90 dB
PL2W 23.88643074 W
PL12W 0.38739258 W
SFO2 400.1318764 MHz
F2 - Processing parameters
SI 32768
SF 100.6127468 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
N3
TMSO
TMSO
O
N3
O
N3
TMSO
O
N3
OTMS
O
N3
OTMS
O
OTMS
TMSO
N3
29
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


7.3296
7.3100
7.2908
7.2281
7.2086
7.1795
7.1617
7.1433
5.0647
5.0570
4.4691
4.4417
4.4236
4.3237
4.3144
4.3055
4.2962
4.2868
4.2779
4.2686
3.8581
3.8370
3.8129
3.5184
3.4971
3.4735
2.7791
2.7713
2.7545
2.7466
1.5981
1.2380
0.1981
0.1449
0.1289
Current Data Parameters
NAME aaj-N3-6-P-all data
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120821
Time 22.37
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 50
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7105396 sec
RG 128
DW 104.400 usec
DE 6.50 usec
TE 299.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.30 usec
PL1 -2.00 dB
PL1W 23.88643074 W
SFO1 400.1320424 MHz
F2 - Processing parameters
SI 16384
SF 400.1300166 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
O
O
P
OPh
OPh
O
TMSO
TMSO
N3OTMS
30
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
2.0
1.5
1.0
0.5
0.0
ppm
4.08
4.15
2.02
1.00
1.07
1.07
2.11
1.04
1.04
9.28
8.53
9.62


151.04
150.97
150.85
150.78
129.94
129.88
125.49
125.44
120.30
120.26
92.98
77.55
77.23
76.91
73.09
72.18
70.60
70.54
67.98
67.93
65.00
1.00
0.96
-0.03
Current Data Parameters
NAME aaj-N3-6-P-all data
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120821
Time 22.56
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg0dc
TD 32768
SOLVENT CDCl3
NS 4000
DS 8
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 0.6832628 sec
RG 18390.4
DW 20.850 usec
DE 6.50 usec
TE 299.8 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P0 6.00 usec
PL1 -1.00 dB
PL1W 47.43416595 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 15.90 dB
PL2W 23.88643074 W
PL12W 0.38739258 W
SFO2 400.1318764 MHz
F2 - Processing parameters
SI 32768
SF 100.6127468 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
O
P
O
OPh
OPh
TMSO
TMSO
O
N3OTMS
30
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

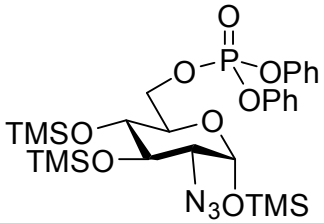
Current Data Parameters
NAME aaj-n3-6p-31p
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130912
Time 22.13
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 38
DS 0
SWH 73529.414 Hz
FIDRES 2.243940 Hz
AQ 0.2228724 sec
RG 645
DW 6.800 usec
DE 6.50 usec
TE 300.0 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1

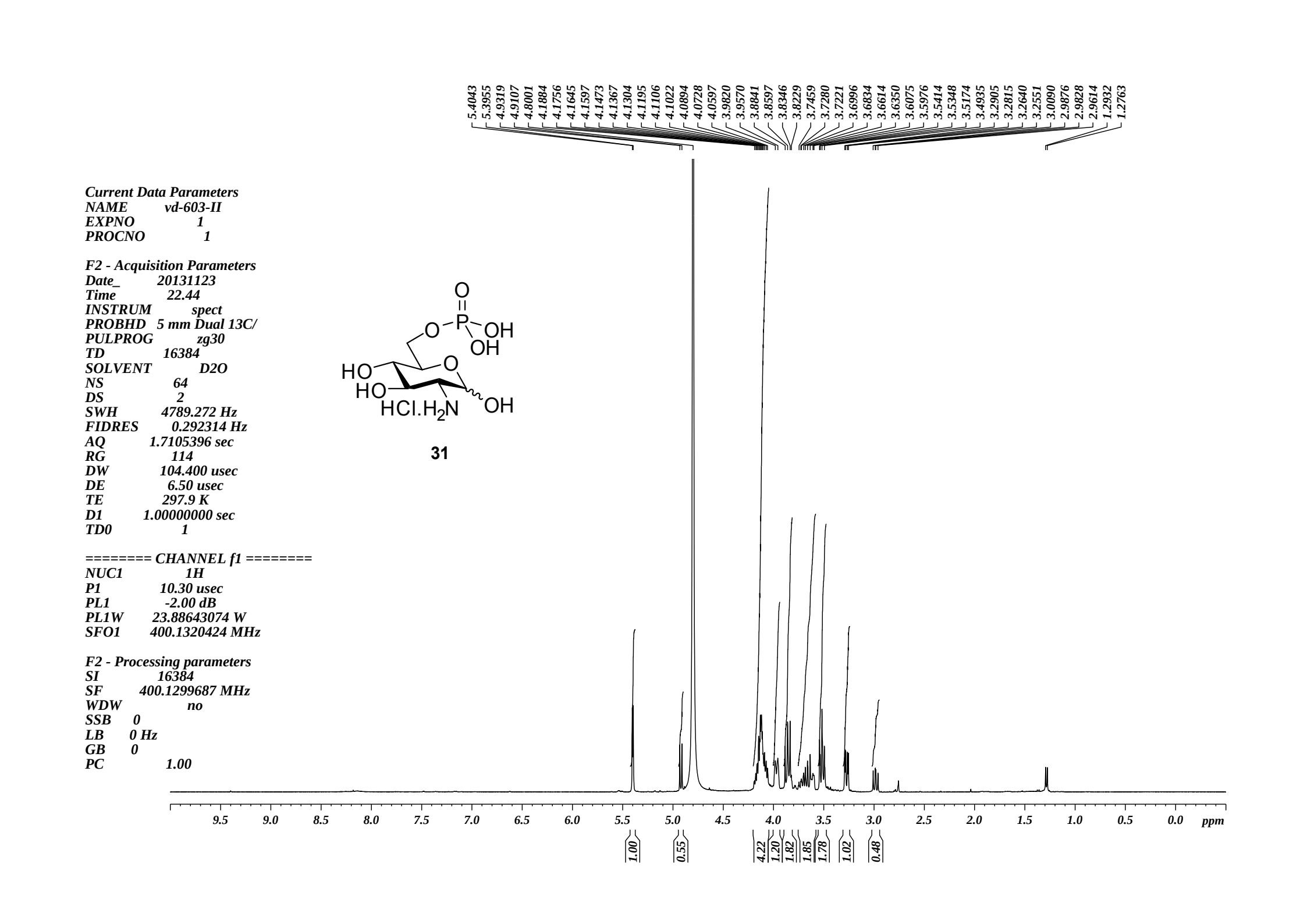
======== CHANNEL f1 ========
NUC1 31P
P1 10.10 usec
PL1 9.00 dB
PL1W 8.75950909 W
SFO1 121.4948509 MHz

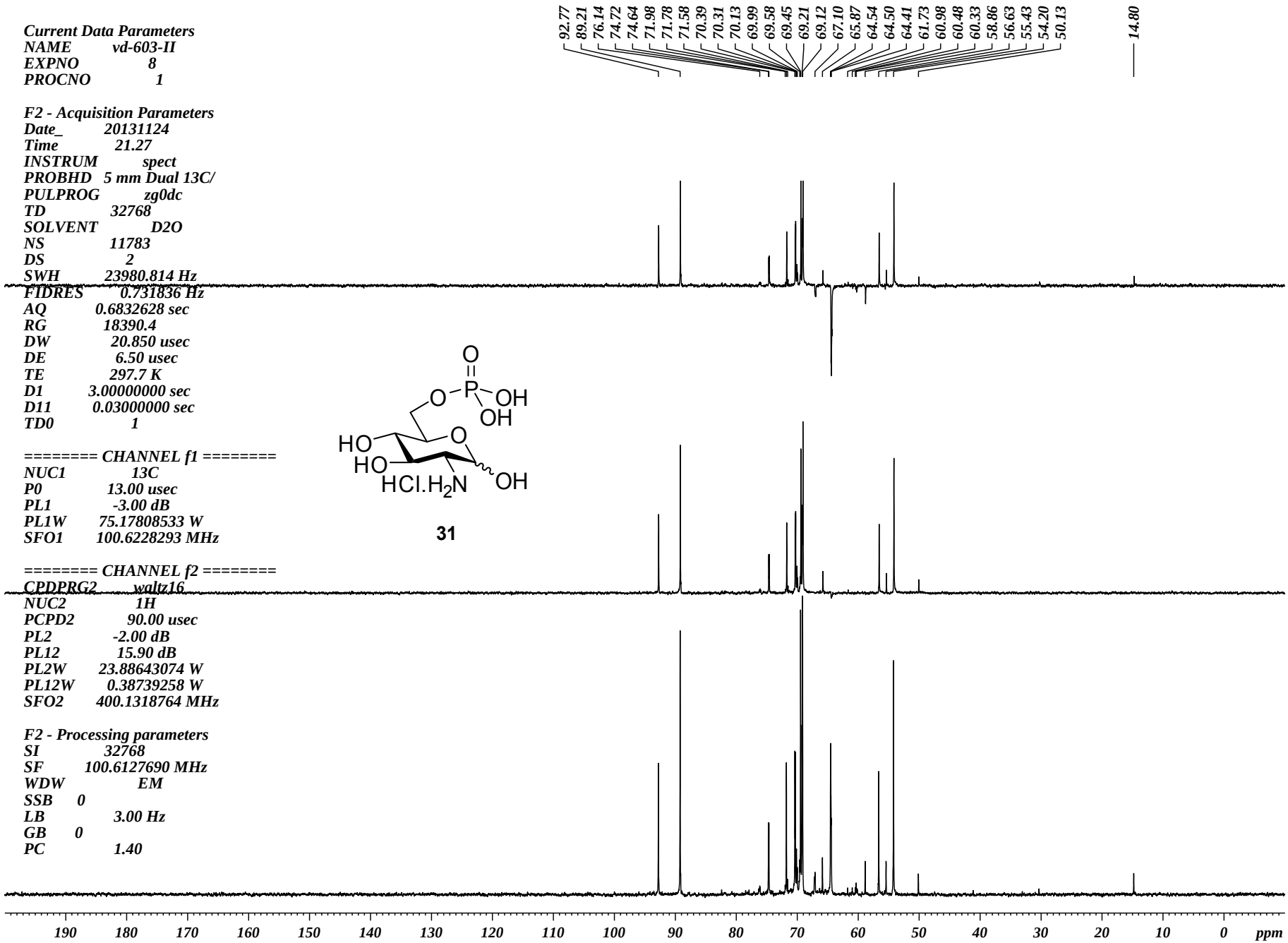
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -4.00 dB
PL12 14.50 dB
PL13 17.50 dB
PL2W 26.37401772 W
PL12W 0.37254289 W
PL13W 0.18671374 W
SFO2 300.1319510 MHz

F2 - Processing parameters
SI 32768
SF 121.4948510 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

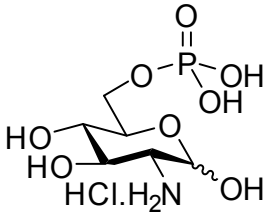


30


5.4043
5.3955
4.9319
4.9107
4.8001
4.1884
4.1756
4.1645
4.1597
4.1473
4.1367
4.1304
4.1195
4.1106
4.1022
4.0894
4.0728
4.0597
3.9820
3.9570
3.8841
3.8597
3.8346
3.8229
3.7459
3.7280
3.7221
3.6996
3.6834
3.6614
3.6350
3.6075
3.5976
3.5414
3.5348
3.5174
3.4935
3.2905
3.2815
3.2640
3.2551
3.0090
2.9876
2.9828
2.9614
1.2932
1.2763
Current Data Parameters
NAME vd-603-II
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131123
Time 22.44
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT D2O
NS 64
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7105396 sec
RG 114
DW 104.400 usec
DE 6.50 usec
TE 297.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.30 usec
PL1 -2.00 dB
PL1W 23.88643074 W
SFO1 400.1320424 MHz
F2 - Processing parameters
SI 16384
SF 400.1299687 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
O
P
O
OH
OH
HO
HO
O
HCl.H2N
OH
31
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
2.0
1.5
1.0
0.5
0.0
ppm
1.00
0.55
4.22
1.20
1.82
1.85
1.78
1.02
0.48


92.77
89.21
76.14
74.72
74.64
71.98
71.78
71.58
70.39
70.31
70.13
69.99
69.58
69.45
69.21
69.12
67.10
65.87
64.54
64.50
64.41
61.73
60.98
60.48
60.33
58.86
56.63
55.43
54.20
50.13
14.80
Current Data Parameters
NAME vd-603-II
EXPNO 8
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131124
Time 21.27
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg0dc
TD 32768
SOLVENT D2O
NS 11783
DS 2
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 0.6832628 sec
RG 18390.4
DW 20.850 usec
DE 6.50 usec
TE 297.7 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P0 13.00 usec
PL1 -3.00 dB
PL1W 75.17808533 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 15.90 dB
PL2W 23.88643074 W
PL12W 0.38739258 W
SFO2 400.1318764 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
O
P
O
OH
OH
HO
O
HO
HCl.H2N
OH
31
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

NAME vd-603
EXPNO 2
PROCNO 1
Date_ 20131115
Time 17.08
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT D2O
NS 29
DS 0
SWH 104166.664 Hz
FIDRES 1.589457 Hz
AQ 0.3146228 sec
RG 2050
DW 4.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1



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======== CHANNEL f1 ========
NUC1 31P
P1 13.50 usec
PL1 2.00 dB
PL1W 16.00742149 W
SFO1 161.9755930 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 14.20 dB
PL13 17.20 dB
PL2W 16.12334061 W
PL12W 0.38677201 W
PL13W 0.19384515 W
SFO2 400.1320007 MHz
SI 32768
SF 161.9755127 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00