Direct synthesis of methyl phosphoramidates in carbohydrates

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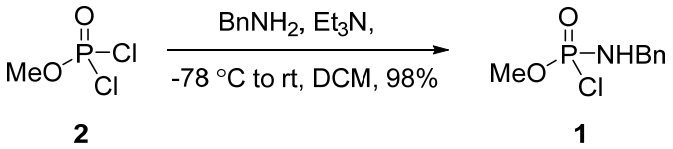
SUPPORTING INFORMATION

General Information

All reactions were conducted in flame-dried glassware, under nitrogen atmosphere. Methanol, acetonitrile and dichloromethane were purified and dried by using a safe purification system filled with anhydrous Al_2O_3. All other reagents were obtained from commercial sources and used without further purification unless otherwise mentioned. Water was either distilled or Mili-Q-purified. Flash column chromatography was carried out on Silica Gel 60 (230-400 mesh, E. Merck). TLC was performed on re-coated glass plates of Silica Gel 60 F254 (0.25 mm, E. Merck); detection was executed by spraying a solution of $Ce(NH_4)_2(NO_3)_6$, $(NH_4)_6Mo_7O_{24}$, and H_2SO_4 in water and subsequent heating on a hot plate. Specific rotations were taken at ambient temperature conditions and reported in $10^{-1}\cdot deg\cdot cm^2\cdot g^{-1}$; the sample concentrations are in $g\cdot dL^{-1}$. ^1H and ^{13}C NMR spectra were recorded on AV 400 MHz, AVIII 400 and DRX 500 MHz Bruker instruments. Chemical shifts are in ppm from Me_4Si, calibrated at δ 7.24 or δ 0.00 for ^1H spectra (residual $CHCl_3$ or TMS respectively), and δ 77.23 for ^{13}C spectra. Splitting patterns were designated as follows: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad. Proton peak were assigned based on 2D NMR spectra (^1H-^1H COSY, HSQC, and NOESY). For the NMR spectrum of diastereomeric mixtures, the assignment of one of the two diastereomers was indicated by "*".

Synthetic procedures and characterization data

Preparation of phosphorylating agent 1



2 **1**

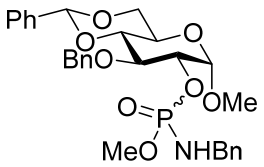
Methyl benzylphosphoramidochloridate (1).

To the solution of benzylamine (710 mg, 740 μL, 6.71 mmol) in dry CH_2Cl_2 was added triethylamine (680 mg, 940 μL, 6.71 mmol) at rt. The mixture was cooled to -78 ^oC. Methyl dichlorophosphate (1.0 g, 670 μL, 6.71 mmol) was added dropwise to the mixture over 20 min. After complete addition, the temperature was raised to rt and stirred for additional 1 h, and the reaction was monitored by TLC using Nihydrin as the stain. Upon completion, the CH_2Cl_2 was evaporated under reduced pressure and a white solid was obtained, which was re-suspended with diethyl ether. The undesired solid was filtered off, and the diethyl ether filtrate was concentrated under a reduced pressure to afford **1** as a colorless oil (1.45 g, 98%); IR ($CHCl_3$) ν 3199, 1496, 1252, 1026, 732, 695 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.32-7.25 (m, 5H, ArH), 4.15 (d, J = 11.6 Hz, 2H, $PhCH_2$), 3.81 (d, J = 13.7 Hz, 3H, OCH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.8 (d, J = 8.5 Hz, C), 128.6 (CH x 2), 127.7 (CH), 127.4 (CH x 2), 54.0 (d, J = 6.1 Hz, CH_3), 45.7 (CH_2); ^{31}P NMR (161.97 MHz, $CDCl_3$) δ 16.8; HRMS (ESI) calcd for $C_8H_{12}NO_2ClP$ $[M+H]^+$ 220.0294, found 220.0290.

NOTE: This reagent is stable only for 5-6h. Prepare freshly before use.

General procedure for direct synthesis of methyl phosphoramidates:

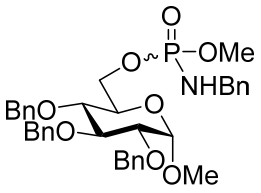
To a solution of an alcohol (100 mg) and DMAP (1.0 equiv) in dry CH_2Cl_2 (2 mL) was added NMI (*N*-methylimidazole) (8.0 equiv) at rt. Methyl benzylphosphoramidochloridate (**1**) (4.0 equiv) in CH_2Cl_2 (1 mL) was added dropwise over a period of 2-3 min at rt. The reaction mixture was allowed to stir at rt as the time indicated in Table 2. The reaction was monitored by TLC, and upon completion, the volatiles were removed *in vacuo*. The residue was purified by column chromatography to afford the desired product in good to excellent yield.



4

Methyl 3-*O*-benzyl-4,6-*O*-benzylidene-2-*O*-(methyl *N*-benzylphosphoramidyl)-α-D-glucopyranoside (4).

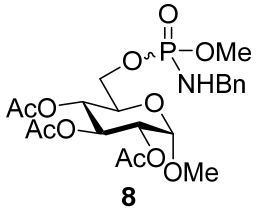
Colorless oil, $[\alpha]^{26}_{D}$ 40.1 (*c* 0.5, $CHCl_3$); IR ($CHCl_3$) ν 2935, 2866, 1496, 1177, 1072, 1050, 994, 934, 697, 675 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.47-7.44 (m, 2H, ArH), 7.47-7.44 (m, 2H, ArH*), 7.38-7.30 (m, 4H, ArH), 7.38-7.30 (m, 4H, ArH*), 7.29-7.18 (m, 8H, ArH), 7.29-7.18 (m, 8H, ArH*), 7.11-7.09 (m, 1H, ArH), 7.11-7.09 (m, 1H, ArH*), 5.56 (s, 1H, PhCH), 5.55 (s, 1H, PhCH*), 5.04 (d, *J* = 3.6 Hz, 1H, H-1), 4.99 (d, *J* = 3.6 Hz, 1H, H-1*), 4.94 (d, *J* = 11.1 Hz, 1H, CH_2Ph), 4.89 (d, *J* = 11.2 Hz, 1H, CH_2Ph*), 4.71 (d, *J* = 11.2 Hz, 1H, CH_2Ph), 4.63 (d, *J* = 11.2 Hz, CH_2Ph), 4.38 (td, *J* = 10.2, 5.0 Hz, 4H, H-2, H-6a, H-2*, H-6a*), 4.0 (td, *J* = 9.2, 2.8 Hz, 2H, H-3, H-3*) 3.95 (dd, *J* = 9.4, 7.0 Hz, 2H, CH_2, CH_2*), 3.90-3.82 (m, 2H, H-5, H-5*), 3.78-3.71 (m, 2H, H-6b, H-6b*), 3.66 (d, *J* = 11.4 Hz, 3H, CH_3), 3.65-3.63 (m, 2H, H-4, H-4*), 3.61 (d, *J* = 11.2 Hz, 3H, CH_3*), 3.45 (s, 3H, OCH_3), 3.37 (s, 3H, OCH_3*), 3.04-2.99 (m, 2H, *N*H, *N*H*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.5 (C), 139.4 (C), 139.3 (C), 138.3 (C*); 138.1 (C*), 137.2 (C*), 128.9 (CH x 2), 128.5 (CH), 128.4 (CH x 3), 128.3 (CH x 3), 128.2 (CH x 3), 128.2 (CH x 3), 127.9 (CH x 3), 127.7 (CH x 2), 127.5 (CH), 127.3 (CH), 127.3 (CH x 3), 127.1 (CH x 2), 125.9 (CH x 3), 101.3 (PhCH, PhCH*), 99.1 (CH), 98.8 (CH*), 82.4 (CH), 82.1 (CH*), 74.8 (CH), 75.9 (CH*), 77.1 (CH), 77.0 (CH*), 74.8 (CH), 75.9 (CH*), 75.2 (CH_2), 69.0 (CH_2), 55.5 (CH_3), 55.3 (CH_3), 53.4 (CH_3, CH_3*), 53.3 (CH_3, CH_3*), 45.2 (CH_2), 45.2 (CH_2*); ^{31}P NMR (161.97 MHz, $CDCl_3$): δ 11.0, 9.5; HRMS (ESI) calcd for $C_{29}H_{34}NNaO_8P$ $[M+Na]^+$ 578.1920, found 578.1924.



6

Methyl 2,3,4-tri-*O*-benzyl-6-*O*-(methyl *N*-benzylphosphoramidyl)-α-D-glucopyranoside (6).

Colorless oil, $[\alpha]^{26}_D$ 27.9 (*c* 0.5, $CHCl_3$); IR ($CHCl_3$) ν 3214, 3029, 2909, 1495, 1243, 1070, 1049, 1017, 737, 697 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.34-7.20 (m, 20H, ArH,), 7.34-7.20 (m, 20H, ArH*), 4.96 (d, *J* = 11.0 Hz, 1H, CH_2Ph), 4.96 (d, *J* = 11.0 Hz, 1H, CH_2Ph*), 4.87-4.73 (m, 3H, CH_2Ph, CH_2Ph), 4.87-4.73 (m, 3H, CH_2Ph*, CH_2Ph*), 4.64-4.54 (m, 3H, CH_2Ph, H-1), 4.64-4.54 (m, 3H, CH_2Ph*, H-1*), 4.27-4.12 (m, 2H, H-6a, H-6b), 4.27-4.12 (m, 2H, H-6a*, H-6b*), 4.07 (d, *J* = 6.8 Hz, 2H, CH_2), 4.06 (d, *J* = 6.8 Hz, 2H, CH_2*), 3.98 (t, *J* = 9.4, 1H, H-3), 3.97 (t, *J* = 9.2, 1H, H-3*), 3.78-3.71 (m, 1H, H-5), 3.78-3.71 (m, 1H, H-5*), 3.68 (d, *J* = 11.0 Hz, 3H, CH_3), 3.65 (d, *J* = 11.4 Hz, 3H, CH_3*), 3.51 (t, *J* = 9.2 Hz, 1H, H-4), 3.51 (t, *J* = 9.2 Hz, 1H, H-4*), 3.48-3.42 (m, 1H, H-2), 3.48-3.42 (m, 1H, H-2*), 3.33 (s, 3H, OCH_3), 3.31 (s, 3H, OCH_3*), 2.97-2.86 (m, 1H, *N*H); 2.97-2.86 (m, 1H, *N*H*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.4 (C), 138.6 (C), 138.0 (C x 2), 128.5 (CH x 2, C x 1), 128.4 (CH x 3), 128.3 (CH x 3), 128.0 (CH x 2), 127.9 (CH x 4), 127.7 (CH x 2), 127.6 (CH), 127.4 (CH), 127.2 (CH x 2), 98.0 (CH), 98.0 (CH), 81.8 (CH), 79.8 (CH), 75.7 (CH_2 x 2), 75.0 (CH_2 x 2), 73.3 (CH_2 x 2), 69.6 (CH), 69.5 (CH), 65.3 (CH_2), 65.3 (CH_2), 55.2 (CH_3), 53.1 (CH_3), 45.3 (CH_2), 45.3 (CH_2*); ^{31}P NMR (161.97 MHz, $CDCl_3$): δ 10.6, 10.4; HRMS (ESI) calcd for $C_{36}H_{42}NNaO_8P$ $[M+Na]^+$ 670.2546, found 670.2551.

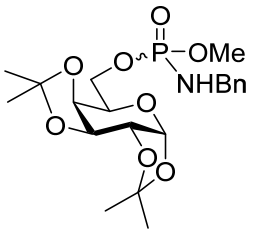


8

Methyl 2,3,4-tri-*O*-acetyl-6-*O*-(methyl *N*-benzylphosphoramidyl)-α-D-glucopyranoside (8).

Colorless oil (a 10:3 mixture of both diastereomers), $[\alpha]^{21}_D$ 107.3 (*c* 0.5, $CHCl_3$); IR ($CHCl_3$) ν 2923, 1733, 1716, 1683, 1540, 1215, 10656, 1025, 746, 695 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 7.29-7.20 (m, 5H, ArH), 7.29-7.20 (m, 5H, ArH*), 5.44-5.38 (m, 2H, H-3, H-3*), 5.06 (t, *J* = 9.8 Hz, 1H, H-4*), 5.0 (t, *J* = 10.0 Hz, 1H, H-4), 4.88 (d, *J* = 6.3 Hz, 1H, H-1), 4.85 (d, *J* = 3.6 Hz, 1H, H-1*), 4.82 (t, *J* = 3.3 Hz, 1H, H-2), 4.80 (t, *J* = 3.4 Hz, 1H, H-2*), 4.08-4.01 (m, 8H, H-6a, H-6a*, H-6b, H-6b*, CH_2, CH_2*), 3.94-3.88 (m, 2H, H-5, H-5*), 3.65 (d, *J* = 11.2 Hz, 3H, CH_3), 3.58 (d, *J* = 9.3 Hz, 3H, CH_3*), 3.34 (s, 3H, CH_3), 3.31 (s, 3H, CH_3*), 3.22 (br, 1H, *N*H), 2.96 (br,

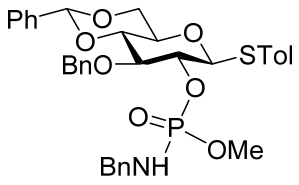
1H, *N*H*), 2.02 (s, 3H, CH_3), 1.99 (s, 3H, CH_3*), 1.98 (s, 3H, CH_3), 1.96 (s, 3H, CH_3*), 1.95 (s, 3H, CH_3), 1.91 (s, 3H, CH_3*);^{13}C NMR (100 MHz, $CDCl_3$): 170.2 (C x 2, C*), 170.1 (C), 169.9 (C*), 169.6 (C*), 140.0 (d, J = 3.8 Hz, C), 139.6 (d, J = 6.1 Hz, C*), 128.7 (CH, CH*), 128.6 (CH, CH*), 127.5 (CH, CH*), 127.4 (CH, CH*), 127.3 (CH, CH*), 96.82 (CH), 96.77 (CH*), 70.85 (CH, CH*), 70.32 (CH*), 70.26 (CH), 68.41 (CH), 67.91 (CH*), 64.30, (d, J = 4.0 Hz, CH_2), 64.07 (d, J = 4.0 Hz, CH_2*), 55.54 (CH_3, CH_3*), 53.44 (CH_3), 52.15 (CH_3*), 45.33 (CH_2), 45.25 (CH_2*), 20.80 (CH_3, CH_3*), 20.76 (CH_3, CH_3*), 20.68 (CH_3, CH_3*); ^{31}P NMR (161.98 MHz, $CDCl_3$): δ 16.59, 10.77, 10.40; HRMS (ESI) calcd for $C_{21}H_{30}NO_{11}PNa$ $[M+Na]^+$ 526.1454, found 526.1461.



11

1,2:3,4-di-*O*-isopropylidene-6-*O*-(methyl *N*-benzylphosphoramidyl)-D-galactopyranose (11).

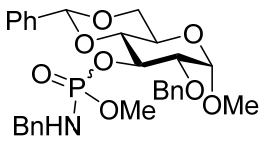
Colorless oil, $[\alpha]^{29}_D$ -28.0 (*c* 0.5, $CHCl_3$); IR ($CHCl_3$) ν 2987, 2934, 1454, 1253, 1067, 1003, 905, 889, 859, 759, 735 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29-7.21 (m, 5H, ArH), 7.29-7.21 (m, 5H, ArH*), 5.48 (d, J = 4.8 Hz, 1H, H-1), 5.47 (d, J = 4.8 Hz, 1H, H-1*), 4.57 (dd, J = 3.8, 1.8 Hz, 1H, H-3), 4.55 (dd, J = 3.8, 1.8 Hz, 1H, H-3*), 4.27 (dd, J = 5.0, 2.4 Hz, 2H, H-2, H-2*), 4.20-4.02 (m, 12 H, H-4, H-4*, H-5, H-5*, H-6a,b, H-6a,b*, *N*CH_2, *N*CH_2*), 3.66 (d, J = 11.2 Hz, 3H, CH_3), 3.66 (d, J = 11.2 Hz, 3H, CH_3*), 3.15 (br, 2H, *N*H, *N*H*), 1.49 (s, 3H, $C(CH_3)_2$), 1.47 (s, 3H, $C(CH_3)_2$), 1.38 (s, 3H, $C(CH_3)_2$), 1.36 (s,3H, $C(CH_3)_2$), 1.27 (s, 6H, 2 x $C(CH_3)_2$), 1.26 (s, 3H, $C(CH_3)_2$), 1.26 (s, 3H, $C(CH_3)_2$); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.6 (C), 139.5 (C*), 128.4 (CH x 2, CH* x 2), 127.3 (CH x 2, CH* x 2), 127.2 (CH), 127.2 (CH*), 109.5 (C), 109.4 (C), 108.7 (C* x 2), 96.2 (CH, CH*), 70.7 (CH), 70.6 (CH*), 70.5 (CH, CH*), 70.4 (CH), 70.3 (CH*), 67.3 (CH), 67.2 (CH*), 66.7 (CH), 66.6 (CH*), 65.3 (CH), 65.3 (CH*), 64.8 (CH), 64.8 (CH), 53.2 (CH_3), 53.1 (CH_3*), 45.2 (CH_2, CH_2*), 25.8 (CH_3 x 2, CH_3*x 2), 24.8 (CH_3), 24.8 (CH_3*), 24.3 (CH_3), 24.3 (CH_3*); ^{31}P NMR (161.97 MHz, $CDCl_3$): δ 10.8, 10.5; HRMS (ESI) calcd for $C_{20}H_{30}NNaO_8P$ $[M+Na]^+$ 466.1607, found 466.1608. Data in agreement with known literature.[1]



13

***p*-Toluenyl 3-*O*-benzyl-4,6-*O*-benzylidene-2-*O*-(methyl *N*-benzylphosphoramidyl)-thio-β-D-glucopyranoside (13).**

$[\alpha]^{24}_{D}$ -34.3 (*c* 0.25, $CHCl_3$); IR ($CHCl_3$) ν 2923, 1733, 1455, 1166, 1065, 1025, 746, 695 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): 7.44 - 7.42 (m, 4H, ArH), 7.37-7.18 (m, 10 H, ArH), 7.15-7.10 (m, 4H, ArH), 5.54 (s, 1H, CH_2Ph), 4.96 (d, 1H, *J* = 10.6 Hz, CH_2Ph), 4.72 (d, *J* = 9.6 Hz, 1H, H-1), 4.68 (d, *J* = 10.6 Hz, 1H, CH_2Ph), 4.37 (dd, *J* = 5.0, 4.8 Hz, 1H, H-6eq), 4.24 (dd, *J* = 10.2, 10.0 Hz, 1H, H-2), 3.99 (m, 2H, CH_2), 3.85(t, *J* = 9.0 Hz, 1H, H-3), 3.76 (t, *J* = 10.2 Hz, 1H, H-6ax), 3.68 (d, *J* = 11.4 Hz, 3H, CH_3), 3.67 (t, *J* = 9.6 Hz, 1H, H-4), 3.49 (ddd, *J* = 5.2, 5.1, 5.0 Hz, 1H, H-5), 3.03 (m, 1H, *N*H), 2.32 (s, 3H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$): 140.0 (d, *J* = 6.4 Hz, C), 138.7 (C), 138.0 (C), 137.2 (C), 133.9 (CH), 129.9 (CH), 129.3(CH), 128.6 (CH), 128.5 (CH), 128.4 (CH), 128.3 (CH), 128.0 (CH), 127.5 (CH), 127.3 (CH), 126.2 (CH), 101.4 (CH), 87.7 (d, *J* = 6.0 Hz, CH), 81.78 (CH), 81.57 (CH), 76.33 (d, *J* = 6.2 Hz, CH), 74.94 (CH_2), 70.40 (CH), 68.77 (CH), 53.86 (d, *J* = 5.4 Hz, CH), 45.5 (CH_2), 21.3 (CH_3); ^{31}P NMR (161.97 MHz, $CDCl_3$): δ 9.20; HRMS (ESI) calcd for $C_{35}H_{38}NO_7NaPS$ $[M+Na]^+$ 670.2004, found 670.2004.

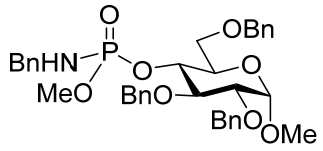


15

Methyl 2-*O*-benzyl-4,6-*O*-benzylidene-3-*O*-(methyl *N*-benzylphosphoramidyl)-α-D-glucopyranoside (15).

Colorless oil, $[\alpha]^{26}_{D}$ 22.2 (*c* 0.5, $CHCl_3$); IR ($CHCl_3$) ν 2927, 2868, 1496, 1453, 1375, 1254, 1088, 1027, 989, 865, 747, 698, 678 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.43-4.41 (m, 1H, ArH), 7.43-4.41 (m, 1H, ArH*), 7.38-7.31 (m, 3H, ArH), 7.38-7.31 (m, 3H, ArH*), 7.30-7.16 (m, 9H, ArH), 7.30-7.16 (m, 9H, ArH*), 7.11-7.05 (m, 2H, ArH), 7.11-7.05 (m, 2H, ArH*), 5.45 (s, 1H, PhCH),

5.43 (s, 1H, PhCH*), 4.79 (d, J = 11.8 Hz, 1H, CH_2Ph), 4.72 (d, J = 12.0 Hz, 1H, CH_2Ph*), 4.74-4.65 (m, 4H, H-3, H-3*,CH_2Ph, CH_2Ph*), 4.62 (d, J = 5.5 Hz, 1H, H-1), 4.57 (d, J = 5.5 Hz, 1H, H-1*), 4.25 (t, J = 4.6 Hz, 1H, H-6eq), 4.22 (t, J = 4.6 Hz, H-6eq*), 4.01-3.88 (m, 4H, CH_2, CH_2*), 3.84-3.77 (m, 2H, H-5, H-5*), 3.674 (t, J = 10.0 Hz, 1H, H-6ax), 3.672 (t, J = 10.0 Hz, 1H, H-6ax*), 3.62 (d, J = 11.2 Hz, 3H, CH_3), 3.62-3.57 (m, 4H, H-2, H-2*, H-4, H-4*), 3.51 (d, J = 11.4 Hz, 3H, CH_3*), 3.35 (s, 6H, CH_3, CH_3*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.8 (d, J = 6.0 Hz, C, C*), 137.6 (C, C*), 137.4 (C, C*), 136.8 (C, C*), 136.6 (C, C*), 129.2 (CH, CH*), 128.5 (CH, CH*), 128.3 (CH, CH*), 128.17 (CH, CH*), 128.10 (CH, CH*), 127.3 (CH, CH*), 127.0 (CH, CH*), 126.2 (CH, CH*), 126.0 (CH, CH*), 102.04 (CH), 101.9 (CH*), 98.8 (CH), 98.7 (CH*), 80.42 (CH), 80.34 (CH*), 78.6 (CH, CH*), 75.72 (d, J = 7.0 Hz, CH), 75.28 (d, J = 6.2 Hz, CH*), 73.60 (CH_2*), 73.23 (CH_2), 68.91 (CH_2, CH_2*), 62.18 (CH), 62.09 (CH*), 55.40 (CH_3), 53.62 (CH_3*), 53.55 (CH_3), 53.50 (CH_3*), 45.0 (CH_2, CH_2*); ^{31}P NMR (161.97 MHz, $CDCl_3$): δ 9.8, 9.0; HRMS (ESI) calcd for $C_{29}H_{33}NO_8P$ $[M-H]^-$ 554.1948, found 554.1944.

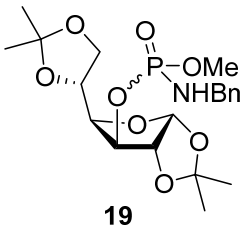


17

Methyl 2,3,6-tri-*O*-benzyl-4-*O*-(methyl *N*-benzylphosphoramidyl)-α-D-glucopyranoside (17).

Colorless oil, $[\alpha]^{27}_D$ 45.5 (*c* 0.5, $CHCl_3$); IR ($CHCl_3$) ν 2922, 1722, 1453, 1248, 1026, 738, 697 cm^{-1};^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36-7.19 (m, 18 H, ArH), 7.00 (d, J = 3.2 Hz, 1H, ArH), 6.98 (d, J = 1.8 Hz, 1H, ArH), 5.01 (d, J = 10.6 Hz, 1H, CH_2Ph), 4.72 (d, J = 12.0 Hz, 1H, CH_2Ph), 4.66 (d, J = 11.8 Hz, 1H, CH_2Ph), 4.62-4.59 (m, 3H, 1H, CH_2Ph), 4.53 (d, J = 11.8 Hz, 1H, CH_2Ph), 4.25 (q, J = 9.5 Hz, 1H, H-5), 3.94-3.87 (m, 3H, H-3, CH_2Ph), 3.84-3.74 (m, 3H, H-4, H-6a, H-6b), 3.55 (m, 1H, H-2), 3.49 (d, J = 11.6 Hz, 3H, CH_3), 3.37 (s, 3H, OCH_3), 3.22-3.16 (m, 1H, *N*H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.0 (d, J = 5.5 Hz, C), 138.5 (C), 138.2 (C), 138.0 (C), 128.7 (CH), 128.5 (CH), 128.4 (CH), 128.3 (CH), 128.2 (CH), 128.14 (CH), 128.13 (CH), 128.0 (CH), 127.6 (CH), 127.5 (CH), 127.2 (CH), 97.86 (CH), 80.63 (CH), 80.13 (CH), 77.40 (CH), 76.0 (CH_2), 73.74 (CH), 73.69 (CH), 73.60 (CH_2), 73.37 (CH_2), 69.84 (d, J = 4.0 Hz, CH), 68.66 (CH_2),

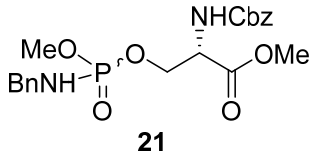
55.54 (CH), 45.48 (CH_2) ^{31}P NMR (161.97 MHz, $CDCl_3$): δ 10.4; HRMS (ESI) calcd for $C_{36}H_{42}NO_8NaP$ $[M+Na]^+$ 670.2546, found 670.2552.



19

1,2:5,6-di-*O*-isopropylidene-3-*O*-(methyl *N*-*O*-benzylphosphoramidyl)-D-glucopyranose (19).

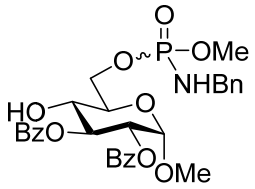
$[\alpha]^{32}_D$ -27.6 (*c* 0.5, $CHCl_3$); IR ($CHCl_3$) *v* 3227, 2987, 2936, 1454, 1374, 1250, 1218, 1195, 1072, 1027, 960, 843, 735 cm^{-1}; 1H NMR (400 M Hz, $CDCl_3$): δ 7.29 -7.21 (m, 10H, ArH), 5.85 (d, 1H, *J* = 3.6 Hz, H-1), 5.76 (d, *J* = 3.6 Hz, 1H, H-1*), 4.81 (d, *J* = 3.6 Hz, 1H, H-2), 4.74 (m, 2H, H-3, H-3*), 4.66 (d, *J* = 3.6 Hz, 1H, H-2*), 4.20-3.99 (m, 10H, H-4, H-4*, H-5, H-5*, H-6a, H-6a*, $PhCH_2$, $PhCH_2$*), 3.95-3.91 (m, 2H, H-6b, H-6b*), 3.69 (d, *J* = 11.2 Hz, 3H, OCH_3), 3.65 (d, *J* = 11.2 Hz, 3H, OCH_3), 3.60 (m, 2H), 1.45 (s, 3H, $C(CH_3)_2$), 1.43 (s, 3H, $C(CH_3)_2$), 1.30 (s, 3H, $C(CH_3)_2$), 1.26 (s, 3H, $C(CH_3)_2$), 1.22 (s, 3H, $C(CH_3)_2$, 1.20 (s, 3H, $C(CH_3)_2$), 1.17 (s, 3H, $C(CH_3)_2$), 1.12 (s, 3H, $C(CH_3)_2$); ^{13}C NMR (100 MHz, $CDCl_3$): δ 139.5 (d, *J* = 4.9 Hz, C, C*), 128.7 (CH x 2, CH* x 2), 127.76 (CH x 2, CH* x 2), 127.4 (CH, CH*), 112.3 (d, *J* = 11.4, C, C*), 109.4 (d, *J* = 10.0 C, C*), 105.3 (CH), 105.2 (CH*), 83.9 (CH), 83.7 (CH*), 80.8 (d, *J* = 8.3 Hz, CH, CH*), 79.53 (d, *J* = 6.0 Hz, CH), 78.5 (d, *J* = 4.1 Hz, CH), 72.5 (CH), 72.3 (CH*), 67.6 (CH_2), 67.5 (CH_2*), 53.5 (d, *J* = 4.5 Hz,CH_3, CH_3*), 45.4 (CH_2), 45.3 (CH_2*), 26.9 (CH_3), 26.8 (CH_3, CH_3*), 26.7 (CH_3), 26.3 (CH_3, CH_3*), 25.3 (CH_3, CH_3*); ^{31}P NMR (161.98 MHz, $CDCl_3$): δ 10.5, 9.2; HRMS (ESI) calcd for $C_{20}H_{31}NO_8P$ $[M+H]^+$ 444.1787, found 444.1786. Data in agreement with known literature.[1]



21

Methyl *O*-((benzylamino)(methoxy)phosphoryl)-*N*-((benzyloxy)carbonyl)-L-serinate (21).

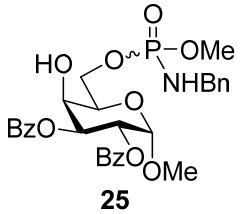
$[\alpha]^{32}_{D}$ 17.2 (*c* 0.5, $CHCl_3$); IR ($CHCl_3$) *v* 3247, 2962, 2924, 1721, 1514, 1455, 1438, 1344, 1317, 1218, 1050, 1017, 973, 917, 874, 739, 659 cm^{-1}; ^1H NMR (400 M Hz, $CDCl_3$): δ 7.37-7.26 (m, 10H, ArH), 6.10 (d, *J* = 8.0 Hz, 1H, *N*HCH), 5.11 (d, *J* = 5.0 Hz, 2H, CH_2Cbz), 4.55 (d, *J* = 7.2 Hz, 1H, $CHCH_2$), 4.44-4.26 (m, 2H, $CHCH_2$), 4.03 (d, *J* = 11.0 Hz, 2H, CH_2Ph), 3.64 (d, *J* = 7.0 Hz, 3H, OCH_3), 3.64 (t, *J* = 11.2 Hz, 3H, OCH_3), 3.21 (br, 1H, *N*H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.8 (C), 156.1 (C), 139.4 (C*), 139.3 (C), 136.4 (C*) 128.8 (CH, CH*), 128.6 (CH, CH*), 128.3 (CH, CH*), 128.2 (CH, CH*), 127.7 (CH, CH*), 127.4 (CH, CH*), 67.28 (CH_2), 66.30 (CH_2), 66.24 (CH_2*), 54.81 (CH), 54.75 (CH*), 53.60 (CH_3), 53.54 (CH_3*), 53.45 (CH_3), 52.90 (CH_3*), 45.46 (CH_2, CH_2*); ^{31}P NMR (161.98 MHz,$CDCl_3$): δ 10.8, 10.6



23

Methyl 2,3-di-*O*-benzoyl-6-*O*-(methyl *N*-benzylphosphoramidyl)-α-D-glucopyranoside (23).

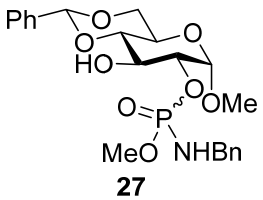
$[\alpha]^{32}_{D}$ 115.9 (*c* 0.5, $CHCl_3$); IR ($CHCl_3$) *v* 3331, 2381, 2341, 1724, 1801, 1452, 1278, 1098, 1069, 1026, 917, 864, 754, 710, 619 cm^{-1}; ^1H NMR (400 M Hz, $CDCl_3$): δ 7.96-7.93 (m, 4H, ArH), 7.96-7.93 (m, 4H, ArH*), 7.48-7.42 (m, 2H, ArH), 7.48-7.42 (m, 2H, ArH*), 7.34-7.24 (m, 9H, ArH), 7.34-7.24 (m, 9H, ArH*) 5.83-5.77 (m, 2H, H-3, H-3*), 5.16-5.09 (m, 4H, H-1, H-1*, H-2, H-2*), 4.44-4.35 (2H, H-6a, H-6b), 4.23 (dddd, *J* = 7.5, 7.4, 2.2, 2.1 Hz, 1H, H-6b*), 4.13-4.06 (m, 5H, CH_2Ph, CH_2Ph*, H-6a*), 3.94-3.81 (m, 4H, H-4, H-4*, H-5, H-5*), 3.7 (d, *J* = 11.2 Hz, 3H, CH_3), 3.64 (d, *J* = 11.2 Hz, 3H, CH_3*), 3.36 (s, 3H, OMe), 3.35 (s, 3H, OMe*), 3.15 (br, 2H, OH, OH*), ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.7 (C, C*), 166.1 (C, C*), 139.6 (C) 139.5 (C*), 139.47 (C), 139.42 (C*), 133.4 (CH, CH*), 133.1 (CH, CH*), 130.0 (CH, CH*), 129.9 (CH, CH*), 129.3 (CH, CH*), 128.8 (CH, CH*), 128.5 (CH, CH*), 128.4 (CH, CH*), 127.6 (CH, CH*), 127.54 (CH, CH*), 127.51 (CH, CH*), 97.44 (CH, CH*), 97.38 (CH, CH*), 73.1 (CH, CH*), 72.9 (CH, CH*), 72.1 (CH, CH*), 70.92 (CH, CH*), 70.88 (CH, CH*), 68.6 (CH, CH*), 68.4 (CH, CH*), 65.4 (d, *J* = 5.0 Hz, CH_2, CH_2*), 64.8 (d, *J* = 4.0 Hz, CH_2, CH_2*), 55.6 (CH_3), 53.6 (CH_3*), 53.6 (d, *J* = 5.6 Hz, CH_3), 53.5 (d, *J* = 5.6 Hz, CH_3*), 45.5 (CH_2), 45.4 (CH_2*); ^{31}P NMR (161.98 MHz,$CDCl_3$) δ 12.1, 12.0; HRMS (ESI) calcd for $C_{29}H_{32}NO_{10}NaP$ $[M+Na]^+$ 608.1662, found 608.1651.



25

Methyl 2,3-di-*O*-benzoyl-6-*O*-(methyl *N*-benzylphosphoramidyl)-α-D-galactopyranoside (25).

Colorless oil, $[\alpha]^{26}_D$ 141.5 (*c* 0.5, $CHCl_3$); IR ($CHCl_3$) ν 3342, 1719, 1451, 1315, 1279, 1051, 1026, 912, 710 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.99-7.93 (m,4H, ArH), 7.99-7.93 (m,4H, ArH*), 7.46 (td, *J* = 7.2, 1.2 Hz, 2H, ArH), 7.46 (td, *J* = 7.2, 1.2 Hz, 2H, ArH*), 7.34-7.22 (m, 9H, ArH), 7.34-7.22 (m, 9H, ArH*), 5.69-5.60 (m, 2H, H-2, H-3), 5.69-5.60 (m, 2H, H-2*, H-3*), 5.13 (s, 1H, H-1), 5.12 (s, 1H, H-1*), 4.37 (t, *J* = 3.6 Hz, 1H, H-4), 4.33 (t, *J* = 4.4 Hz, 1H, H-4*), 4.31-4.23 (m, 1H, H-6a), 4.31-4.23 (m, 1H, H-6a*), 4.13 (t, *J* = 7.2 Hz, 1H, H-6b), 4.13 (t, *J* = 7.2 Hz, 1H, H-6b*), 4.06-3.99 (m, 3H, CH_2, H-5), 4.06-3.99 (m, 3H, CH_2*, H-5*), 3.84 (d, *J* = 4.7 Hz, 1H, OH), 3.68 (d, *J* = 4.8 Hz, 1H, OH*), 3.65 (d, *J* = 11.3 Hz, 3H, CH_3), 3.65 (d, *J* = 11.3 Hz, 3H, CH_3*), 3.37 (s, 3H, OCH_3), 3.37 (s, 3H, OCH_3*), 3.27-3.16 (m, 1H, *N*H), 3.27-3.16 (m, 1H, *N*H*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.0 (C), 165.9 (C), 139.3 (C), 139.2 (C, C*), 139.1 (C*), 133.1 (CH, CH*), 129.8 (CH, CH*), 129.6 (CH, CH*), 129.5 (CH, CH*), 128.6 (CH CH*), 128.3 (CH, CH*), 127.5 (CH, CH*), 127.3 (CH, CH*), 97.64 (CH, CH*), 70.5 (CH, CH*), 70.4 (CH, CH*), 69.0 (CH, CH*), 68.54 (CH, CH*), 68.47 (CH, CH*), 68.41 (CH, CH*), 68.3 (CH, CH*), 67.1 (CH, CH*), 67.0 (CH, CH*), 64.2 (d, *J* = 4.8 Hz, CH_2, CH_2*), 63.9 (d, *J* = 3.8 Hz, CH_2, CH_2*), 55.5 (CH_3, CH_3*), 53.4 (CH_3), 53.3 (CH_3*), 45.3 (CH_2, CH_2*); ^{31}P NMR (161.97 MHz, $CDCl_3$): δ 11.7, 11.4; HRMS (ESI) calcd for $C_{29}H_{32}NNaO_{10}P$ $[M+Na]^+$ 608.1662, found 608.1664.

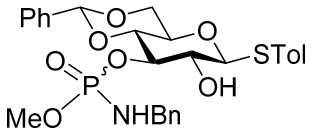


27

Methyl 2-*O*-(methyl *N*-benzylphosphoramidyl)-4,6-*O*-benzylidene-α-D-glucopyranoside (27).

Colorless oil, $[\alpha]^{26}_D$ 55.7 (*c* 0.5, $CHCl_3$); IR ($CHCl_3$) ν 3267, 2934, 1453, 1375, 1215, 1147, 1067, 1042, 991, 933, 915, 889, 747, 698, 674 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.48-7.45 (m, 2H,

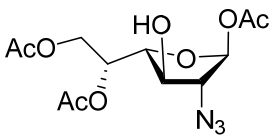
ArH), 7.36-7.21 (m, 8H, ArH), 5.50 (s, 1H, PhCH), 4.91 (d, J = 3.6 Hz, 1H, H-1), 4.28-4.21 (m, 2H, H-2, H-6eq), 4.18-4.04 (m, 3H, H-3, CH_2), 3.83 (td, J = 10.8, 4.6 Hz, 1H, H-5), 3.71 (t, J = 10.2 Hz, 1H, H-6ax), 3.68 (d, J = 11.4 Hz, 3H, CH_3), 3.64 (br, 1H, *N*H), 3.48 (t, J = 9.2 Hz, 1H, H-4), 3.41 (s, 3H, OCH_3), 3.35-3.29 (m, 1H, OH); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.4 (C), 137.0 (C x 2), 129.1 (CH), 128.5 (CH x 2), 128.2 (CH x 2), 127.4 (CH x 3), 126.3 (CH x 2), 101.9 (PhCH), 98.9 (CH), 81.2 (CH), 69.5 (CH), 69.4 (CH), 68.9 (CH_2), 62.0 (CH), 55.5 (OCH_3), 53.4 (CH_3), 45.4 (CH_2); ^{31}P NMR (161.97 MHz, $CDCl_3$): δ 11.5, 11.0; HRMS (ESI) calcd for $C_{22}H_{28}NNaO_8P$ $[M+Na]^+$ 488.1450, found 488.1444.



29

p-toluenyl 3-*O*-(methyl *N*-benzylphosphoramidyl)-4,6-*O*-benzylidene-thio-β-D-glucopyranoside (29).

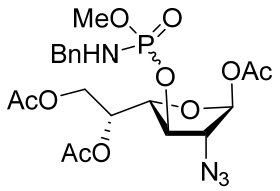
$[\alpha]^{32}_D$ -38.6 (*c* 0.5, $CHCl_3$); IR ($CHCl_3$) *v* 3314, 2923, 2889, 2360, 1493, 1454, 1383, 1314, 1218, 1086, 1016, 891, 868, 809. 760, 699 cm^{-1}; ^{1}H NMR (400 MHz, CD_3OD): δ 7.46-7.42 (m, 4H, ArH), 7.33-7.28 (m, 3H, ArH), 7.22-7.10 (m, 7H, ArH), 5.53 (s, 1H, PhCH), 4.67 (d, J = 9.8 Hz, 1H, H-1), 4.44 (q, J = 8.7, 8.7 Hz, 1H, H-3), 4.28 (dd, J = 10.3, 4.8 Hz, 1H, H-6eq), 3.90 (d, J = 11.0 Hz, 2H, CH_2), 3.73 (t, J = 11.4 Hz, 1H, H-6ax), 3.62 (d, J = 11.4 Hz, 3H, OMe), 3.60 (t, J = 9.1 Hz, 1H, H-4), 3.54-3.51 (m, 1H, H-5), 3.47 (t, J = 9.0 Hz, 1H, H2), 2.33 (s, 3H, CH_3); ^{13}C NMR (100 MHz, CD_3OD): δ 141.3 (C), 141.3 (C), 139.5 (C), 138.7 (C), 134.5 (CH), 130.6 (CH), 130.1 (CH), 129.8 (CH), 129.3 (CH), 129.1 (CH), 128.3 (CH), 127.9 (CH), 127.5 (CH), 102.9 (CH), 89.89 (CH), 81.47 (d, J = 6.0 Hz, CH), 80.41 (d, J = 2.0 Hz, CH), 73.19 (CH), 71.29 (CH), 69.46 (CH_2), 54.23 (d, J = 5.9 Hz, CH_3), 45.75 (CH_2), 21.16 (CH_3); ^{31}P NMR (161.98 MHz, CD_3OD): δ 11.4, 10.7; HRMS (ESI) calcd for $C_{28}H_{32}NO_7NaSP$ $[M+Na]^+$ 580.1535, found 580.1542.



30

1,5,6-Tri-*O*-acetyl-2-azido-2-deoxy-D-galactofuranose (30).

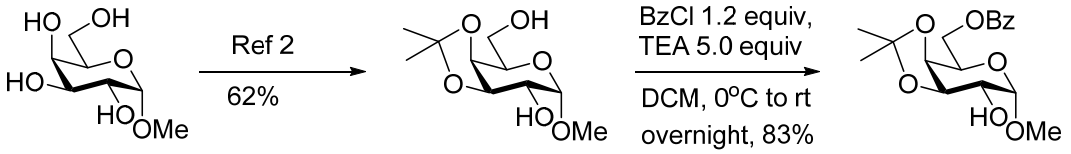
Colorless oil, $[\alpha]^{20}_D$ -73.9 (*c* 0.5, $CHCl_3$); IR ($CHCl_3$) ν 2105, 1731, 1370, 1214, 1003, 928 cm^{-1} ; ^1H NMR (400 MHz, $CDCl_3$); δ 6.0 (d, *J* = 2.3 Hz, 1H, H-1), 5.26-5.24 (m, 1H, H-5), 4.31-4.18 (m, 2H, H6a, H6b), 4.14, (dd, *J* = 7.0, 3.3 Hz, 1H, H-4), 4.0 (dd, *J* = 5.0, 2.3 Hz, 1H, H-2), 3.94-3.89 (m, 1H, H-3), 3.39 (d, *J* = 5.0 Hz, 1H, OH), 2.12 (s, 3H, CH_3), 2.09 (s, 3H, CH_3), 2.02 (s, 3H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.3 (CO), 170.8 (CO), 169.6 (CO), 99.5 (CH), 82.57 (CH), 75.62 (CH), 71.34 (CH), 69.24 (CH), 62.81 (CH_2), 21.0 (CH_3), 20.81 (CH_3), 20.67 (CH_3); (HR-ESI) calcd for $C_{12}H_{17}N_3O_8Na$ $[M+Na]^+$ 354.0913, found 354.0914.



31

1,5,6-tri-*O*-acetyl-2-azido-2-deoxy-3-*O*-(methyl-*N*-benzylphosphoramidyl)-β-D-galactofuranose (31).

Colorless oil, $[\alpha]^{20}_D$ -74.6 (*c* 0.5, $CHCl_3$); IR ($CHCl_3$) ν 2116, 1749, 1454, 1220, 1033, 969, 772 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$); δ 7.34-7.24 (m, 10H, ArH), 6.08 (s, 2H, H-1, H-1*), 5.35-5.27 (m, 2H, H-5, H-5*), 4.48 (ddd, *J* = 12.9, 7.5, 2.7 Hz, 2H, H-3, H-3*), 4.33 (ddd, *J* = 8.8, 5.5, 3.5 Hz, 2H, H-4, H-4*), 4.30-4.23 (m, 3H, H-2, H-2*, H6a), 4.19-4.13 (m, 3H, H6a*, H6b, H6b*), 4.08 (dd, *J* = 10.8, 7.0 Hz, 4H, CH_2, CH_2*), 3.7 (d, *J* = 11.2 Hz, 3H, CH_3), 3.69 (d, *J* = 11.1 Hz, 3H, CH_3*), 3.33-3.23 (m, 2H, *N*H, *N*H*), 2.09 (s, 3H, CH_3), 2.07 (s, 3H, CH_3*), 2.06 (s, 3H, CH_3), 2.05 (s, 3H, CH_3*), 2.01 (s, 6H, CH_3, CH_3*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.5 (C, C*), 170.4 (C), 170.2 (C*), 169.3 (C, C*), 139.0 (d, *J* = 5.1 Hz, C), 128.6 (CH, CH*, CH, CH*), 127.6 (CH, CH*), 127.4 (CH, CH*), 127.3 (CH, CH*), 99.92 (d, *J* = 5.6 Hz, CH), 83.0 (d, *J* = 6.5 Hz, CH*), 82.8 (d, *J* = 8.2 Hz, CH), 78.71 (CH, CH*), 70.61 (d, *J* = 1.7 Hz, CH), 70.35 (d, *J* = 3.8 Hz, CH*), 68.71 (d, *J* = 8.3 Hz, CH_2), 62.58 (d, *J* = 6.5 Hz, CH_2*), 53.43 (d, *J* = 3.5 Hz, CH_3, CH_3*), 45.36 (CH_2, CH_2*), 20.92 (CH_3, CH_3*), 20.69 (CH_3, CH_3*), 20.65 (CH_3, CH_3*); ^{31}P NMR (161.97 MHz, $CDCl_3$): δ 9.7, 9.5; (HR-ESI)calcd for $C_{20}H_{27}N_4O_{10}PNa$ $[M+Na]^+$ 537.1363, found 537.1364.

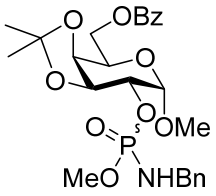


32

Methyl 3,4-*O*-Isopropylidene-α-D-galactopyranoside prepared by known protocol. Data is in agreement with the literature[2].

Methyl 3,4-*O*-Isopropylidene-6-*O*-benzoyl-α-D-galactopyranoside (32).

Colorless oil, $[\alpha]^{27}_{D}$ 90.3 (*c* 0.5, $CHCl_3$); IR ($CHCl_3$) ν 2986, 2838, 2360, 1717, 1315, 1380, 1023, 988, 709 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$); δ 8.0 (d, *J* = 7.8 Hz, 2H, ArH), 7.54 (t, *J* = 7.5 Hz, 1H, ArH), 7.42 (t, *J* = 7.58 Hz, 2H, ArH), 4.7 (d, *J* = 3.9 Hz, 1H, H-1), 4.58-4.49 (m, 2H, CH_2), 4.32-4.25 (m, 3H, H-3, H-4, H-5), 3.86 (dd, *J* = 9.2, 5.0 Hz, 1H, H-2), 3.42 (s, 3H, OCH_3), 2.46 (d, *J* = 5.2 Hz, 1H, OH), 1.49 (s, 3H, CH_3), 1.33 (s, 3H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.5 (CO), 133.2 (CH), 130.2 (C), 129.8 (CH), 128.6 (CH), 110.1 (C), 98.2 (CH), 76.0 (CH), 73.2 (CH), 69.0 (CH), 66.8 (CH), 64.3 (CH_2), 55.5 (CH_3), 27.7 (CH_3), 26.0 (CH_3); (HR-ESI)calcd for $C_{17}H_{22}O_7Na$ $[M+Na]^+$ 361.1263, Found 361.1272.

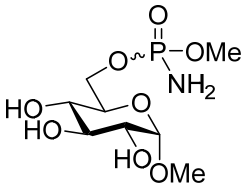


33

Methyl 2-*O*-(methyl *N*-benzylphosphoramidyl)-3,4-*O*-isopropylidene-α-D-galactopyranoside (33).

Colorless oil, $[\alpha]^{27}_{D}$ 92.6 (*c* 0.5, $CHCl_3$); IR ($CHCl_3$) ν 2986, 2936, 2359, 1718, 1315, 1270, 1020, 932, 821, 710 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$); δ 8.03 (d, *J* = 7.5 Hz, 4H, ArH), 7.55 (t, *J* = 7.6 Hz, 2H, ArH), 7.42 (t, *J* = 7.8 Hz, 4H, ArH), 7.32-7.24 (m, 10 H, ArH), 4.98 (d, *J* = 3.2 Hz, 1H, H-1), 4.91 (d, *J* = 3.4 Hz, 1H, H-1*), 4.62-4.5 (m, 4H, CH_2, CH_2*), 4.43 (ddd, *J* = 16.0, 8.0, 3.5 Hz, 1H, H-2*), 4.39-4.26 (m, 7H, H-2, H-3, H-3*, H-4, H-4*, H-5, H-5*), 4.18-4.08 (m, 4H, CH_2, CH_2*), 3.7 (d, *J* = 11.0 Hz, 3H, CH_3), 3.67 (d, *J* = 11.7 Hz, 3H, CH_3*), 3.40 (s, 3H, CH_3), 3.34 (s, 3H, CH_3*), 3.13-3.05 (m, 2H, *N*H, *N*H*), 1.52 (s, 3H, CH_3), 1.46 (s, 3H, CH_3*), 1.33 (s, 3H, CH_3),

1.32 (s, 3H, CH_3*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.52 (CO), 139.8 (C), 139.7 (C, C*), 139.6 (C*), 133.3 (CH, CH*, CH), 130.2 (CH*), 129.8 (CH, CH*, CH, CH*), 128.7 (CH, CH*, CH, CH*), 128.6 (CH, CH*, CH, CH*), 127.7 (CH, CH*), 127.5 (CH, CH*), 110.2 (C), 110.17 (C*), 98.54 (CH), 98.28 (CH*), 75.0 (d, *J* = 6.0 Hz, CH), 74.89 (d, *J* = 6.4 Hz, CH*), 74.79 (CH), 74.74 (CH*), 73.93 (CH, CH*), 66.03 (CH), 65.76 (CH*), 64.15 (CH_2, CH_2*), 55.86 (CH_3), 55.76 (CH_3*), 53.62 (d, *J* = 5.5 Hz, CH_3), 53.45 (d, *J* = 5.25 Hz, CH_3), 45.67 (CH_2), 45.58 (CH_2*), 28.18 (CH_3), 28.06 (CH_3*), 26.56 (CH_3, CH_3*); ^{31}P NMR (161.97 MHz, $CDCl_3$): δ 10.7, 9.7; (HR-ESI)calcd for $C_{25}H_{32}NO_9NaP$ $[M+Na]^+$ 544.1712, Found 544.1708.

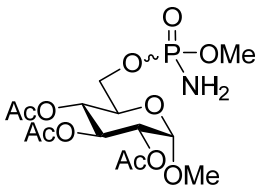


34

Methyl 6-*O*-(methyl phosphoramidyl)-α-D-glucopyranoside (34).

To a solution of compound **6** (25 mg, 0.041 mmol) in EtOH (0.6 mL) was added $Pd(OH)_2$ (120 mg) and stirred reaction mixture overnight under H_2 atmosphere.[3] Reaction was monitored by TLC. The reaction was diluted with EtOH and filtered through a celite bed. The bed was washed with EtOH (2mL). The EtOH was concentrated under a reduced pressure and dried under high vacuum to afford desired compound **36** quantitatively (11 mg).

Colorless oil, ^{1}H NMR (500 MHz, CD_3OD); δ 4.67 (d, *J* = 3.4 Hz, 2H, H-1, H-1*), 4.23 (ddd, *J* = 11.0, 6.2, 1.7 Hz, 2H, H-6a, H6a*), 4.16-4.11 (m, 2H, H-6b, H-6b*), 3.71 (d, *J* = 11.4 Hz, 3H, CH_3), 3.71 (d, *J* = 11.3 Hz, CH_3*), 3.69-3.66 (m, 2H, H-5, H-5*), 3.61 (t, *J* = 9.2 Hz, 2H, H-3, H-3*), 3.40 (m, 8H, H-2, H-2*, CH_3, CH_3*), 3.34-3.32 (m, 2H, H-4, H-4*); ^{13}C NMR (125 MHz, CD_3OD) δ 101.3 (CH, CH*), 70.98 (CH, CH*), 73.45 (CH, CH*), 72.03 (CH, CH*), 71.33 (CH, CH*), 72.05 (d, *J* = 6.3 Hz, CH), 72.01 (d, *J* = 3.8 Hz, CH*), 66.85 (d, *J* = 5.4 Hz, CH_2), 66.80 (d, *J* = 5.3 Hz, CH_2*), 55.65 (CH_3, CH_3*), 53.76 (CH_3), 53.72 (CH_3*); ^{31}P NMR (202.44 MHz, CD_3OD): δ 13.9 (HR-ESI) calcd for $C_8H_{18}NO_8NaP$ $[M+Na]^+$ 310.0668, Found 310.0659. Data in agreement with known literature.[1]

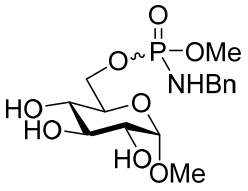


35

Methyl 2,3,4-tri-*O*-acetyl-6-*O*-(methyl phosphoramidyl)-α-D-glucopyranoside (35).

To a solution of compound **8** (30 mg, 0.059 mmol) in ethyl acetate: H_2O (1:0.7 mL) was added $NaBrO_3$ (27 mg, 0.178 mmol, 3 equiv) and $Na_2S_2O_4$ (31 mg, 0.178 mmol, 3 equiv). The reaction mixture ws stirred vigorously for 2 h at room temperature. The reaction was monitored by TLC. Upon completion the reaction mixture was diluted with ethyl acetate (5 mL) and washed with brine. The organic layer was dried over $MgSO_4$, concentrated and purified through column chromatography ($CHCl_3$/MeOH = 19/1) to afford desired compound as a colorless oil (19 mg, 76%).

Colorless oil, $[\alpha]^{28}_D$ 1.7 (*c* 0.5, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$); δ 5.48-5.42 (m, 2H, H-3, H-3*), 5.09 (td, *J* = 11.2, 1.6 Hz, 2H, H-4, H-4*), 4.94 (s, 1H, H-1), 4.93 (s, 1H, H-1*), 4.87 (t, *J* = 2.8 Hz, 1H, H-2), 4.84 (t, *J* = 2.8 Hz, 1H, H-2*), 4.13-4.08 (m, 4H, H6a, H6a*, H6b, H6b*), 3.96-3.94 (m, 2H, H-5, H-5*), 3.73 (d, *J* = 11.5 Hz, 3H, CH_3), 3.72 (d, *J* = 11.4 Hz, 3H, CH_3*), 3.39 (s, 6H, CH_3, CH_3*), 2.82 (bs,4H, *N*H_2, *N*H_2*), 2.05 (s, 6H, CH_3, CH_3*), 2.02 (s, 3H, CH_3), 2.01 (s, 3H, CH_3*), 1.98 (s, 6H, CH_3, CH_3*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.1 (C, C*), 170.06 (C, C*), 170.00 (C), 169.6 (C*), 96.84 (CH, CH*), 70.78 (CH, CH*), 70.16 (CH, CH*), 68.47 (CH), 68.34 (CH*), 67.86 (CH), 67.75 (CH*), 64.34 (d, *J* = 4.1 Hz, CH_2), 64.20 (d, *J* = 4.1 Hz, CH_2*), 55.56 (CH_3), 55.53 (CH_3*), 20.64 (CH_3, CH_3, CH_3), 20.61 (CH_3*, CH_3*, CH_3*); ^{31}P NMR (161.97 MHz, $CDCl_3$): δ 11.5, 10.9; (HR-ESI) calcd for $C_{14}H_{24}NO_{11}NaP$ $[M+Na]^+$ 436.0985, Found 436.0976.



36

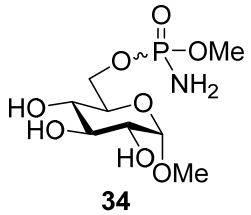
Methyl 6-*O*-(methyl *N*-benzylphosphoramidyl)-α-D-glucopyranoside (36).

Compound **8** (50 mg, 0.099 mmol) was dissolved in a 7:2:1 mixture of CH_3OH:H_2O:Et_3N (4.5 mL). The reaction mixture was stirred at room temperature for 2h and was monitored by TLC. The solution was concentrated under a reduced pressure, followed by toluene azeotrop (10mL x 3) and the resulting residue purified through column chromatography ($CHCl_3$/MeOH = 9/1) to afford desired compound **36** as colorless oil (31 mg, 85%).

We also prepare compound **36** using an alternative method.

To a solution of compound **8** (27 mg, 0.053mmol) in MeOH/H_2O = 9/1 (1mL) was added K_2CO_3 (16 mg, 0.112mmol) and the mixture was stirred for 2h at room temperature. The reaction was monitored by TLC and upon completion neutralized with Amberlite IR-120 (H^+), followed by filtration. The filtrate was concentrated and purified by column chromatography ($CHCl_3$/MeOH = 9/1) to afford desired compound **36** as colorless oil (15 mg, 74%).

Colorless oil, ^{1}H NMR (400 MHz, CD_3OD); δ 7.37-7.29 (m, 8H, ArH, ArH*), 7.25-7.15 (m, 2H, ArH, ArH*) 4.64 (t, *J* = 3.6 Hz, 2H, H-1, H-1*), 4.26-4.18 (m, 2H, H6a, H6a*), 4.16-4.07 (m 6H, H6b, H6b*, *N*HBn, *N*HBn*), 3.68-3.65 (m, 2H, H-5, H-5*), 3.67 (d, *J* = 11.1 Hz, 3H, CH_3), 3.66 (d, *J* = 11.1 Hz, 3H, CH*), 3.61 (td, *J* = 10.2, 1.2 Hz, 2H, H-3, H-3*), 3.39-3.34 (m, 2H, H-2, H-2*), 3.38 (s, 3H, CH_3), 3.36 (s, 3H, CH_3*), 3.30-3.25 (m, 2H, H-4, H-4*); ^{13}C NMR (100MHz, CD_3OD) δ 141.60 (C, C*), 129.4 (CH, CH*), 128.50 (CH, CH*), 128.15 (CH, CH*), 101.3 (CH, CH*), 75.00 (CH, CH*), 73.46 (CH, CH*), 71.99 (d, *J* = 7.5 Hz, CH, CH*), 71.41 (CH), 71.23 (CH*), 67.02 (d, *J* = 5.2 Hz, CH_2), 66.80 (d, *J* = 4.9 Hz, CH_2*), 55.66 (CH_3, CH_3*), 53.78 (CH_3. CH_3*), 45.91(CH_2, CH_2*); ^{31}P NMR (161.97 MHz, CD_3OD): δ 12.1; (HR-ESI) calcd for $C_{15}H_{24}NO_8NaP$ $[M+Na]^+$ 400.1137, Found 400.1136.

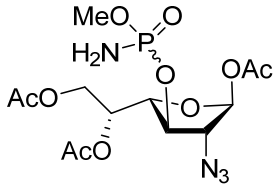


34

Methyl 6-*O*-(methyl phosphoramidyl)-α-D-glucopyranoside (34).

To a solution of compound **36** (11 mg, 0.029 mmol) in EtOH (0.3 mL) was added $Pd(OH)_2$ (60 mg) and the mixture was stirred overnight at room temperature under H_2 atmosphere.[3] Reaction

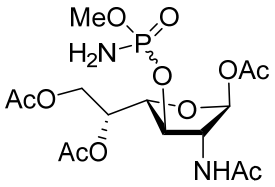
was monitored by TLC. The reaction was diluted with EtOH (2 mL) and was filter through a celite bed. The bed was further washed with EtOH (3 mL), and the EtOH was concentrated under reduced pressure and dried under high vacuum to afford desired compound **34** as a colorless oil in quantitative yield (10 mg). Data is in agreement with the known literature.[1]



37

1,5,6-tri-*O*-acetyl-2-azido-2-deoxy-3-*O*-(methyl phosphoramidyl)-β-D-galactofuranose (37).
To a solution of compound **31** (50 mg, 0.097 mmol) in ethyl acetate (2 mL) a solution of $NaBrO_3$ (44 mg, 0.296mmol, 3 equiv) in H_2O (1 mL) was added by dropper. To the above reaction mixture, a solution of $Na_2S_2O_4$ (50 mg, 0.296mmol, 3 equiv) in H_2O (2mL) was added dropwise using dropper. The reaction mixture stirred vigorously for 1h at room temperature. Reaction was monitored by TLC. Upon completion the reaction mixture was then diluted with ethyl acetate and washed with brine. The organic layers were combined and the mixture was dried over $MgSO_4$, concentrated and purified through column chromatography to afford desired compound as a colorless oil (33 mg, 81%).

Colorless oil, $[\alpha]^{28}_D$ -1.2 (*c* 0.15, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$); δ 6.08 (s, 2H, H-1, H-1*), 5.34 (tt, *J* = 2H, 6.5, 3.8 Hz, H-3, H-3*), 4.45 (td, *J* = 6.6, 3.0 Hz, 2H, H-5, H-5*), 4.38 (dd, *J* = 5.7, 3.3 Hz, 1H, H-4), 4.34 (dd, *J* = 6.1, 3.4 Hz, 1H, H-4*), 4.30-4.17 (m, 6H, H-2, H-2*, H6a, H6a*, H6b, H6b*), 3.75 (d, *J* = 11.4 Hz, 3H, CH_3), 3.74 (d, *J* = 11.2 Hz, 3H, CH_3*), 3.32 (d, *J* = 5.0 Hz, 4H, *N*H_2, *N*H_2*), 2.11 (s, 3H, CH_3), 2.10 (CH_3*), 2.08 (S, 6H, CH_3, CH_3*), 2.02 (s, 6h, CH_3, CH_3*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.65 (C), 170.60 (C, C*), 170.4 (C), 169.4 (C*), 169.3 (C), 100.0 (CH), 99.94 (CH*), 82.70 (d, *J* = 6.8 Hz, CH), 82.37 (d, *J* = 8.5 Hz, CH*), 78.92 (d, *J* = 5.3 Hz, CH), 78.77 (d, *J* = 4.2 Hz, CH*), 70.7 (CH), 70.54 (d, *J* = 2.8 Hz, CH*), 68.80 (CH), 68.71 (CH*), 62.60 (CH_2, CH_2*), 53.53 (t, *J* = 7.6 Hz, CH_3, CH_3*), 20.93 (CH_3, CH_3*), 20.75 (CH_3, CH_3*), 20.64 (CH_3, CH_3*); ^{31}P NMR (161.97 MHz, $CDCl_3$): δ 10.9, 10.6; (HR-ESI) calcd for $C_{13}H_{21}N_4O_{10}NaP$ $[M+Na]^+$ 447.0893, Found 447.0901.



38

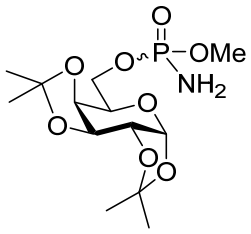
1,5,6-tri-*O*-acetyl-2-acetamido-2-deoxy-3-*O*-(methyl phosphoramidyl)-β-D-galactofuranose (38).

To a solution of compound **37** (30g, 0.070 mmol) in EtOH (1 mL) was added Pd $(OH)_2$ (150 mg) and stirred reaction mixture for 5 h at room temperature under H_2 atmosphere.[3] Reaction was monitored by TLC. After completion filter the reaction mixture through a celite bed. The bed was further washed with EtOH (3 mL). The EtOH was concentrated under a reduced pressure and the residue was dried under high vacuum, which was dissolved in pyridine (1 mL) and added Ac_2O (8.02 μL, 0.0848 mmol, 1.0 equiv). The mixture was stirred for additional 3 h at room temperature. The reaction was monitored by TLC, and upon completion was purified through column chromatography ($CHCl_3$/MeOH = 4/1) to afford desired compound **38** over two steps (22 mg, 71%).

Alternatively, to a solution of compound **31** (30 mg, 0.058 mmol) in EtOH (1 mL) was added Pd $(OH)_2$ (150 mg) and stirred reaction mixture overnight at room temperature under H_2 atmosphere.[3] The reaction was monitored by TLC. Upon completion, the mixture was filtered through a celite bed. The bed was further washed with EtOH (3 mL) and the EtOH was concentrated under a reduced pressure and the residue was dried under high vacuum. The residue was dissolved in pyridine (1 mL), added Ac_2O (1.0 equiv) and stirred for additional 3 h at room temperature. The reaction was monitored by TLC, and upon completion, co-evaporated with toluene (10 mL x 3), and the residue was purified through column chromatography ($CHCl_3$/MeOH = 4/1) to afford the desired compound **38** over two steps (17 mg, 68%).

Colorless oil, $[\alpha]^{28}_D$ 1.2 (*c* 1.0, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$); δ 7.08 (d, *J* = 4.5 Hz, 1H, NH), 7.06 (d, *J* = 4.4 Hz, 1H, *N*H*), 6.12 (d, *J* = 0.9 Hz, 1H, H-1), 6.08 (s, 1H, H-1*), 5.28-5.24 (m, 2H, H-3, H-3*), 4.60-4.50 (m, 2H, H-5, H-5*), 4.42-4.28 (m, 6H, H4, H-4*, H-2, H-2*, H6a, H6b), 4.17 (dd, *J* = 12.0, 7 Hz, 2H, H6a*, H6b*), 3.71 (d, *J* = 11.3 Hz, 3H, CH_3), 3.70 (d, *J* = 11.5 Hz, 3H, CH_3*), 3.53 (d, *J* = 5.3 Hz, 2H, *N*H_2), 3.50 (d, *J* = 5.8 Hz, 2H, *N*H_2*), 2.11 (s, 3H, CH_3), 2.10 (S, 3H, CH_3*), 2.08 (S, 3H, CH_3), 2.07 (S, 3H, CH_3*), 2.02 (S, 6H, CH_3, CH_3*), 1.98 (S, 6H,

CH_3, CH_3*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.9 (C), 170.8 (C*), 170.7 (C), 170.6 (C, C*), 170.4 (C), 169.6 (C*), 169.5 (C), 99.54 (CH), 99.28 (CH*), 82.19 (d, *J* = 7.5 Hz, CH), 81.72 (d, *J* = 8.1 Hz, CH*), 79.45 (d, *J* = 4.1 Hz, CH), 78.63 (d, *J* = 5.3 Hz, CH*), 69.53 (CH), 69.20 (CH*), 62.61 (CH_2) , 62.50 (CH_2*), 61.34 (CH), 60.97 (CH*), 53.55 (d, *J* = 5.5 Hz, CH3), 53.44 (d, *J* = 5.6 Hz, CH_3*), 22.88 (CH_3), 22.83 (CH_3*), 21.03 (CH_3, CH_3*), 20.81 (CH_3, CH_3*), 20.64 (CH_3, CH_3*); ^{31}P NMR (161.97 MHz, $CDCl_3$): δ 11.3, 10.6; (HR-ESI) calcd for $C_{15}H_{25}N_2O_{11}NaP$ $[M+Na]^+$ 463.1094, Found 463.1089.



1,2:3,4-di-*O*-isopropylidene-6-*O*-(methyl phosphoramidyl)-D-galactopyranose.

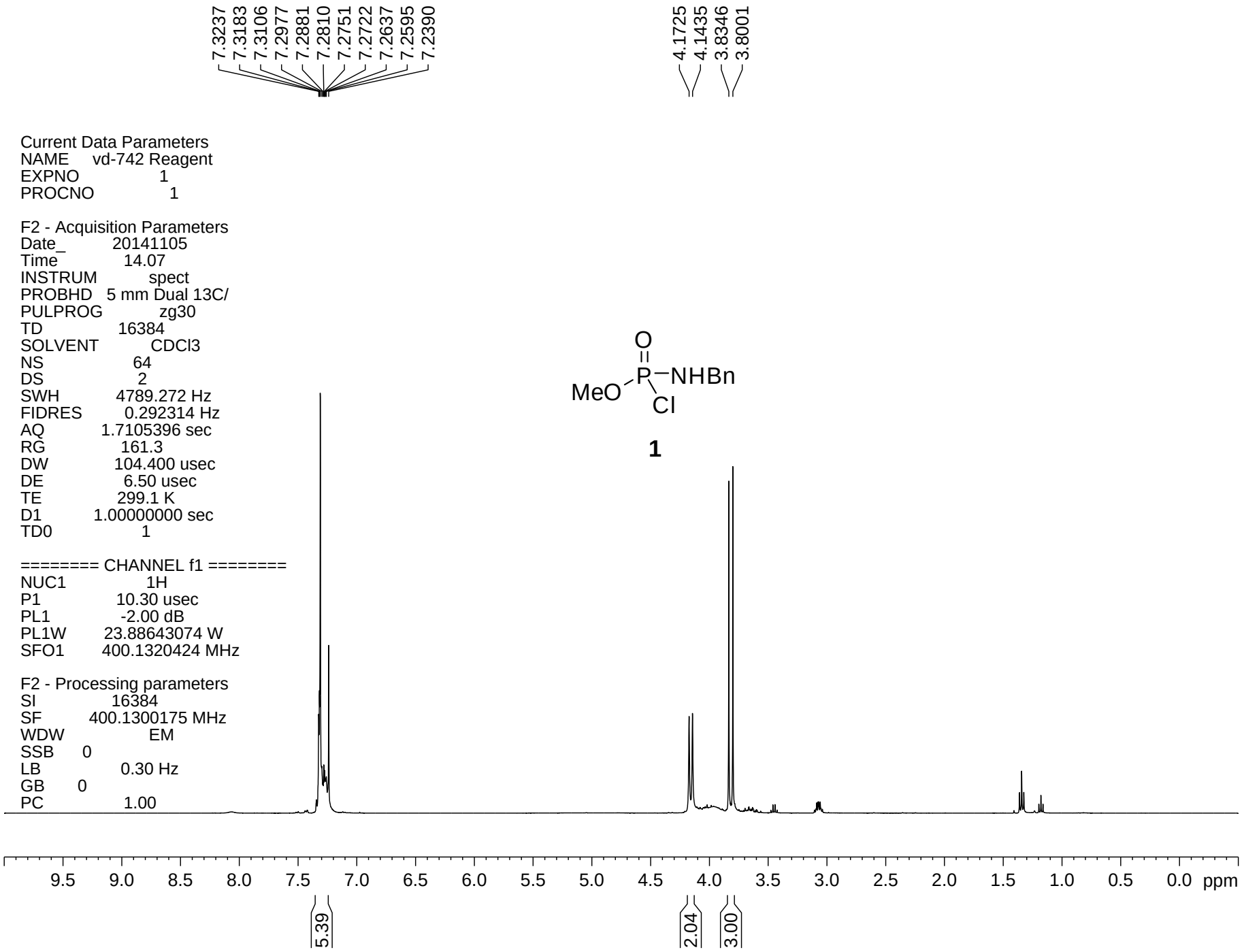
To a solution of compound **11** (133 mg, 0.300 mmol) in ethyl acetate (4 mL) a solution of $NaBrO_3$ (136 mg, 0.900 mmol, 3 equiv) in H_2O (3 mL) was added by dropper. To the above reaction mixture, a solution of $Na_2S_2O_4$ (157 mg, 0.900 mmol, 3 equiv) in H_2O (6mL) was added dropwise using dropper. The reaction mixture stirred vigorously for 1h at room temperature. Reaction was monitored by TLC. Upon completion the reaction mixture was then diluted with ethyl acetate and washed with brine. The organic layer was dried over $MgSO_4$, concentrated and purified through column chromatography to afford desired compound as a colorless oil (29 mg, 83%).

Colorless oil, IR ($CHCl_3$) ν 3397, 2987, 1383, 1253, 1212, 1168, 1068, 1002, 903, 857, 776 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$); δ 5.51 (s, 1H, H-1), 5.50 (s, 1H, H-1*), 4.60 (t, *J* = 2.2 Hz, 1H, H-3), 4.57 (t, *J* = 2.2 Hz, 1H, H-3*), 4.30 (ddd, *J* = 5.0, 2.5, 1.1 Hz, 2H, H-2, H-2*), 4.23 (t, *J* = 1.8 Hz, 1H, H-4), 4.20 (t, *J* = 1.7 Hz, 1H, H-4*), 4.19-4.01 (m, 6H, H-5, H-5*, H6a, H6b, H6a*, H6b*), 3.71 (d, *J* = 11.3 Hz, 3H, $POCH_3$), 3.70 (d, *J* = 11.4 Hz, 3H, $POCH_3$*), 3.0 (d, *J* = 11.0 Hz, 4H, *N*H_2, *N*H_2*), 1.51 (s, 3H, CH_3), 1.50 (s, 3H, CH_3*), 1.40 (s, 3H, CH_3), 1.40 (s, 3H, CH_3*), 1.30 (s, 12H, CH_3, CH_3*, CH_3, CH_3*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 109.66 (C), 109.61 (C*), 108.8 (C, C*), 96.29 (CH), 96.26 (CH*), 70.81 (CH), 70.67 (CH*), 70.45 (CH), 70.42 (CH*), 67.50 (CH), 67.43 (CH*), 66.93 (CH), 66.87 (CH*), 65.61 (d, *J* = 5.5 Hz, CH_2), 65.38 (d, *J* = 5.3 Hz, CH_2*), 53.32 (CH), 53.26 (CH*), 25.91 (CH_3 x 2, CH_3* x 2), 24.90 (CH_3), 24.88 (CH_3*), 24.45 (CH_3),

24.40 (CH_3*) ^{31}P NMR (161.97 MHz, $CDCl_3$): δ 11.5, 11.1; (HR-ESI) calcd for $C_{13}H_{24}NO_8NaP$ $[M+Na]^+$ 376.1137, Found 376.1129. Data in agreement with known literature.[1]

References:

1. R. Ashmus and T. L. Lowary, *Org. Lett*., 2014, **16**, 2518.
2. M. Matweijuk and J. Thiem, *Eur. J. Org. Chem*., 2012, 2180.
3. A. A. Joseph, V. M. Dhurandhare, C.-W. Chang, V.P. Verma, G. P. Mishra, C.-C. Ku, C.-C. Lin and C.-C. Wang, *Chem. Commun.*, 2015, **51**, 104.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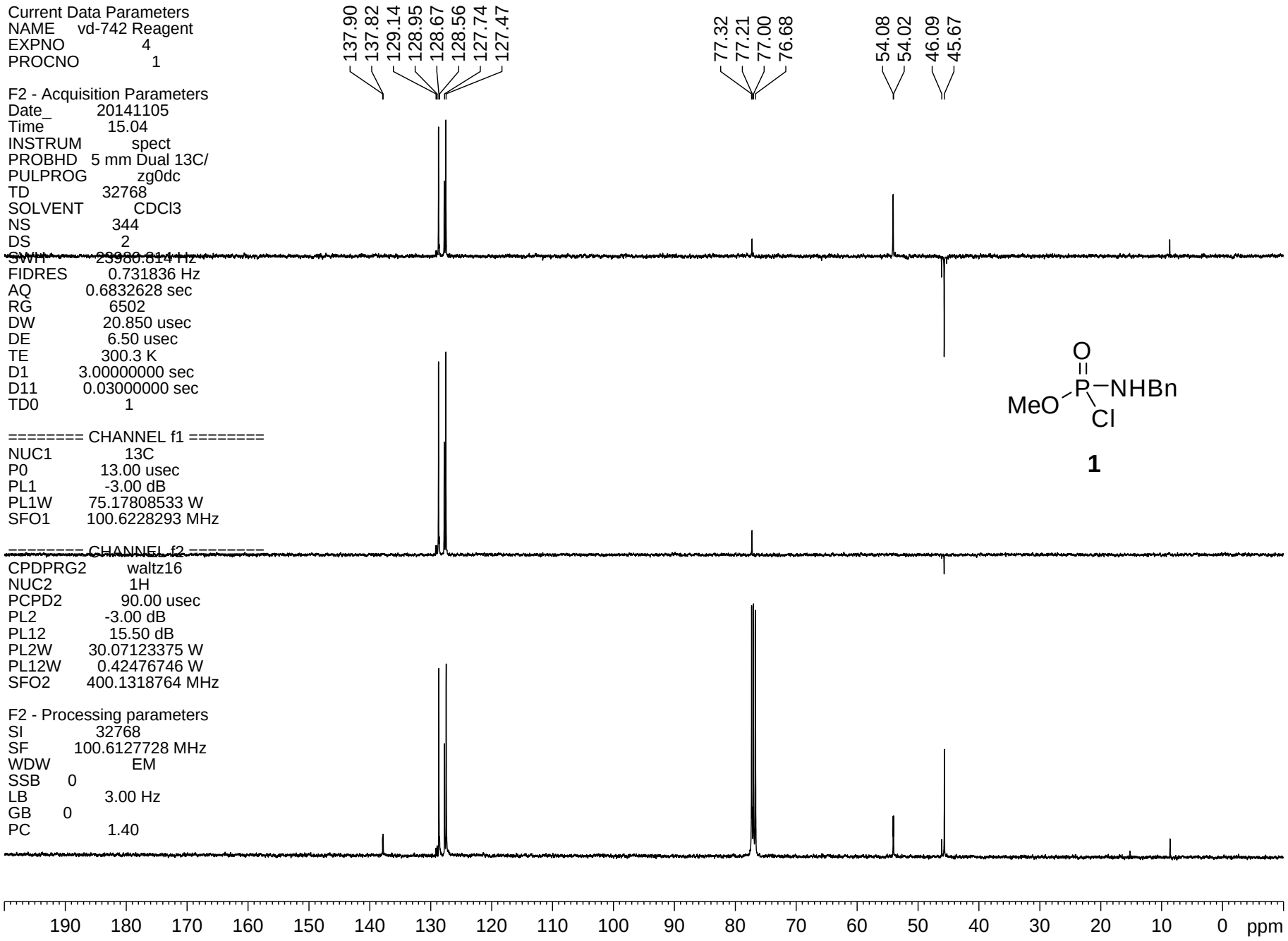
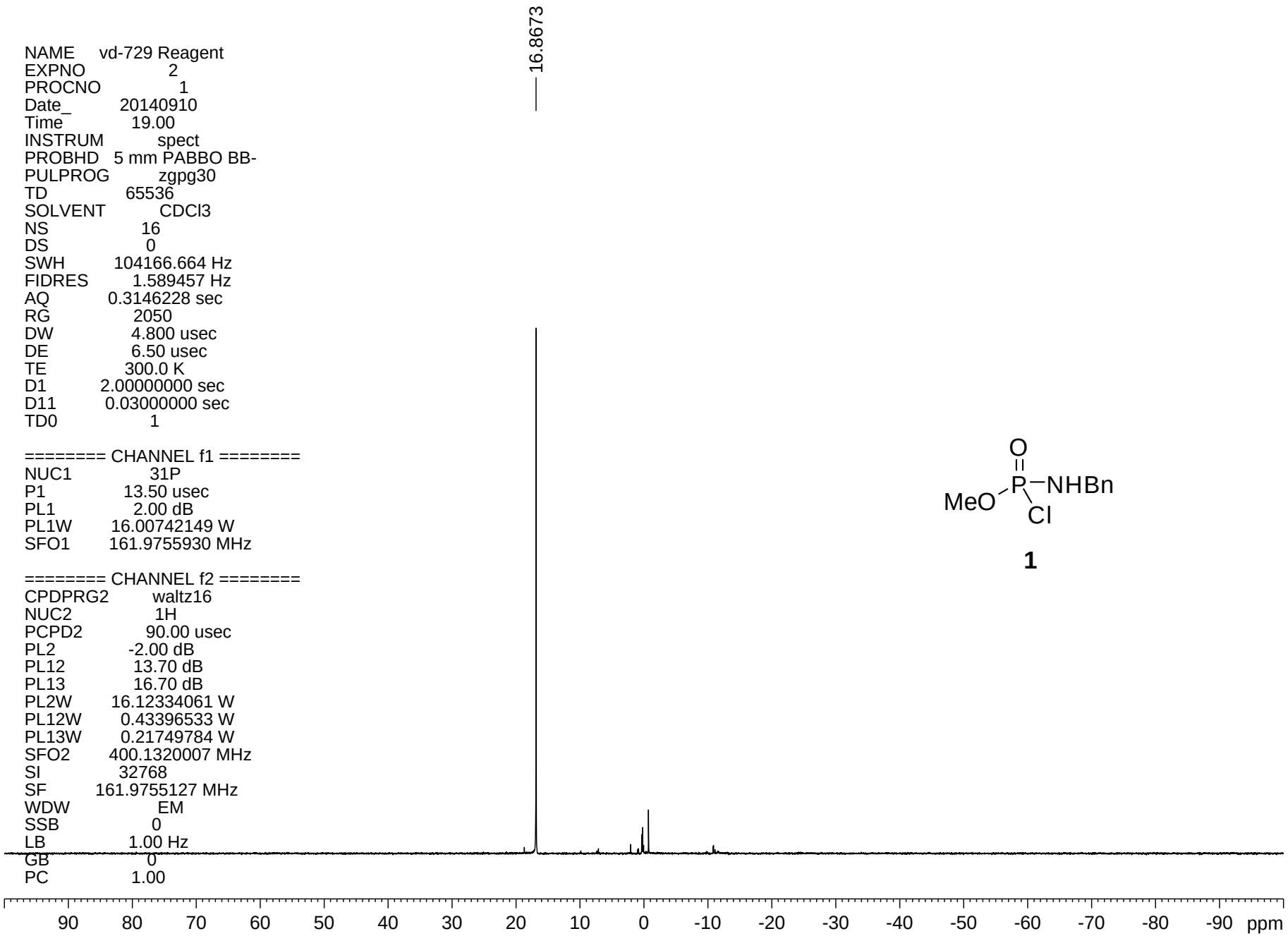
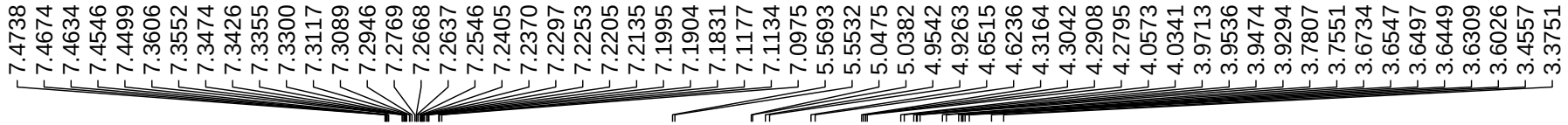
7.2637
7.2595
7.2390
4.1725
4.1435
3.8346
3.8001
Current Data Parameters
NAME vd-742 Reagent
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141105
Time 14.07
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 64
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7105396 sec
RG 161.3
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 400.1300175 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
P
NHBn
MeO
Cl
1
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
5.39
2.04
3.00


Current Data Parameters
NAME vd-742 Reagent
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141105
Time 15.04
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg0dc
TD 32768
SOLVENT CDCl3
NS 344
DS 2
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 0.6832628 sec
RG 6502
DW 20.850 usec
DE 6.50 usec
TE 300.3 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 -3.00 dB
PL12 15.50 dB
PL2W 30.07123375 W
PL12W 0.42476746 W
SFO2 400.1318764 MHz
F2 - Processing parameters
SI 32768
SF 100.6127728 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
137.90
137.82
129.14
128.95
128.67
128.56
127.74
127.47
77.32
77.21
77.00
76.68
54.08
54.02
46.09
45.67
O
P
NHBn
MeO
Cl
1
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


16.8673
NAME vd-729 Reagent
EXPNO 2
PROCNO 1
Date_ 20140910
Time 19.00
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
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
======== 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 13.70 dB
PL13 16.70 dB
PL2W 16.12334061 W
PL12W 0.43396533 W
PL13W 0.21749784 W
SFO2 400.1320007 MHz
SI 32768
SF 161.9755127 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
O
P
NHBn
MeO
Cl
1
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 ppm

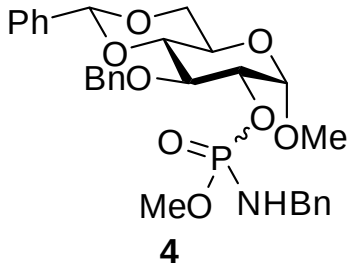


Current Data Parameters
NAME vd-711 AV400
EXPNO 1
PROCNO 1

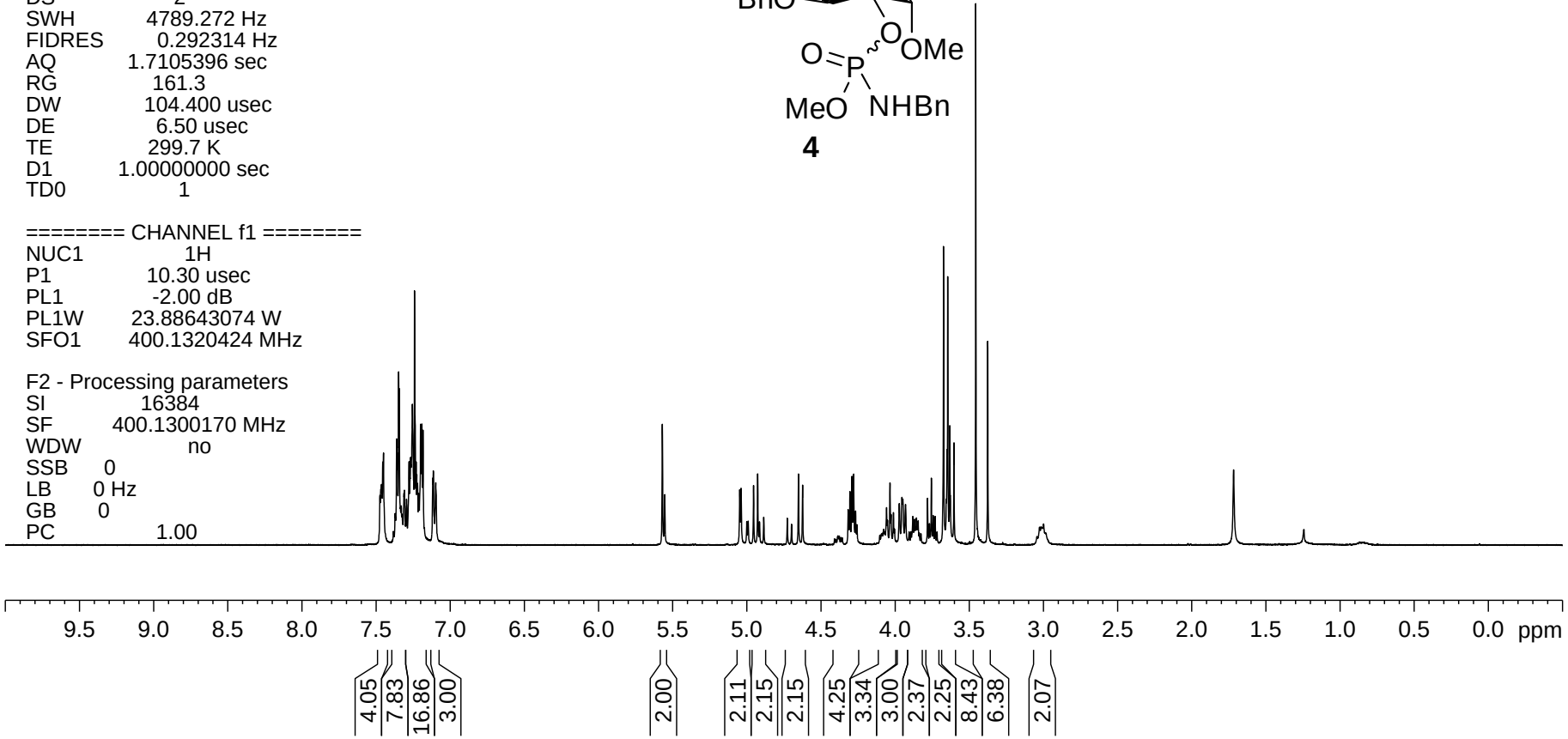
F2 - Acquisition Parameters
Date_ 20140809
Time 17.14
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 64
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7105396 sec
RG 161.3
DW 104.400 usec
DE 6.50 usec
TE 299.7 K
D1 1.00000000 sec
TD0 1

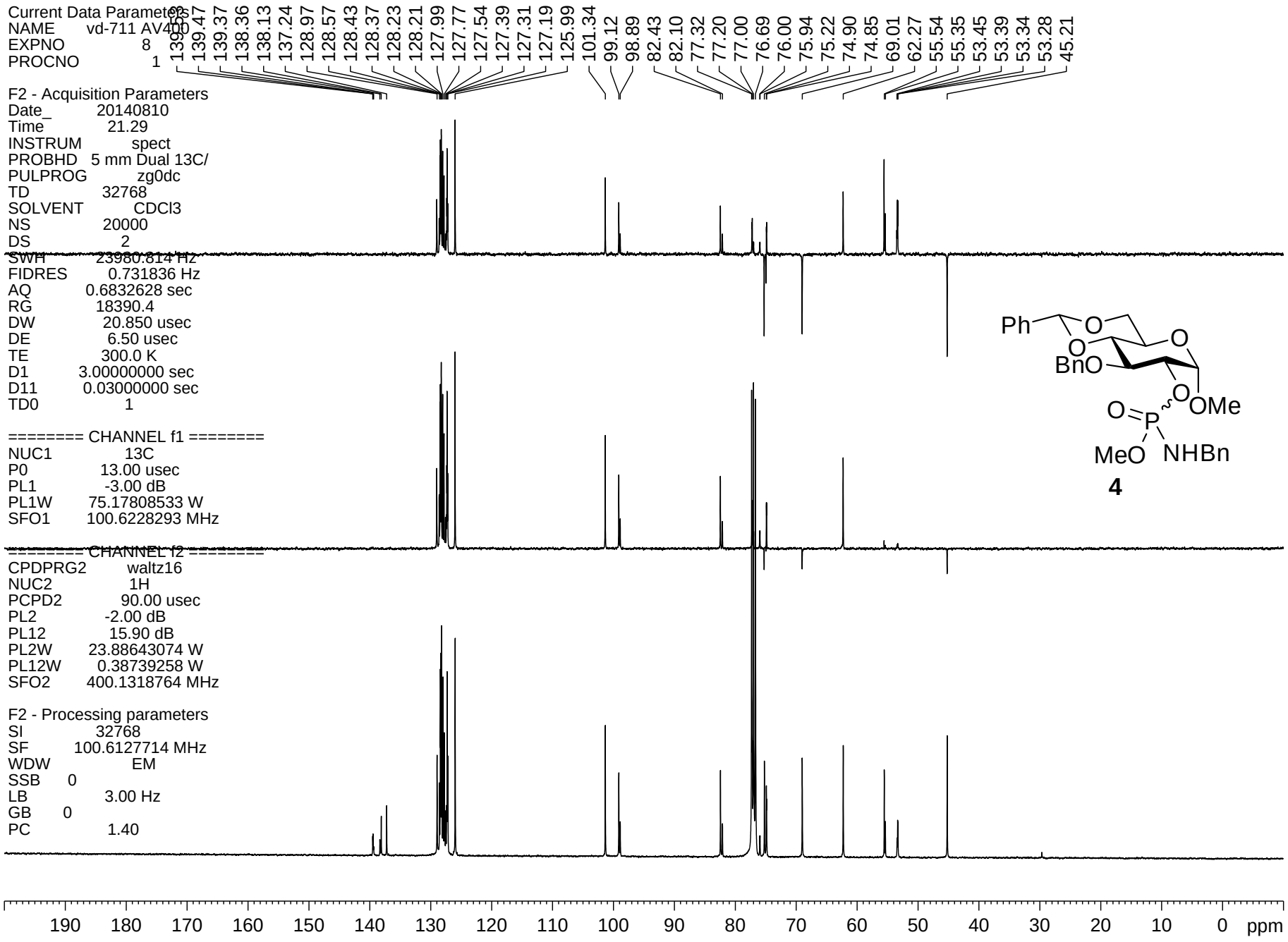
======== CHANNEL f1 ========
NUC1 1H
P1 10.30 usec
PL1 -2.00 dB
PL1W 23.88643074 W
SFO1 400.1320424 MHz

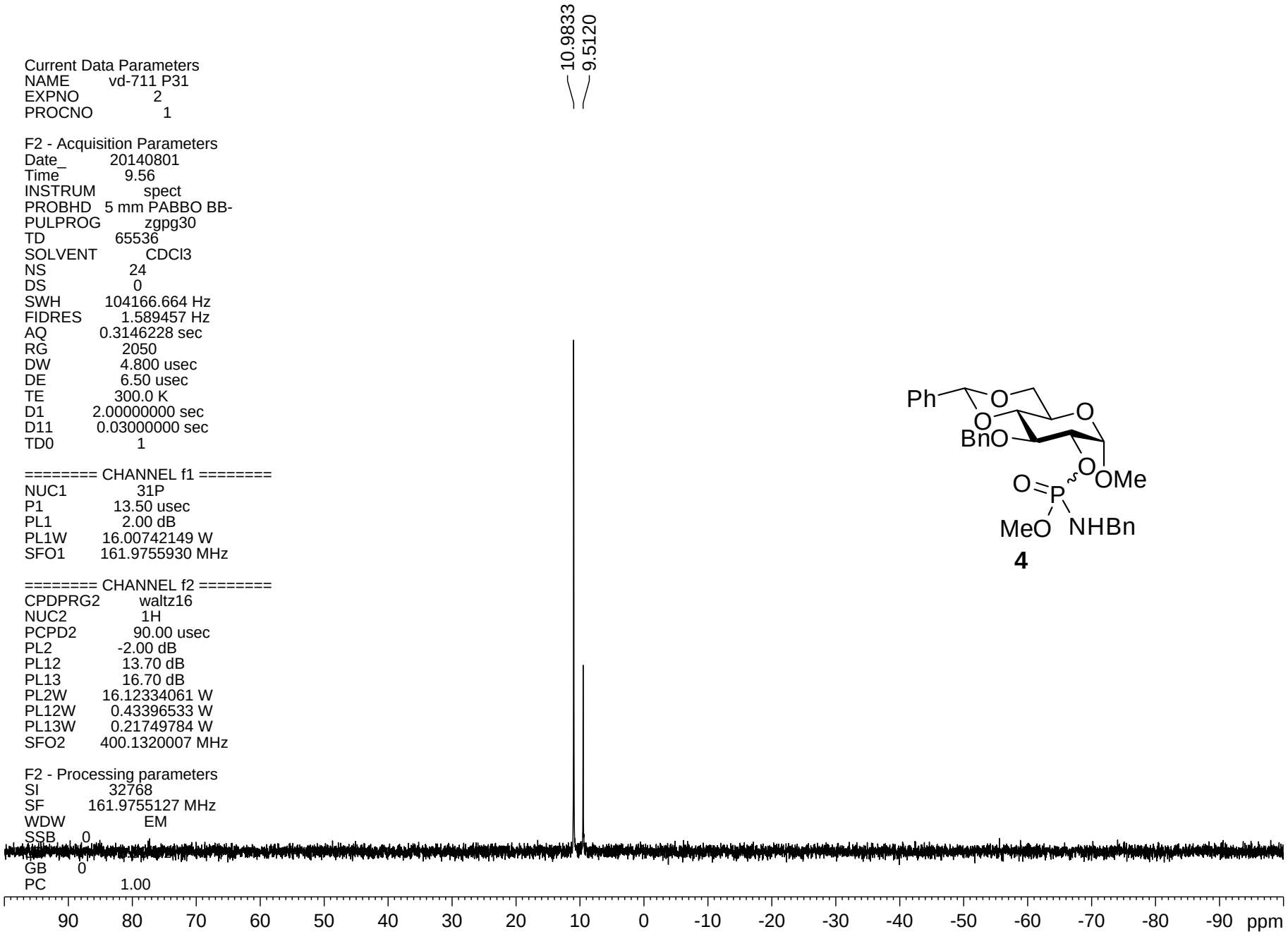
F2 - Processing parameters
SI 16384
SF 400.1300170 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

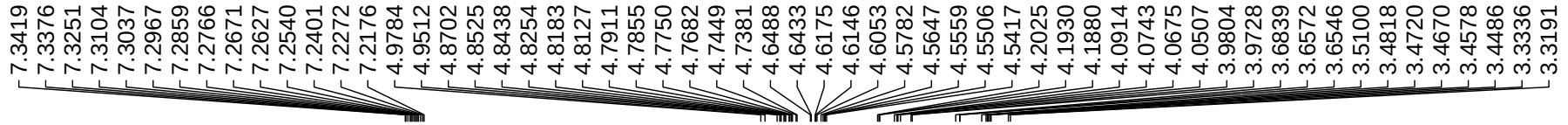


4




Current Data Parameters
NAME vd-711 AV400
EXPNO 8
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140810
Time 21.29
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg0dc
TD 32768
SOLVENT CDCl3
NS 20000
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 300.0 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 100.6127714 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
139.58
139.47
139.37
138.36
138.13
137.24
128.97
128.57
128.43
128.37
128.23
128.21
127.99
127.77
127.54
127.39
127.31
127.19
125.99
101.34
99.12
98.89
82.43
82.10
77.32
77.20
77.00
76.69
76.00
75.94
75.22
74.90
74.85
69.01
62.27
55.54
55.35
53.45
53.39
53.34
53.28
45.21
Ph
O
O
BnO
O
O
OMe
O
P
MeO
NHBn
4
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


10.9833
9.5120
Current Data Parameters
NAME vd-711 P31
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140801
Time 9.56
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 24
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
======== 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 13.70 dB
PL13 16.70 dB
PL2W 16.12334061 W
PL12W 0.43396533 W
PL13W 0.21749784 W
SFO2 400.1320007 MHz
F2 - Processing parameters
SI 32768
SF 161.9755127 MHz
WDW EM
SSB 0
LB 1
GB 0
PC 1.00
Ph
O
O
O
BnO
O
OMe
O
P
MeO
NHBn
4
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
ppm

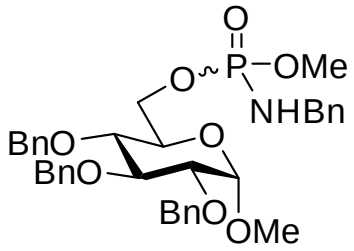


Current Data Parameters
NAME vd-692 all
EXPNO 1
PROCNO 1

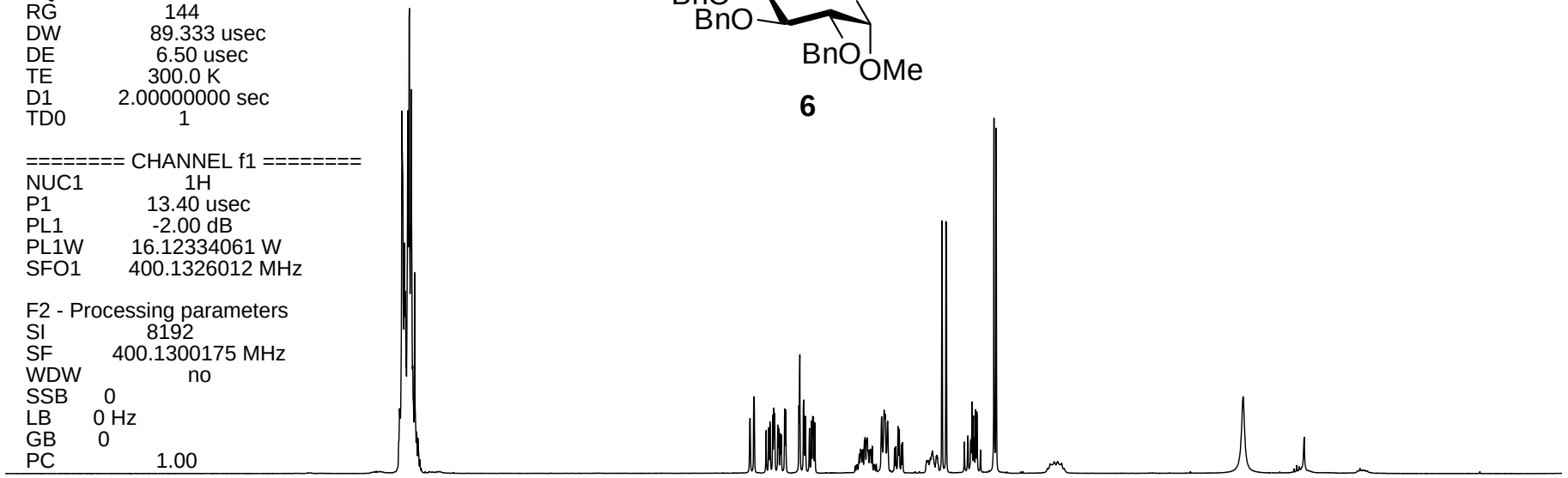
F2 - Acquisition Parameters
Date_ 20140606
Time 22.33
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 64
DS 0
SWH 5597.015 Hz
FIDRES 0.341615 Hz
AQ 1.4636873 sec
RG 144
DW 89.333 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

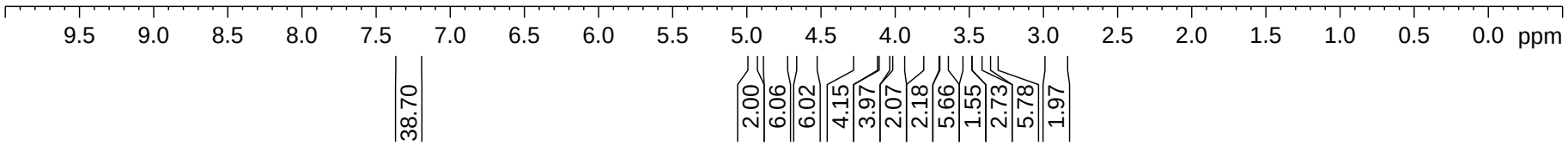
======== CHANNEL f1 ========
NUC1 1H
P1 13.40 usec
PL1 -2.00 dB
PL1W 16.12334061 W
SFO1 400.1326012 MHz

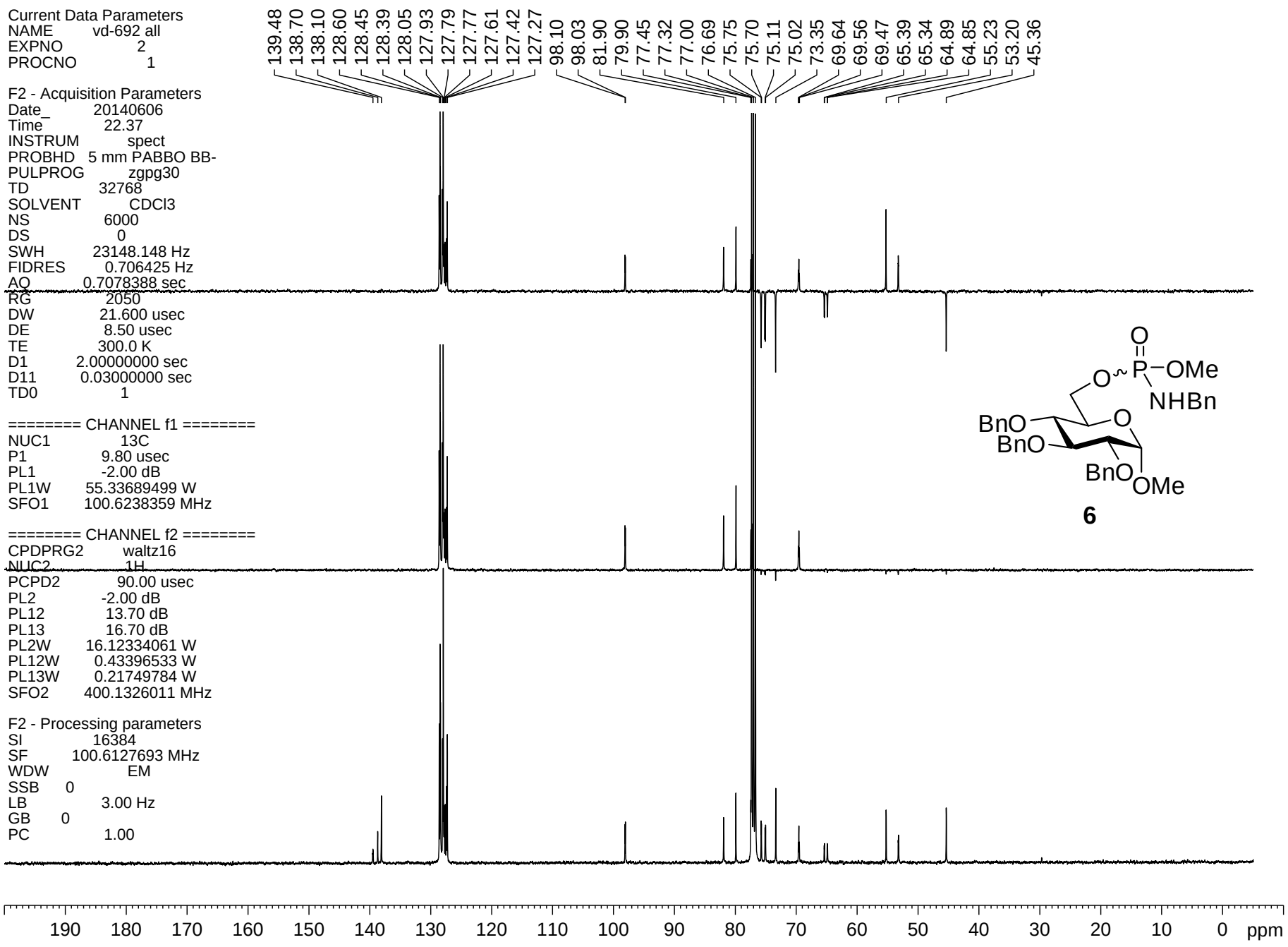
F2 - Processing parameters
SI 8192
SF 400.1300175 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

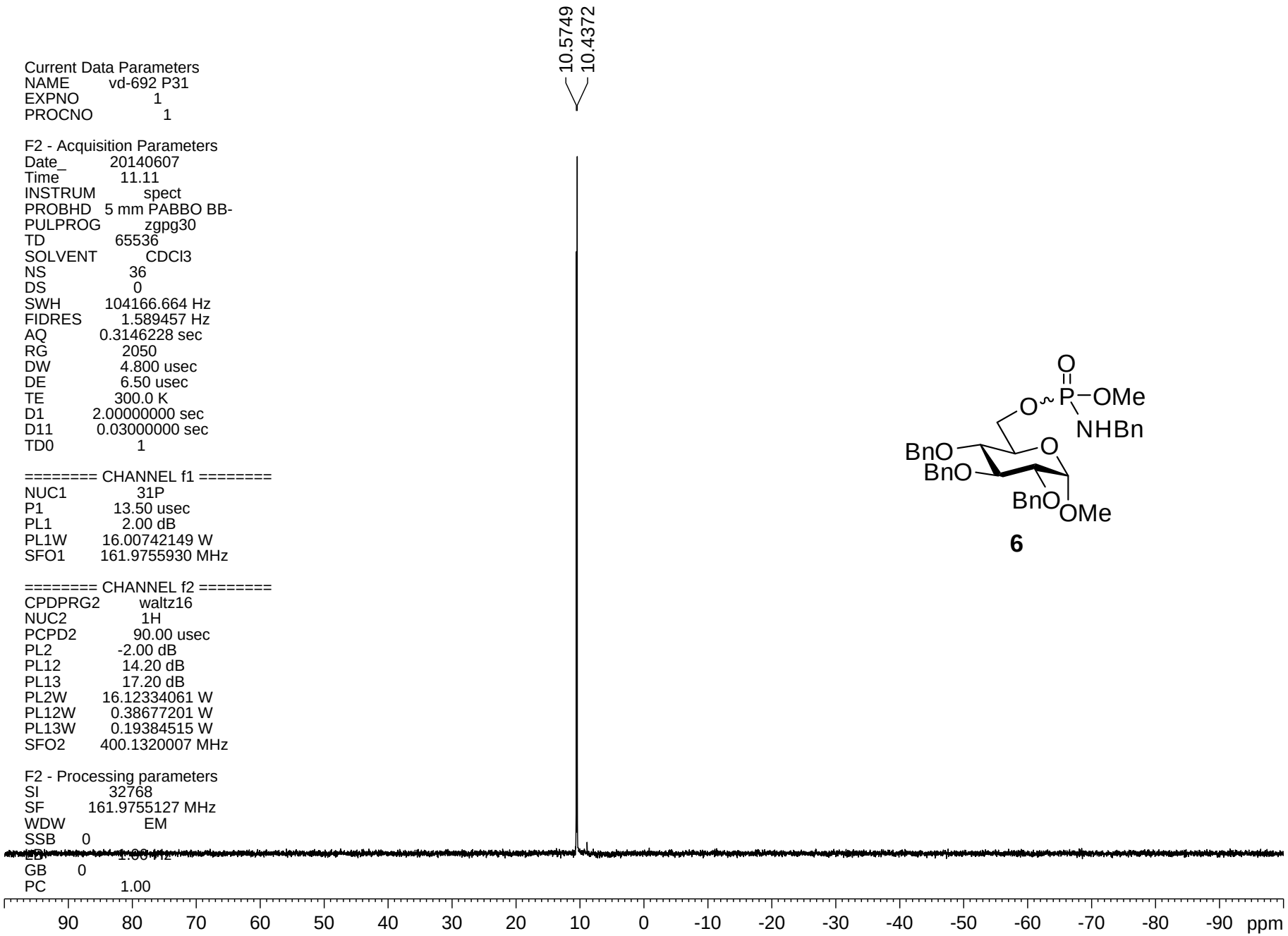


6






Current Data Parameters
NAME vd-692 all
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140606
Time 22.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 6000
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 100.6127693 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
139.48
138.70
138.10
128.60
128.45
128.39
128.05
127.93
127.79
127.77
127.61
127.42
127.27
98.10
98.03
81.90
79.90
77.45
77.32
77.00
76.69
75.75
75.70
75.11
75.02
73.35
69.64
69.56
69.47
65.39
65.34
64.89
64.85
55.23
53.20
45.36
O
O
P
OMe
NHBn
BnO
BnO
O
BnO
OMe
6
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


10.5749
10.4372
Current Data Parameters
NAME vd-692 P31
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140607
Time 11.11
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 36
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
======== 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
F2 - Processing parameters
SI 32768
SF 161.9755127 MHz
WDW EM
SSB 0
GB 0
PC 1.00
O
O
P
OMe
NHBn
BnO
BnO
O
BnO
OMe
6
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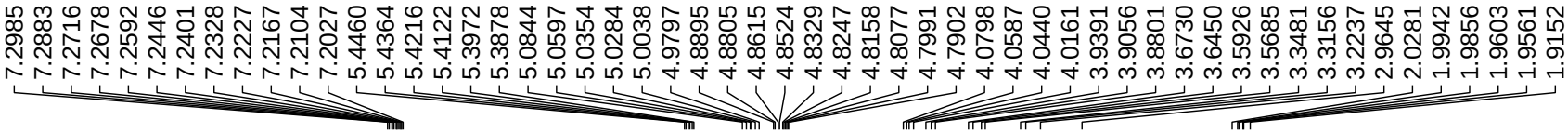
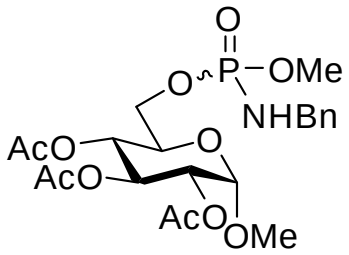


Current Data Parameters
NAME gpm-190
EXPNO 1
PROCNO 1

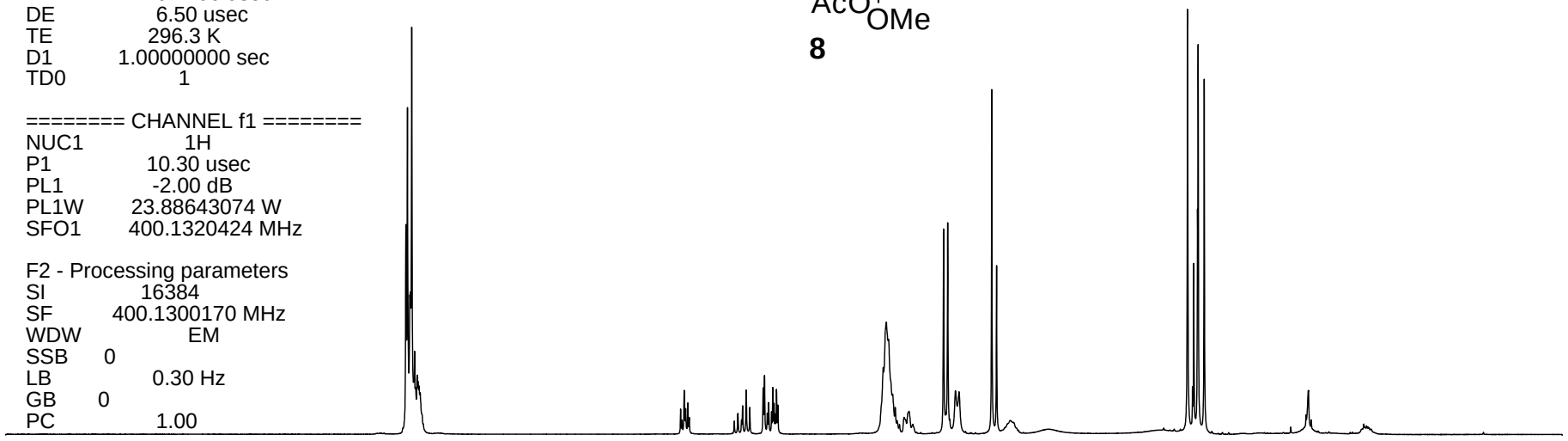
F2 - Acquisition Parameters
Date_ 20140927
Time 20.08
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 8
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7105396 sec
RG 57
DW 104.400 usec
DE 6.50 usec
TE 296.3 K
D1 1.00000000 sec
TD0 1

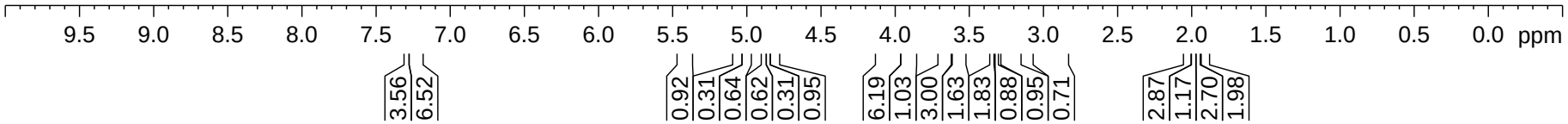
======== CHANNEL f1 ========
NUC1 1H
P1 10.30 usec
PL1 -2.00 dB
PL1W 23.88643074 W
SFO1 400.1320424 MHz

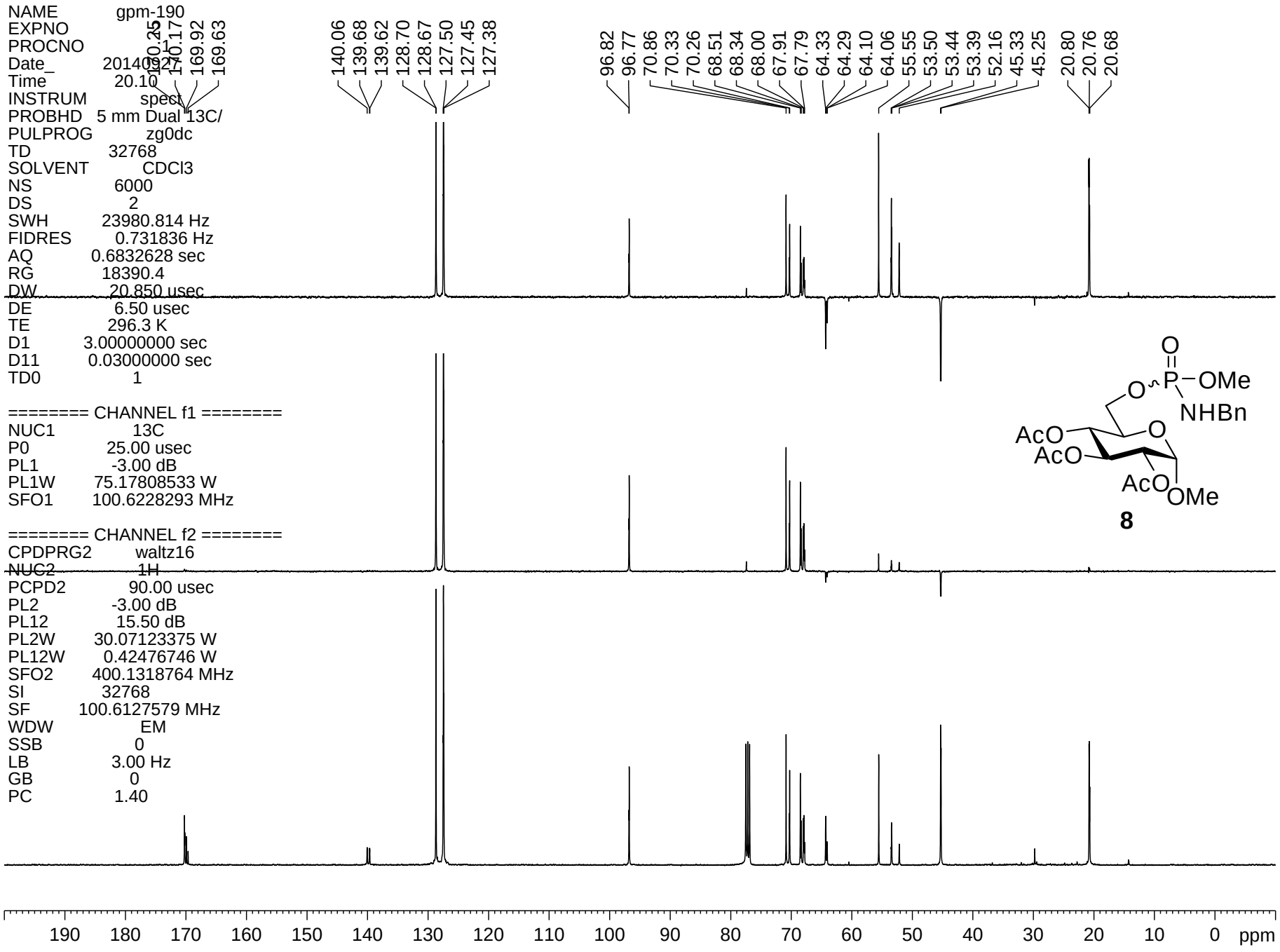
F2 - Processing parameters
SI 16384
SF 400.1300170 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

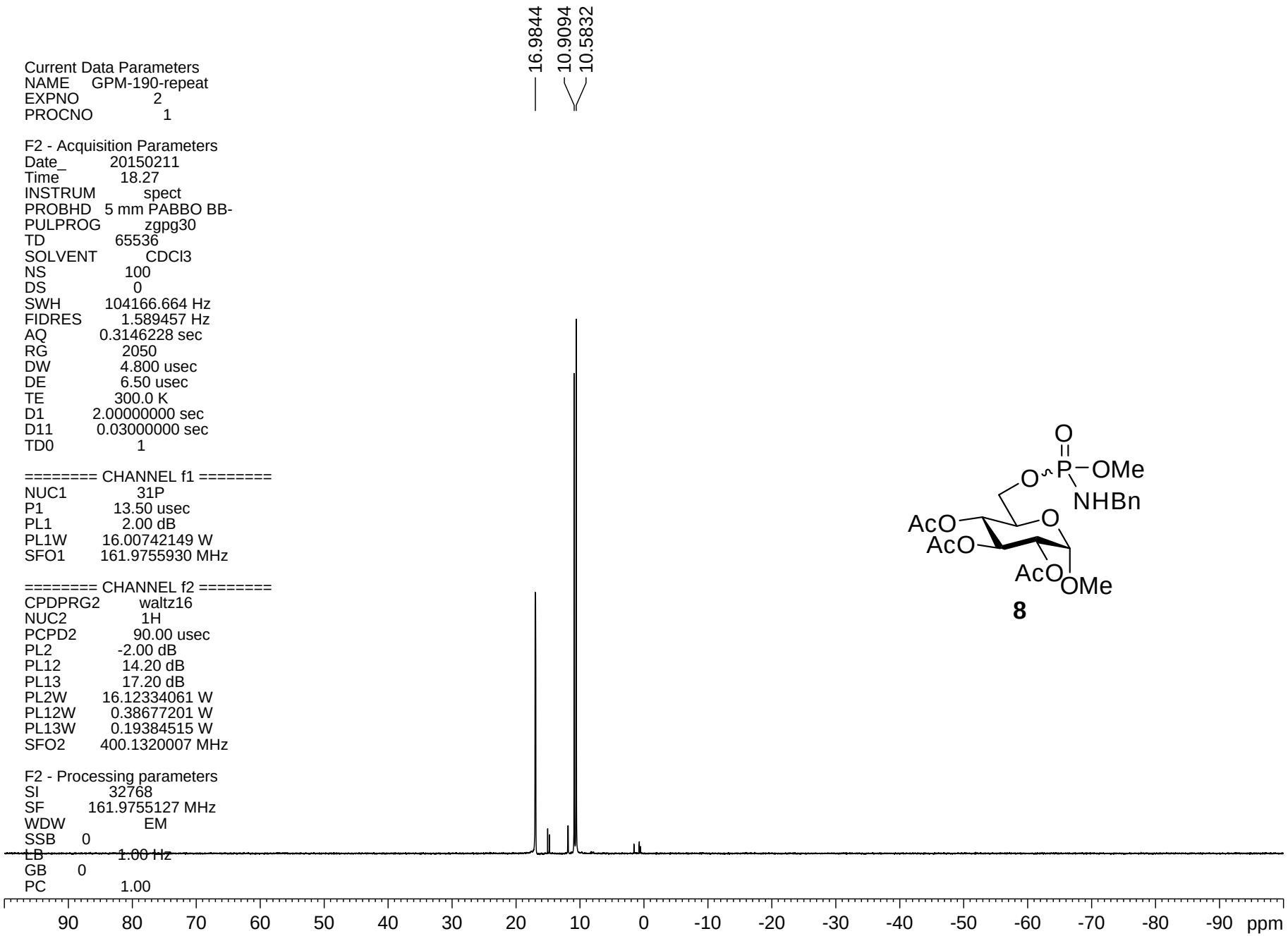


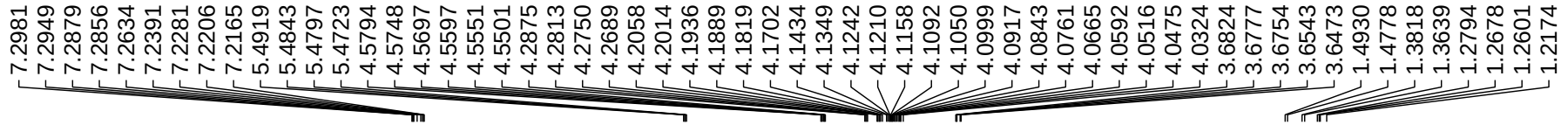
8






NAME gpm-190
EXPNO 1
PROCNO 1
Date_ 20140927
Time 20.10
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg0dc
TD 32768
SOLVENT CDCl3
NS 6000
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 296.3 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P0 25.00 usec
PL1 -3.00 dB
PL1W 75.17808533 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 15.50 dB
PL2W 30.07123375 W
PL12W 0.42476746 W
SFO2 400.1318764 MHz
SI 32768
SF 100.6127579 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
170.25
170.17
169.92
169.63
140.06
139.68
139.62
128.70
128.67
127.50
127.45
127.38
96.82
96.77
70.86
70.33
70.26
68.51
68.34
68.00
67.91
67.79
64.33
64.29
64.10
64.06
55.55
53.50
53.44
53.39
52.16
45.33
45.25
20.80
20.76
20.68
O
O
P
OMe
NHBn
AcO
AcO
O
AcO
OMe
8
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


16.9844
10.9094
10.5832
Current Data Parameters
NAME GPM-190-repeat
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150211
Time 18.27
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 100
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
======== 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
F2 - Processing parameters
SI 32768
SF 161.9755127 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
O
P
OMe
NHBn
O
AcO
AcO
O
AcO
OMe
8
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 ppm

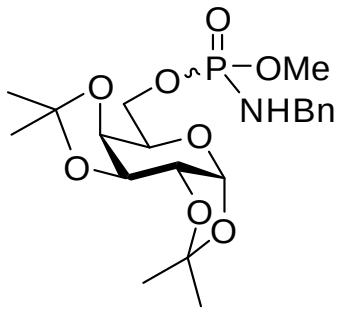


Current Data Parameters
NAME vd-690 all
EXPNO 1
PROCNO 1

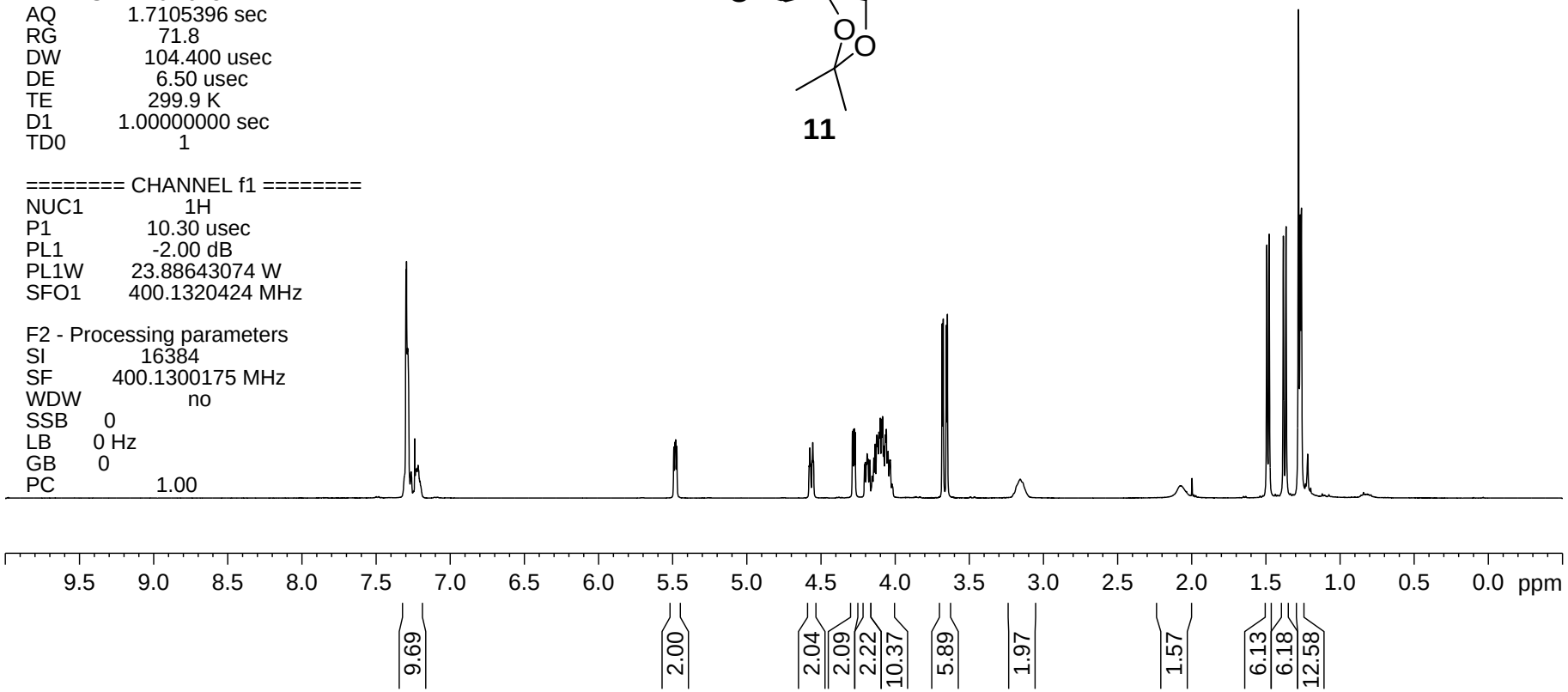
F2 - Acquisition Parameters
Date_ 20140521
Time 21.56
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 64
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7105396 sec
RG 71.8
DW 104.400 usec
DE 6.50 usec
TE 299.9 K
D1 1.00000000 sec
TD0 1

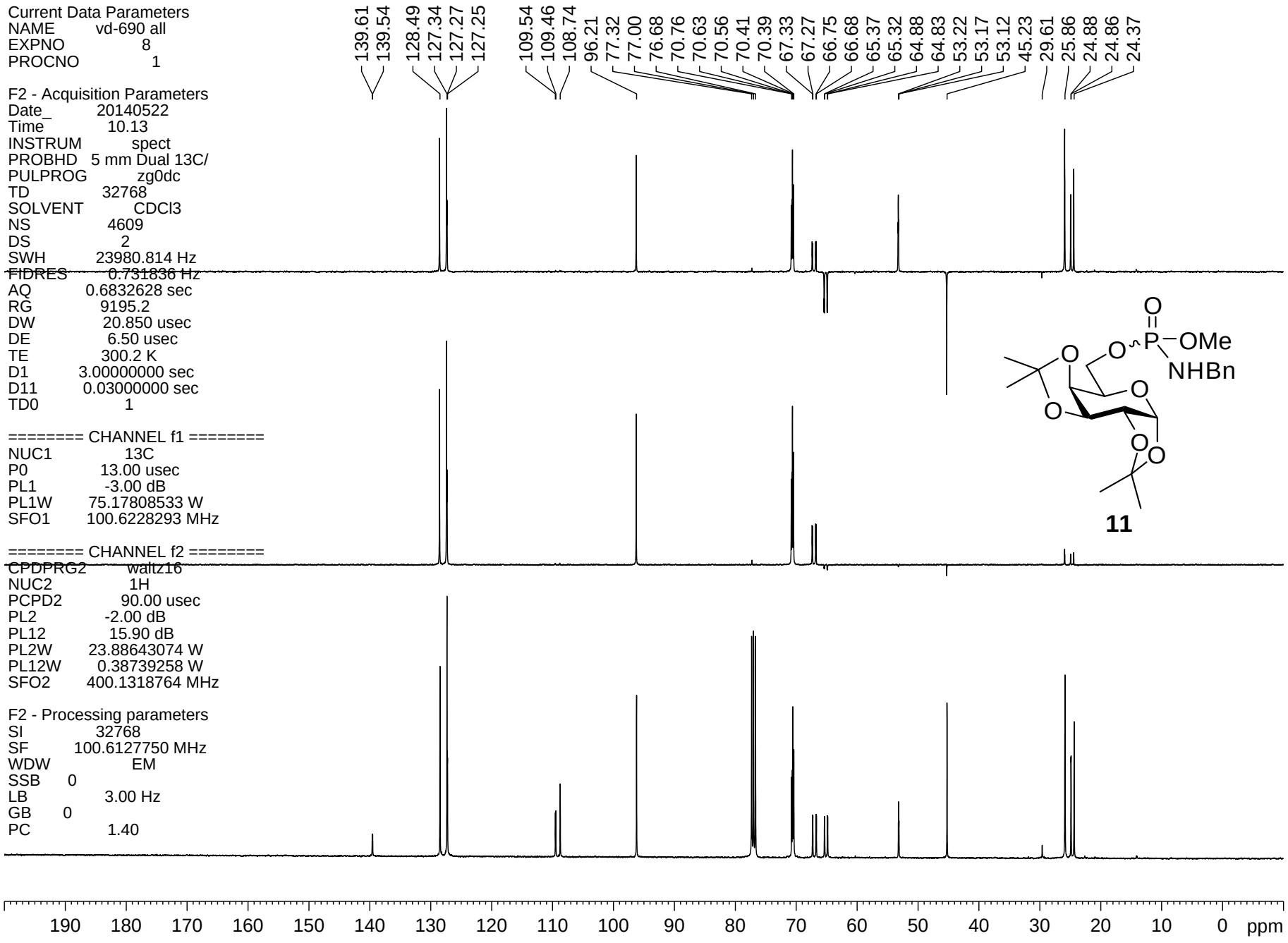
======== CHANNEL f1 ========
NUC1 1H
P1 10.30 usec
PL1 -2.00 dB
PL1W 23.88643074 W
SFO1 400.1320424 MHz

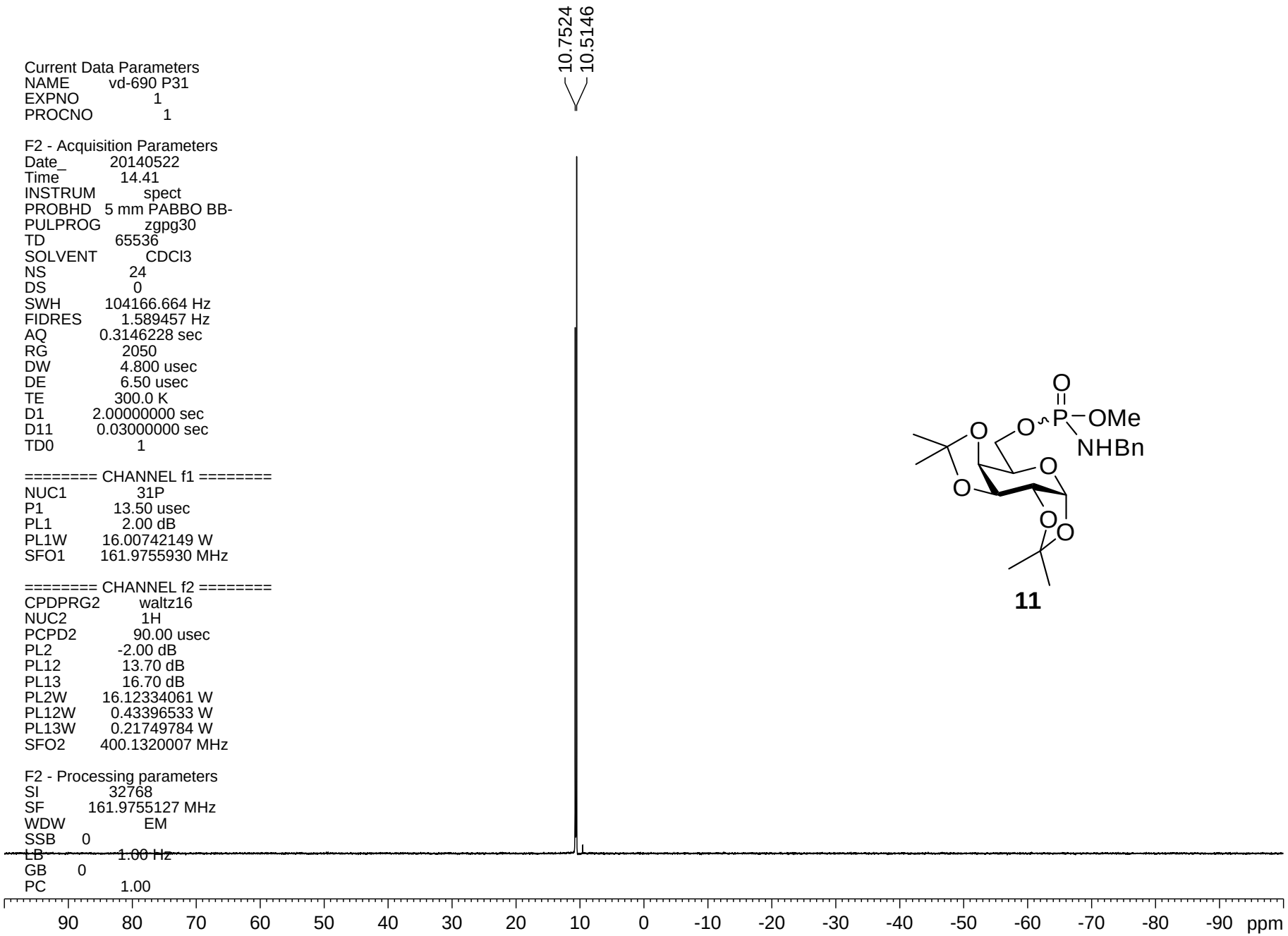
F2 - Processing parameters
SI 16384
SF 400.1300175 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

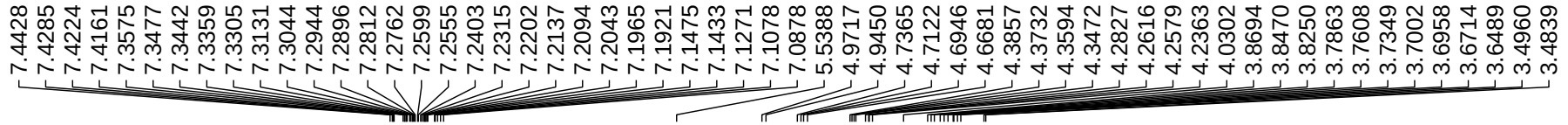


11




Current Data Parameters
NAME vd-690 all
EXPNO 8
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140522
Time 10.13
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg0dc
TD 32768
SOLVENT CDCl3
NS 4609
DS 2
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 0.6832628 sec
RG 9195.2
DW 20.850 usec
DE 6.50 usec
TE 300.2 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 100.6127750 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
139.61
139.54
128.49
127.34
127.27
127.25
109.54
109.46
108.74
96.21
77.32
77.00
76.68
70.76
70.63
70.56
70.41
70.39
67.33
67.27
66.75
66.68
65.37
65.32
64.88
64.83
53.22
53.17
53.12
45.23
29.61
25.86
24.88
24.86
24.37
O
P
OMe
NHBn
11
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


10.7524
10.5146
Current Data Parameters
NAME vd-690 P31
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140522
Time 14.41
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 24
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
======== 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 13.70 dB
PL13 16.70 dB
PL2W 16.12334061 W
PL12W 0.43396533 W
PL13W 0.21749784 W
SFO2 400.1320007 MHz
F2 - Processing parameters
SI 32768
SF 161.9755127 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
O
O
P
OMe
NHBn
O
O
O
O
O
11
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 ppm

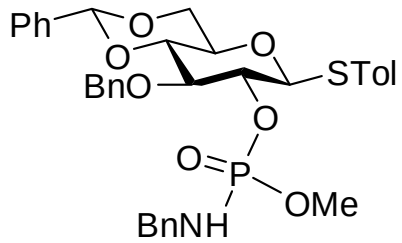


Current Data Parameters
NAME vd-717-1 All
EXPNO 1
PROCNO 1

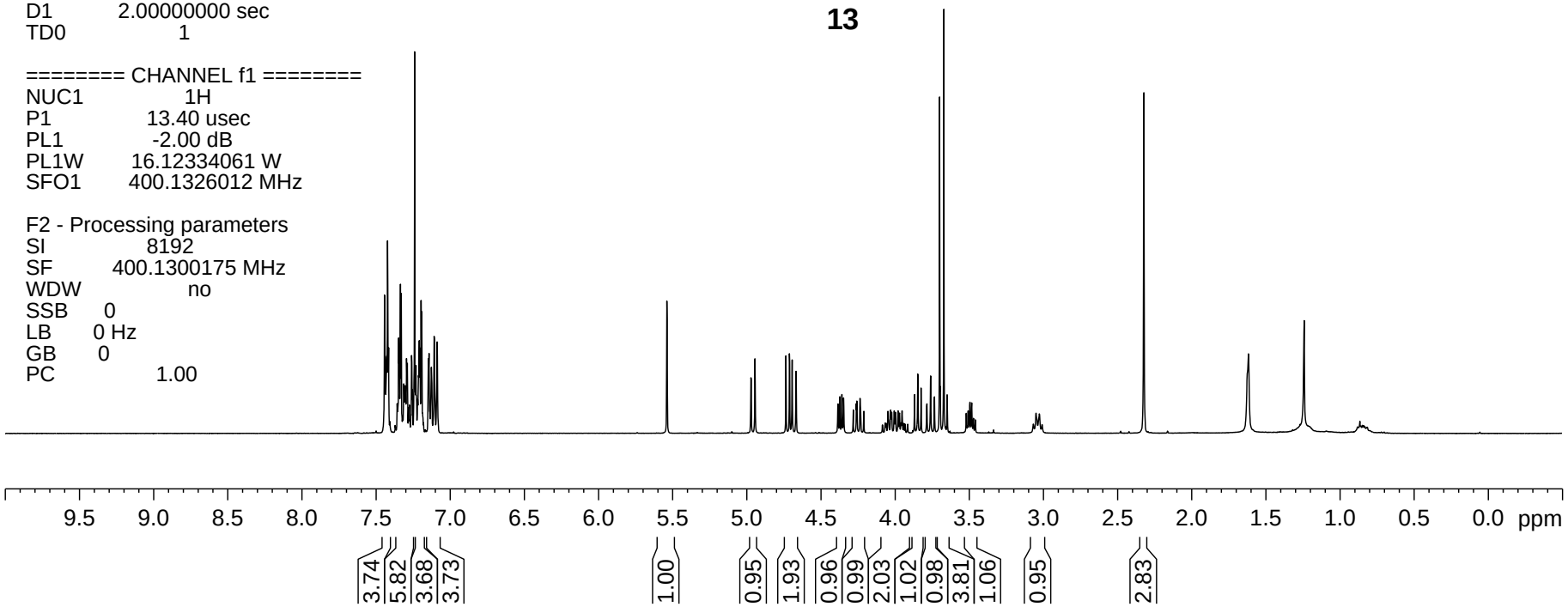
F2 - Acquisition Parameters
Date_ 20140811
Time 21.48
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 64
DS 0
SWH 5597.015 Hz
FIDRES 0.341615 Hz
AQ 1.4636873 sec
RG 161
DW 89.333 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

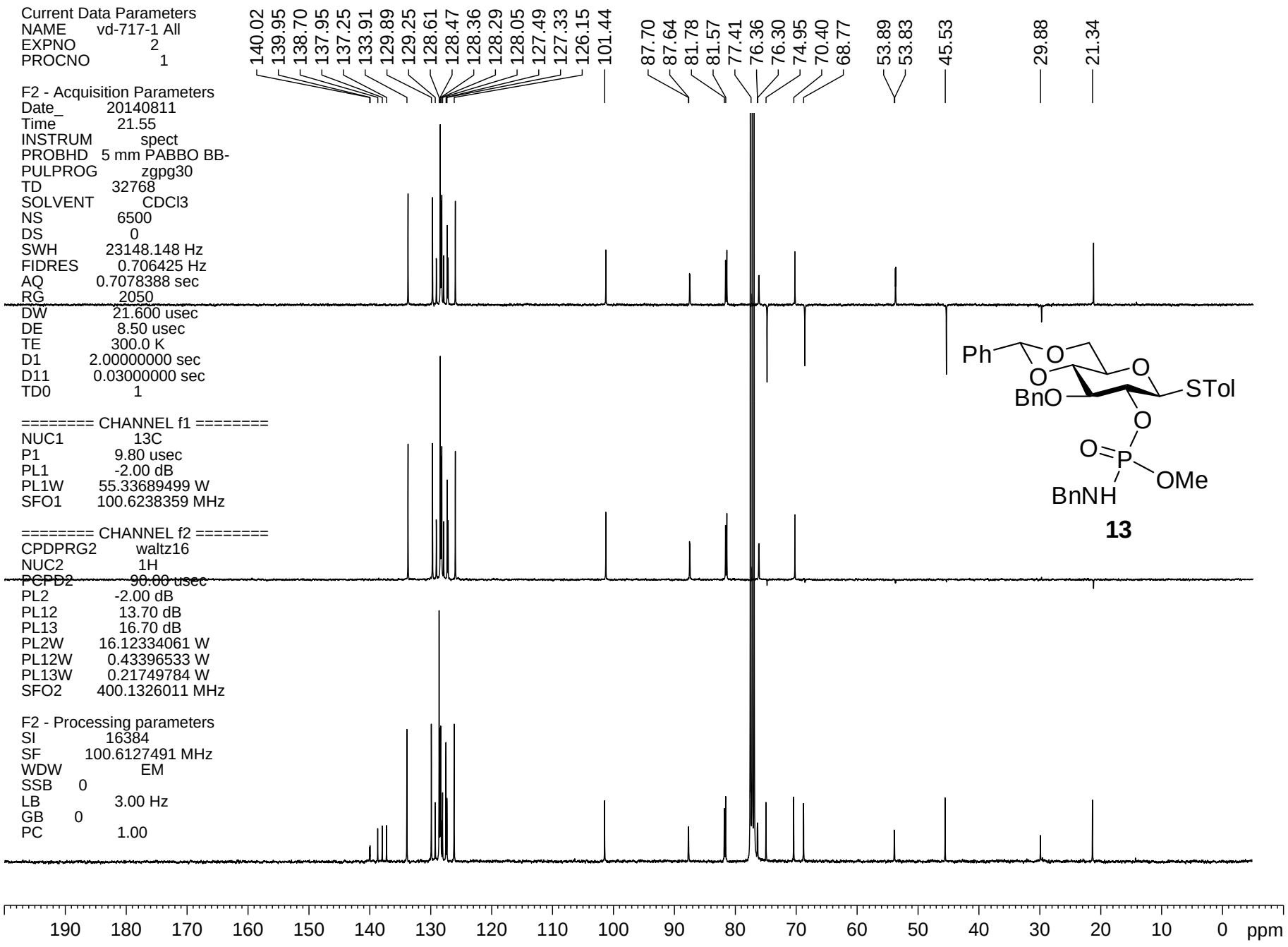
======== CHANNEL f1 ========
NUC1 1H
P1 13.40 usec
PL1 -2.00 dB
PL1W 16.12334061 W
SFO1 400.1326012 MHz

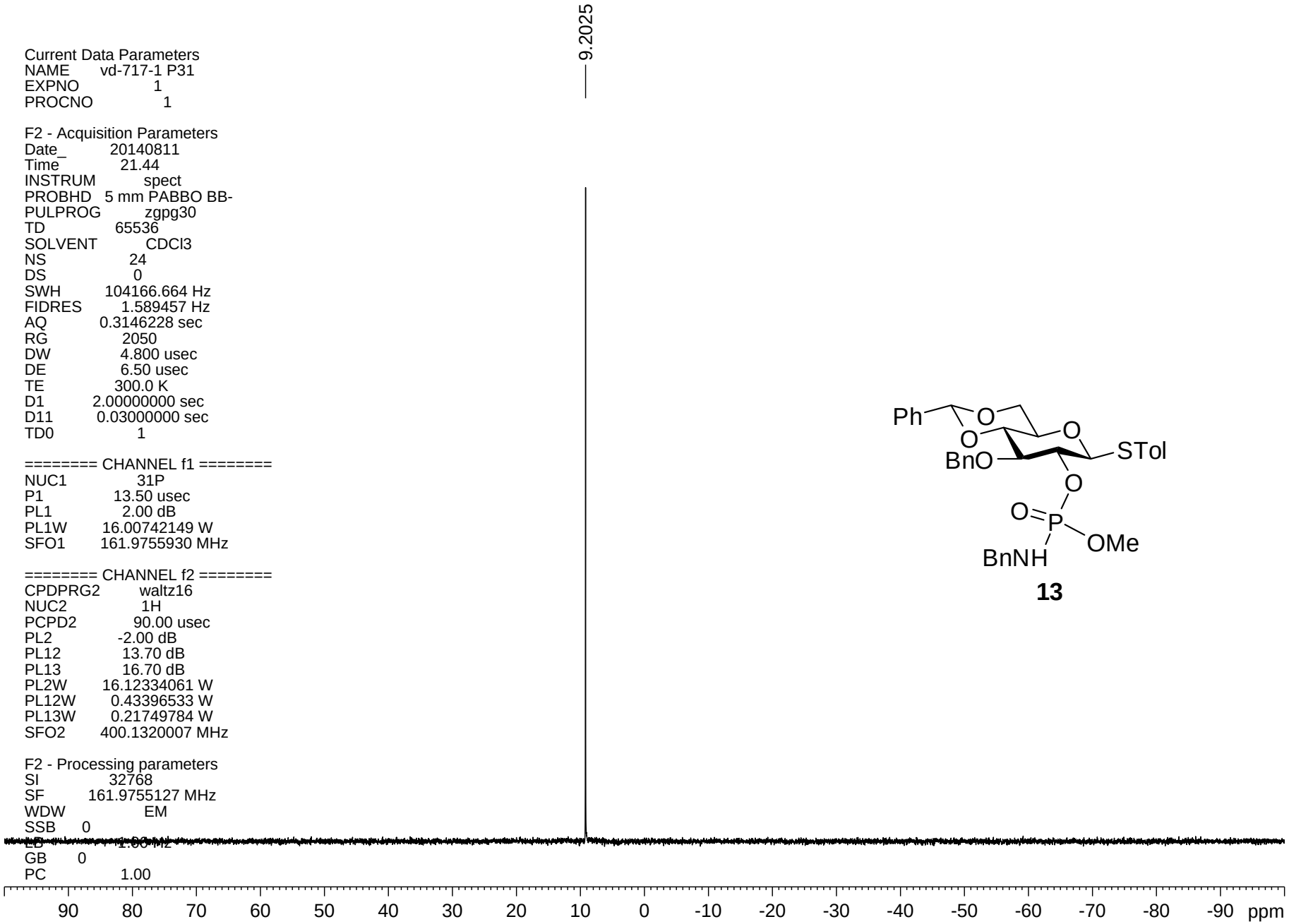
F2 - Processing parameters
SI 8192
SF 400.1300175 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

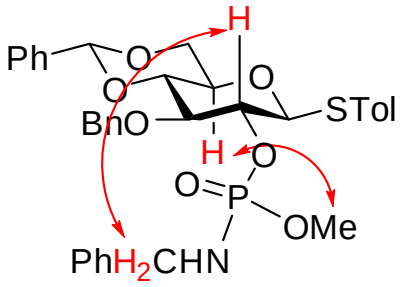


13

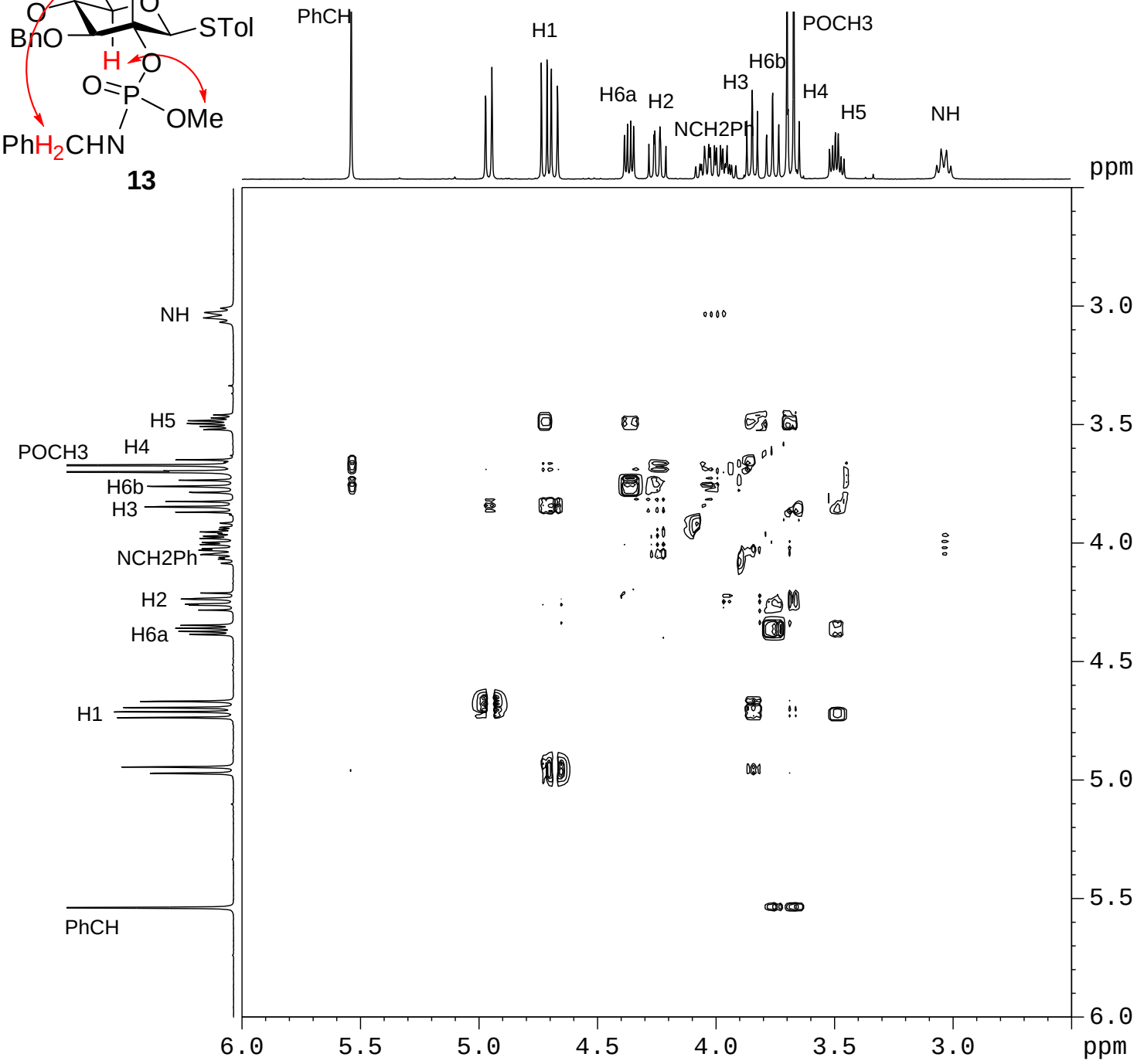



Current Data Parameters
NAME vd-717-1 All
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140811
Time 21.55
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 6500
DS 0
SWH 23148.148 Hz
FIDRES 0.706425 Hz
AQ 0.7078388 sec
RG 2050
DW 21.600 usec
DE 8.50 usec
TE 300.0 K
D1 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 100.6127491 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
140.02
139.95
138.70
137.95
137.25
133.91
129.89
129.25
128.61
128.47
128.36
128.29
128.05
127.49
127.33
126.15
101.44
87.70
87.64
81.78
81.57
77.41
76.36
76.30
74.95
70.40
68.77
53.89
53.83
45.53
29.88
21.34
Ph
O
O
O
BnO
STol
O
O
P
OMe
BnNH
13
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


9.2025
Current Data Parameters
NAME vd-717-1 P31
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140811
Time 21.44
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 24
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
======== 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 13.70 dB
PL13 16.70 dB
PL2W 16.12334061 W
PL12W 0.43396533 W
PL13W 0.21749784 W
SFO2 400.1320007 MHz
F2 - Processing parameters
SI 32768
SF 161.9755127 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
Ph
O
O
O
BnO
STol
O
O
P
OMe
BnNH
13
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 ppm



13

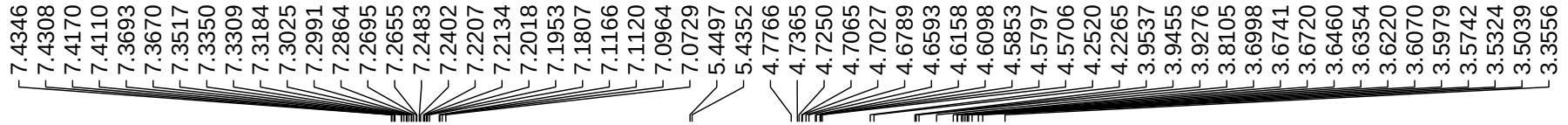


Current Data Parameters
NAME vd-717-1 All
EXPNO 7
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140812
Time 8.29
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG noesygpphpp
TD 2048
SOLVENT CDCl3
NS 4
DS 16
SWH 5197.505 Hz
FIDRES 2.537844 Hz
AQ 0.1970676 sec
RG 256
DW 96.200 usec
DE 6.50 usec
TE 300.0 K
D0 0.00007914 sec
D1 2.00000000 sec
D8 0.40000001 sec
D11 0.03000000 sec
D12 0.00002000 sec
D16 0.00020000 sec
IN0 0.00019240 sec

======== CHANNEL f1 ======
NUC1 1H
P1 13.40 usec
P2 26.80 usec
P17 2500.00 usec
PL1 -2.00 dB
PL10 3.30 dB
PL1W 16.12334061 W
PL10W 4.75833511 W
SFO1 400.1318006 MHz

====== GRADIENT CHANNEL =
GPNAM1 SMSQ10.100
GPNAM2 SMSQ10.100
GPZ1 40.00 %

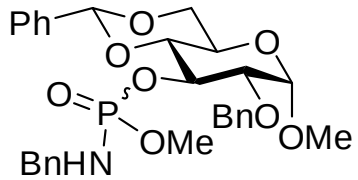


Current Data Parameters
NAME vd-718 all
EXPNO 1
PROCNO 1

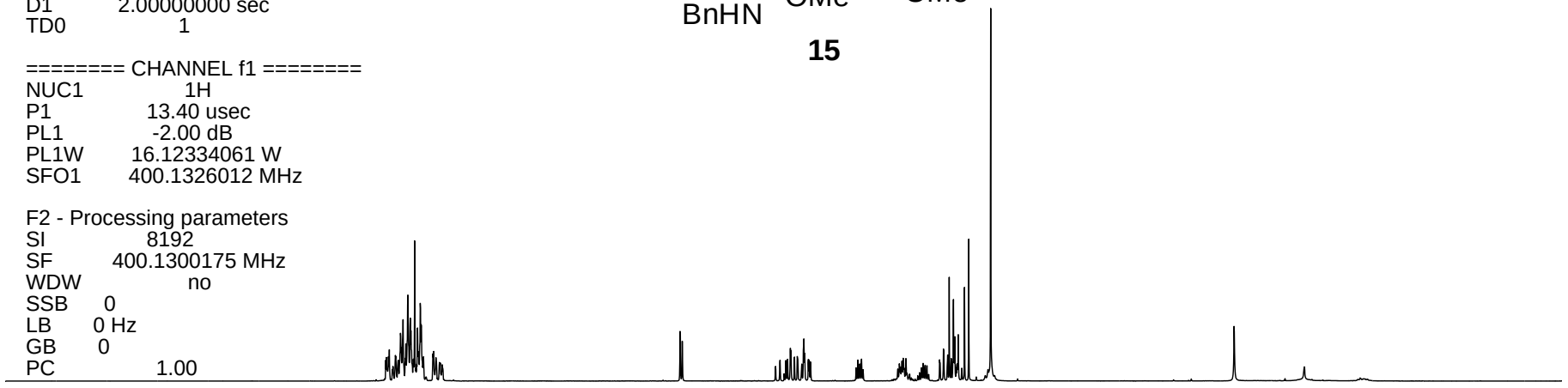
F2 - Acquisition Parameters
Date_ 20140815
Time 21.52
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 64
DS 0
SWH 5597.015 Hz
FIDRES 0.341615 Hz
AQ 1.4636873 sec
RG 144
DW 89.333 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

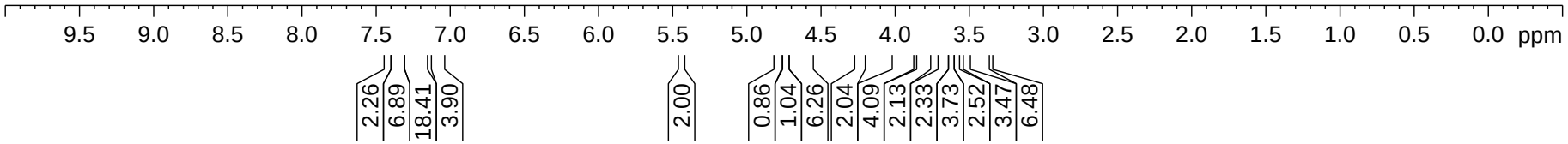
======== CHANNEL f1 ========
NUC1 1H
P1 13.40 usec
PL1 -2.00 dB
PL1W 16.12334061 W
SFO1 400.1326012 MHz

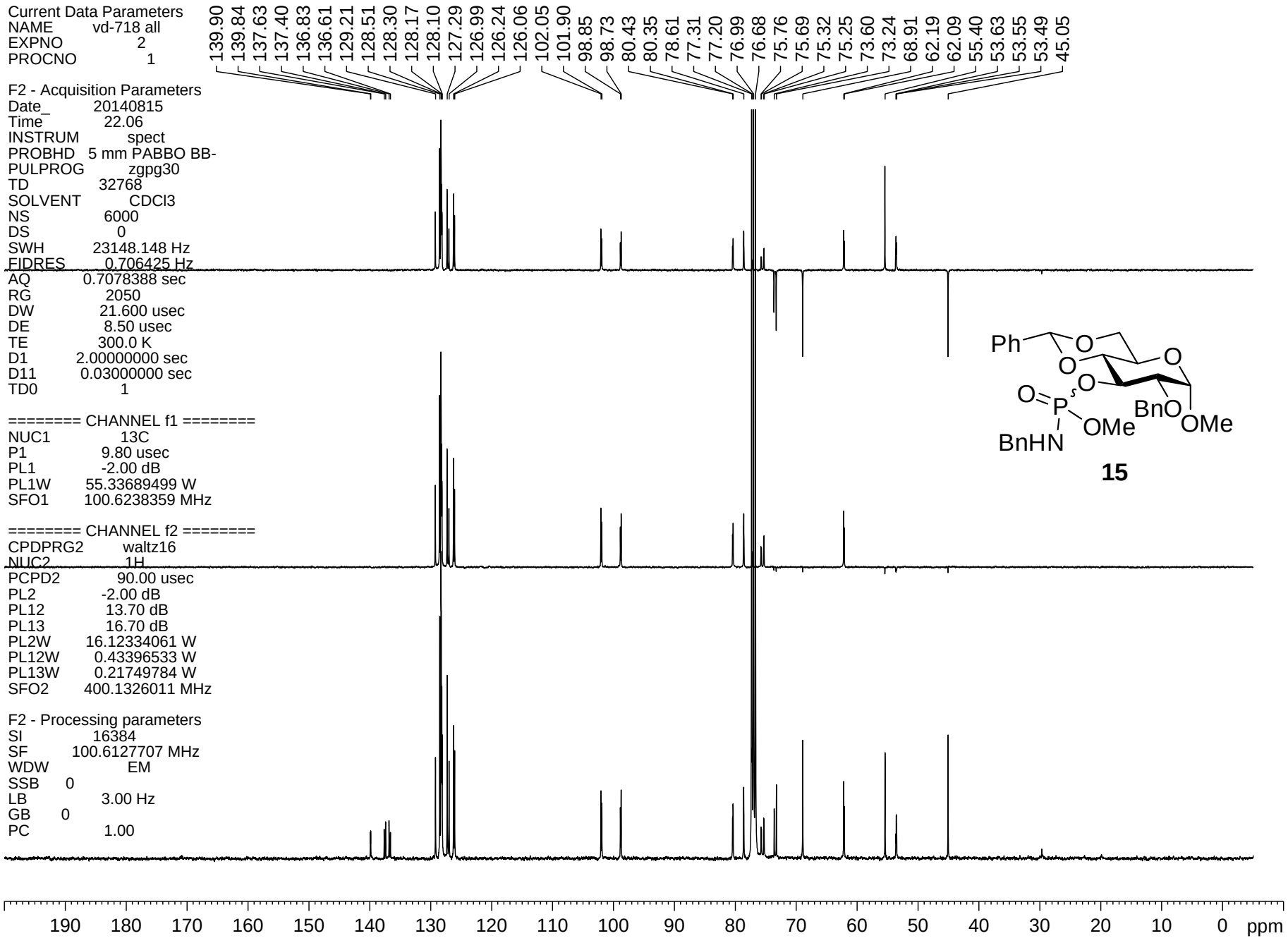
F2 - Processing parameters
SI 8192
SF 400.1300175 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00



15






Current Data Parameters
NAME vd-718 all
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140815
Time 22.06
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 6000
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 100.6127707 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
139.90
139.84
137.63
137.40
136.83
136.61
129.21
128.51
128.30
128.17
128.10
127.29
126.99
126.24
126.06
102.05
101.90
98.85
98.73
80.43
80.35
78.61
77.31
77.20
76.99
76.68
75.76
75.69
75.32
75.25
73.60
73.24
68.91
62.19
62.09
55.40
53.63
53.55
53.49
45.05
Ph
O
O
O
O
O
O
P
s
BnO
OMe
OMe
BnHN
15
190
180
170
160
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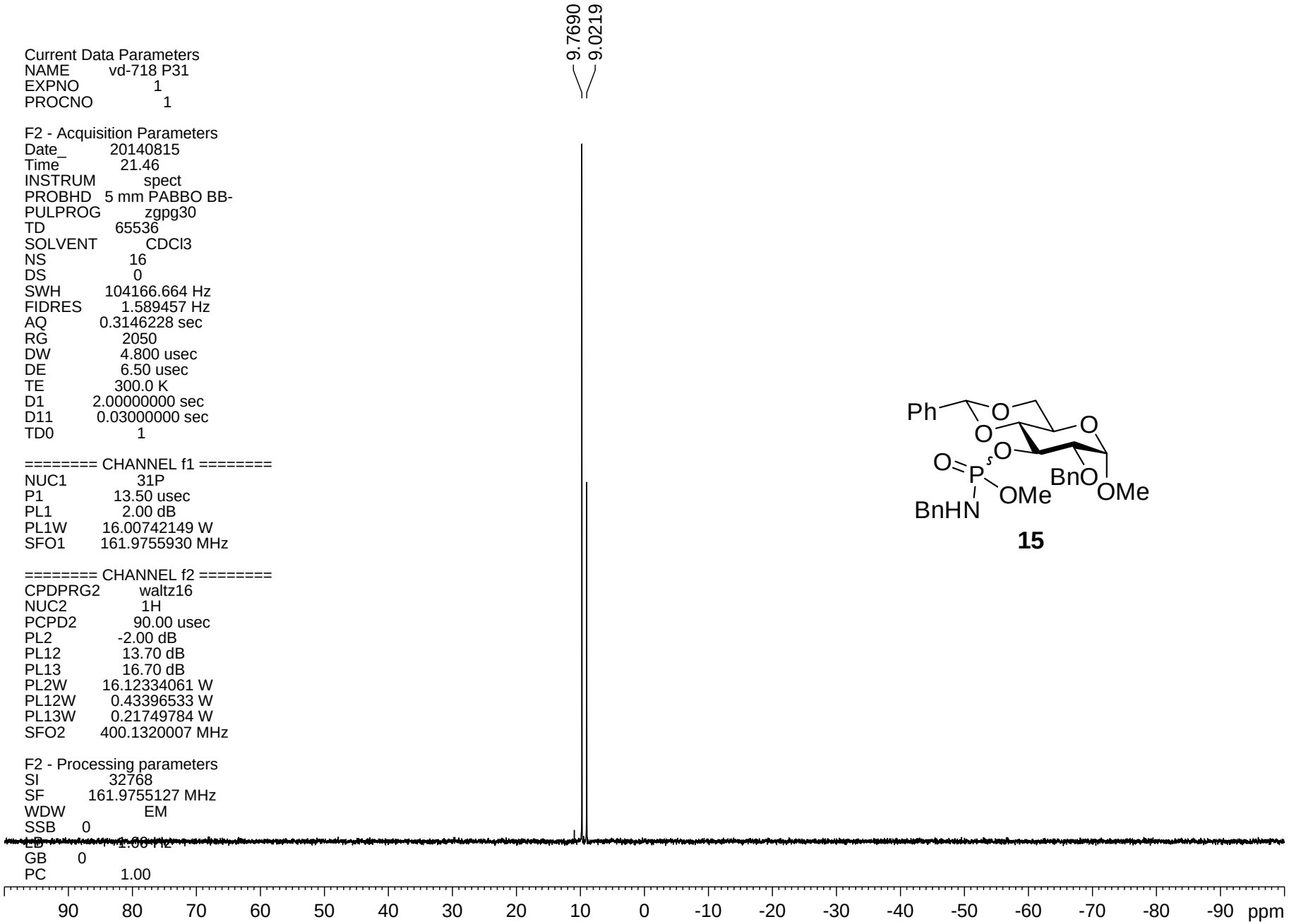
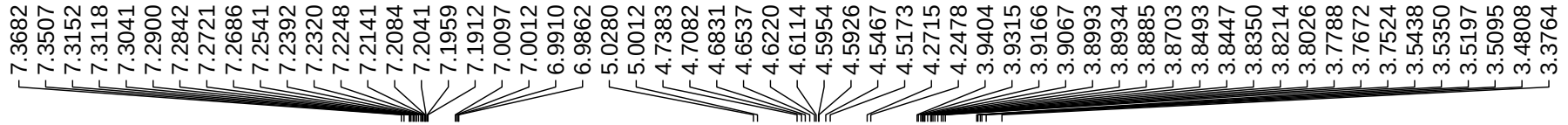
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


9.7690
9.0219
Current Data Parameters
NAME vd-718 P31
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140815
Time 21.46
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
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
======== 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 13.70 dB
PL13 16.70 dB
PL2W 16.12334061 W
PL12W 0.43396533 W
PL13W 0.21749784 W
SFO2 400.1320007 MHz
F2 - Processing parameters
SI 32768
SF 161.9755127 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
Ph
O
O
O
O
O
P
s
BnO
OMe
OMe
BnHN
15
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
ppm

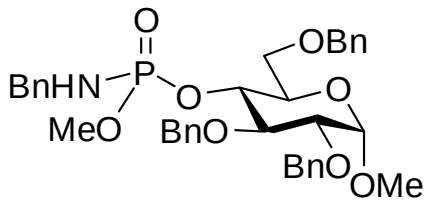


Current Data Parameters
NAME vd-719-1 AV400
EXPNO 1
PROCNO 1

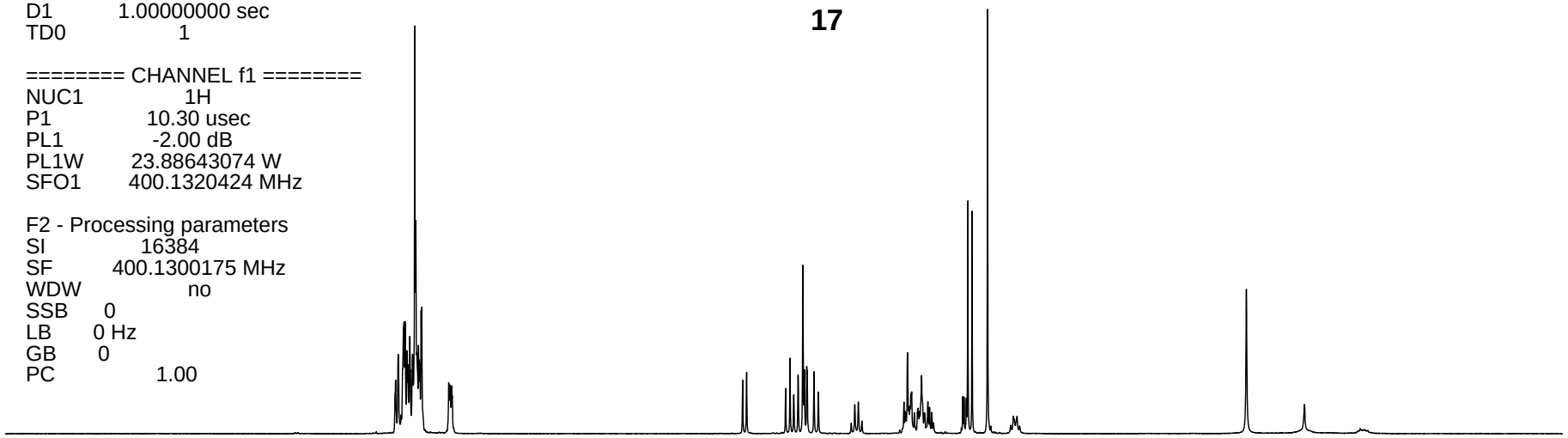
F2 - Acquisition Parameters
Date_ 20140815
Time 22.01
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 64
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7105396 sec
RG 228.1
DW 104.400 usec
DE 6.50 usec
TE 299.6 K
D1 1.00000000 sec
TD0 1

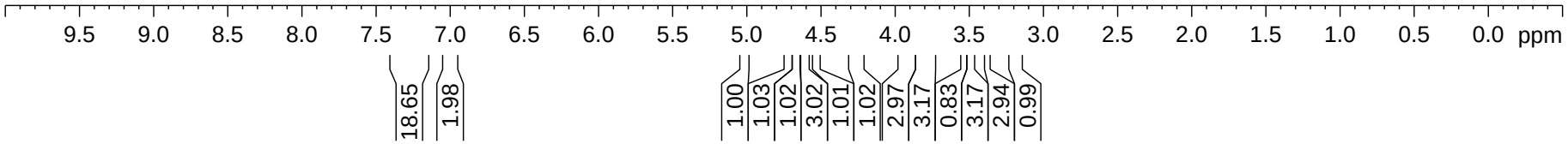
======== CHANNEL f1 ========
NUC1 1H
P1 10.30 usec
PL1 -2.00 dB
PL1W 23.88643074 W
SFO1 400.1320424 MHz

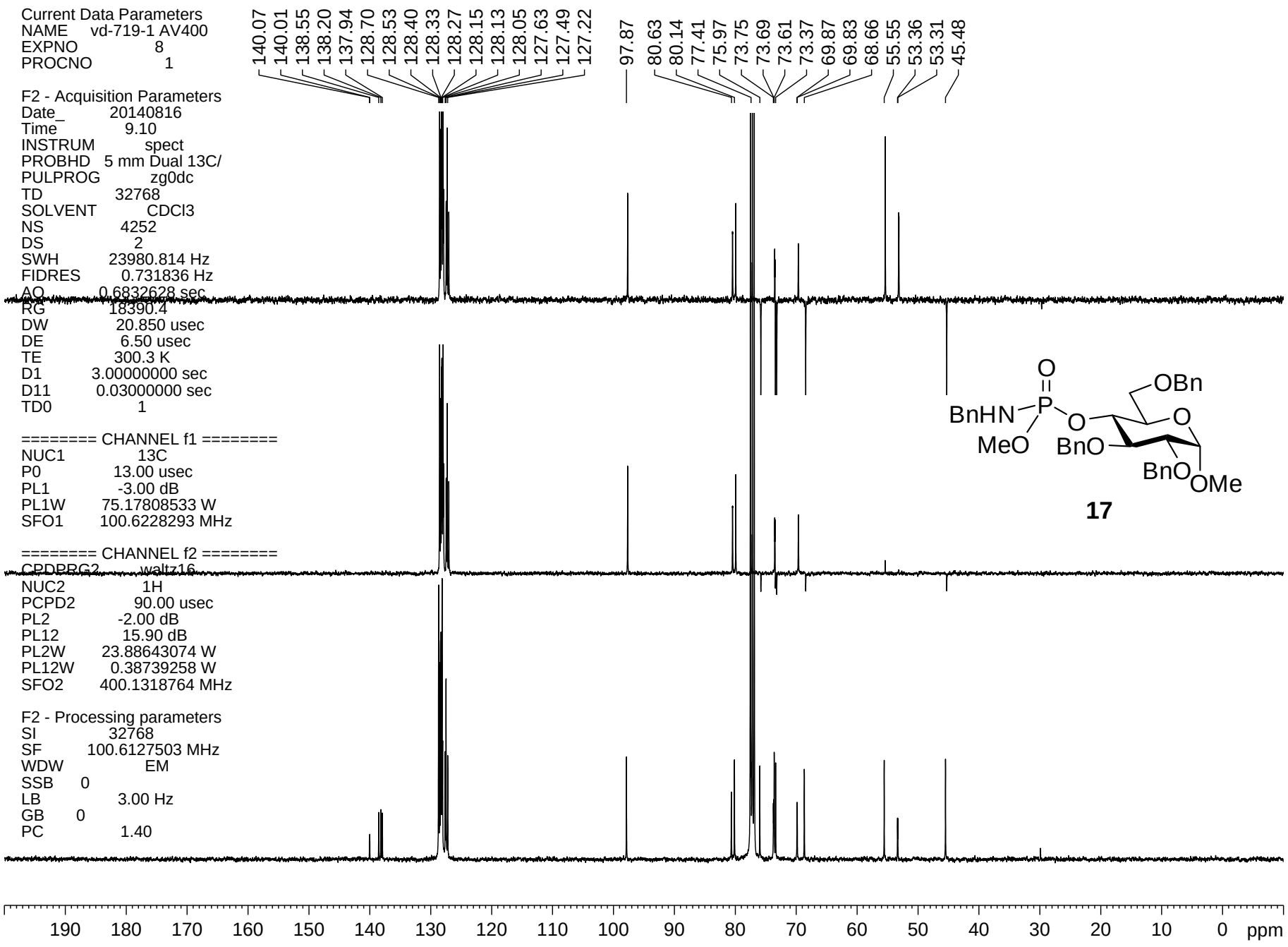
F2 - Processing parameters
SI 16384
SF 400.1300175 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

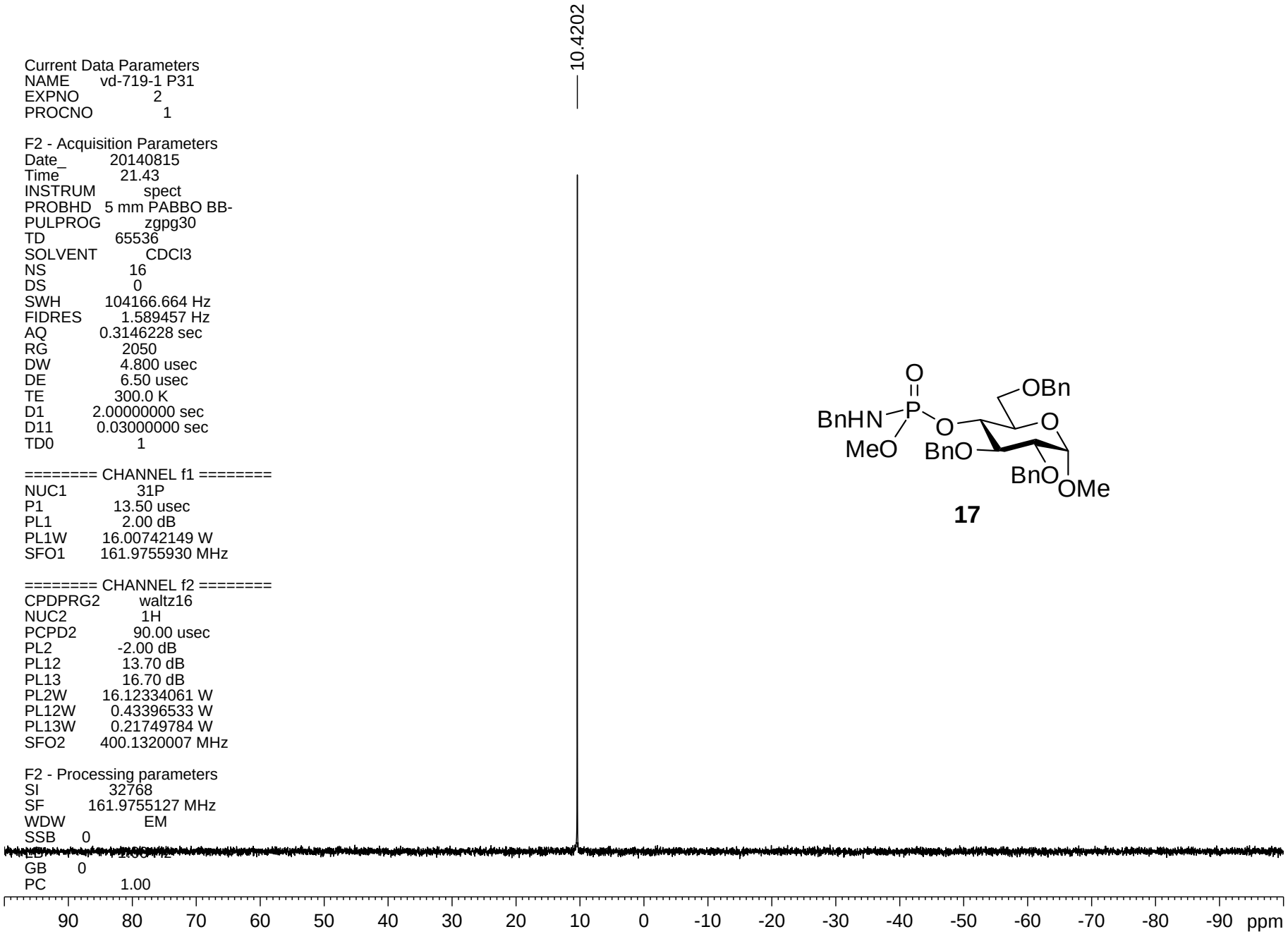


17



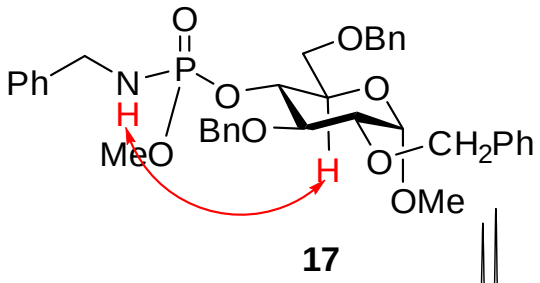



Current Data Parameters
NAME vd-719-1 AV400
EXPNO 8
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140816
Time 9.10
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg0dc
TD 32768
SOLVENT CDCl3
NS 4252
DS 2
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 0.6832628 sec
RG 18390.4
DW 20.850 usec
DE 6.50 usec
TE 300.3 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 100.6127503 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
140.07
140.01
138.55
138.20
137.94
128.70
128.53
128.40
128.33
128.27
128.15
128.13
128.05
127.63
127.49
127.22
97.87
80.63
80.14
77.41
75.97
73.75
73.69
73.61
73.37
69.87
69.83
68.66
55.55
53.36
53.31
45.48
O
P
BnHN
MeO
O
BnO
OBn
O
BnO
OMe
17
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

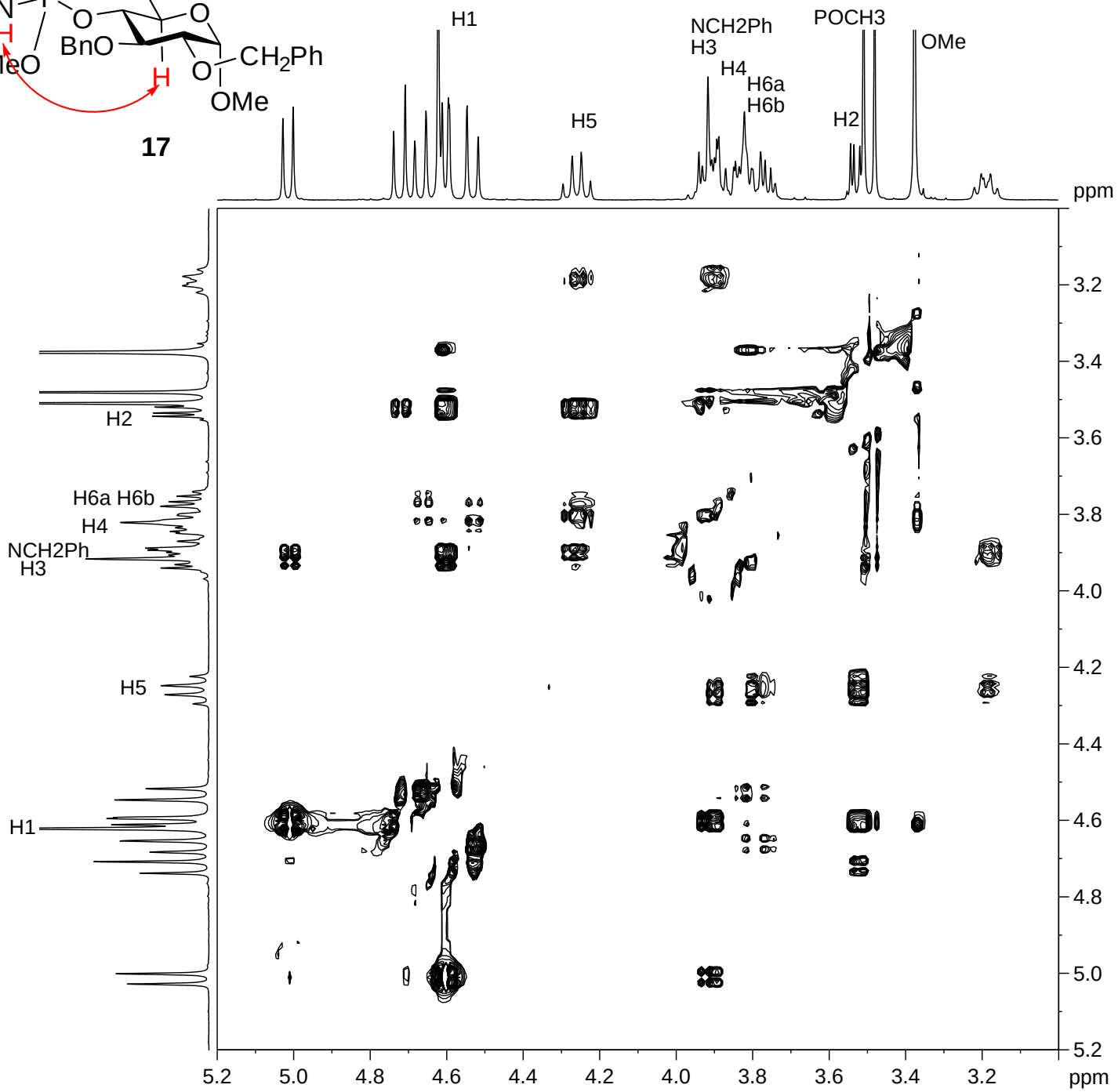

10.4202
Current Data Parameters
NAME vd-719-1 P31
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140815
Time 21.43
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 104166.664 Hz
FIDRES 1.589457 Hz
AQ 0.3146228 sec
RG 2050
DW 4.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== 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 13.70 dB
PL13 16.70 dB
PL2W 16.12334061 W
PL12W 0.43396533 W
PL13W 0.21749784 W
SFO2 400.1320007 MHz
F2 - Processing parameters
SI 32768
SF 161.9755127 MHz
WDW EM
SSB 0
GB 0
PC 1.00
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 ppm

O
OBn
BnHN
P
O
O
MeO
BnO
BnO
OMe

17



17



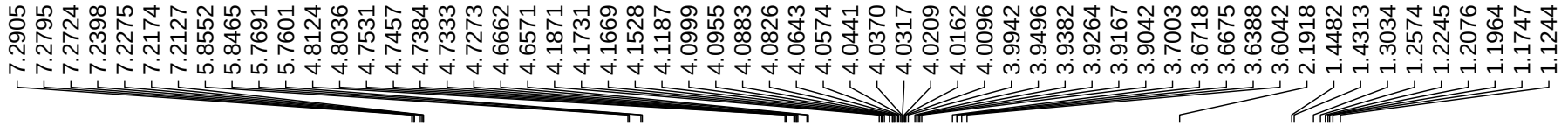
Current Data Parameters
NAME vd-719-1 AV400
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140815
Time 23.05
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG noesygpph
TD 2048
SOLVENT CDCl3
NS 4
DS 16
SWH 4084.967 Hz
FIDRES 1.994613 Hz
AQ 0.2507252 sec
RG 812.7
DW 122.400 usec
DE 6.50 usec
TE 299.8 K
D0 0.00010929 sec
D1 1.50000000 sec
D8 0.80000001 sec
D16 0.00020000 sec
IN0 0.00024480 sec

======== CHANNEL f1 ======
NUC1 1H
P1 10.30 usec
P2 20.60 usec
PL1 -2.00 dB
PL1W 23.88643074 W
SFO1 400.1318806 MHz

====== GRADIENT CHANNEL =
GPNAM1 SINE.100
GPZ1 40.00 %
P16 1000.00 usec

F1 - Acquisition parameters
TD 256
SFO1 400.1319 MHz
FIDRES 15.956903 Hz

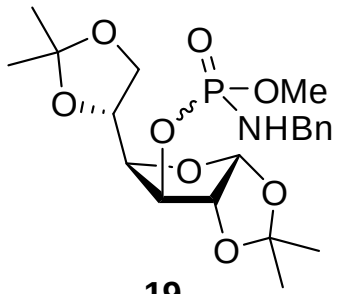


Current Data Parameters
NAME GPM-161 all
EXPNO 1
PROCNO 1

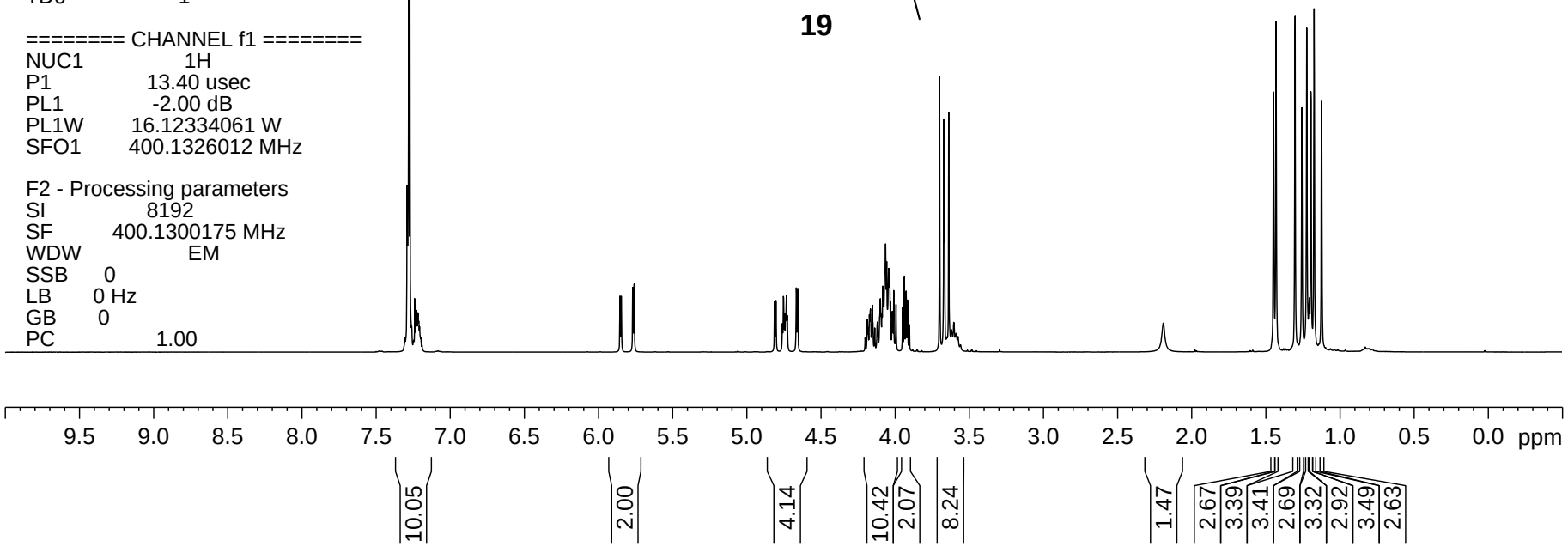
F2 - Acquisition Parameters
Date_ 20140814
Time 21.30
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 64
DS 0
SWH 5597.015 Hz
FIDRES 0.341615 Hz
AQ 1.4636873 sec
RG 45.2
DW 89.333 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

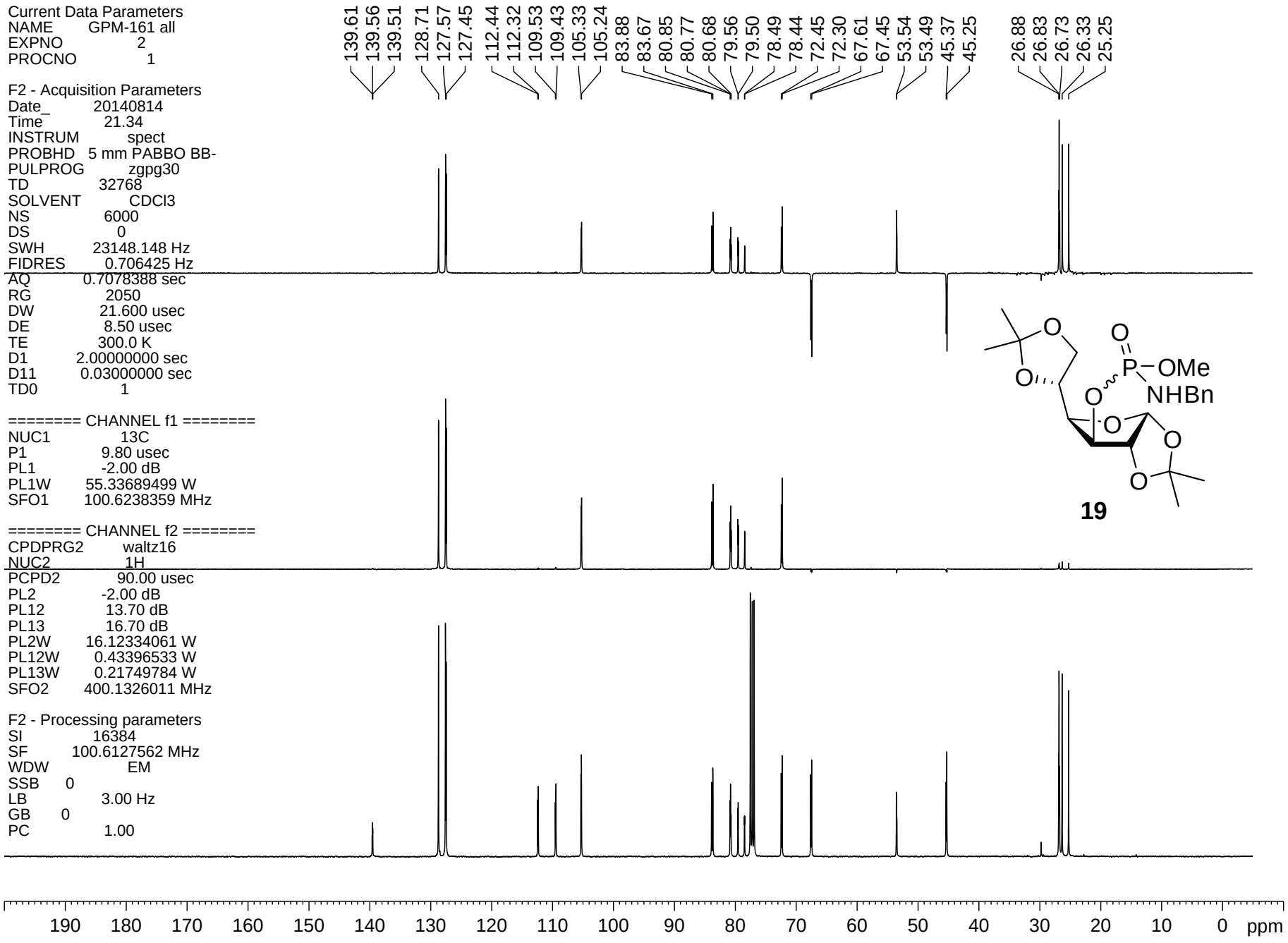
======== CHANNEL f1 ========
NUC1 1H
P1 13.40 usec
PL1 -2.00 dB
PL1W 16.12334061 W
SFO1 400.1326012 MHz

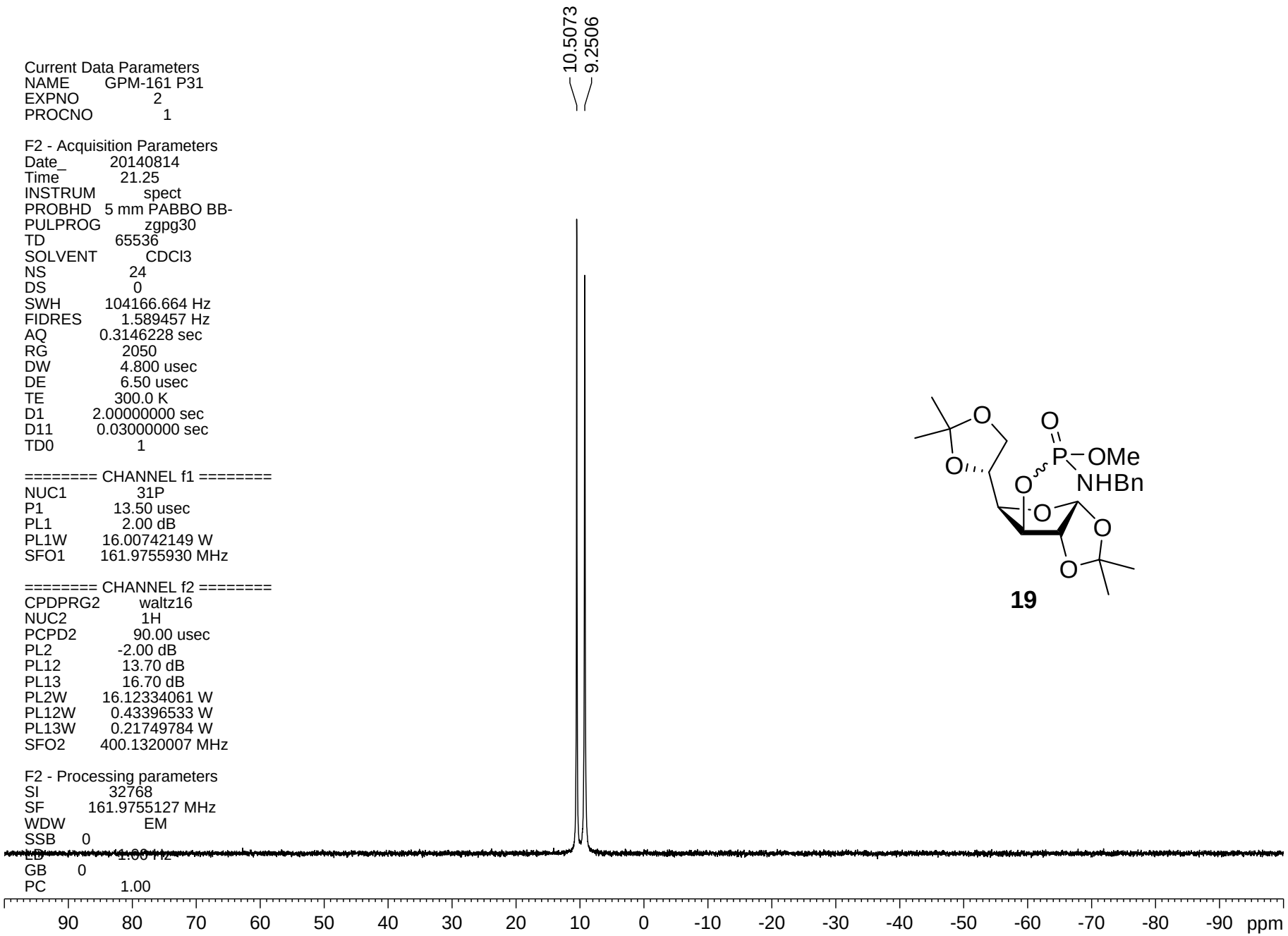
F2 - Processing parameters
SI 8192
SF 400.1300175 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00

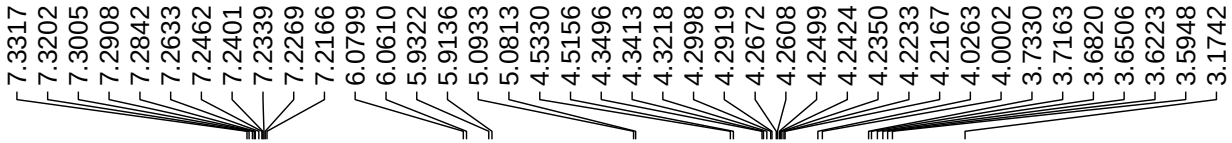


19




Current Data Parameters
NAME GPM-161 all
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140814
Time 21.34
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 6000
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 100.6127562 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
139.61
139.56
139.51
128.71
127.57
127.45
112.44
112.32
109.53
109.43
105.33
105.24
83.88
83.67
80.85
80.77
80.68
79.56
79.50
78.49
78.44
72.45
72.30
67.61
67.45
53.54
53.49
45.37
45.25
26.88
26.83
26.73
26.33
25.25
O
O
O
P−OMe
NHBn
O
O
O
O
19
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


10.5073
9.2506
Current Data Parameters
NAME GPM-161 P31
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140814
Time 21.25
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 24
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
======== 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 13.70 dB
PL13 16.70 dB
PL2W 16.12334061 W
PL12W 0.43396533 W
PL13W 0.21749784 W
SFO2 400.1320007 MHz
F2 - Processing parameters
SI 32768
SF 161.9755127 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
O
O
P
OMe
NHBn
O
O
O
O
O
19
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 ppm

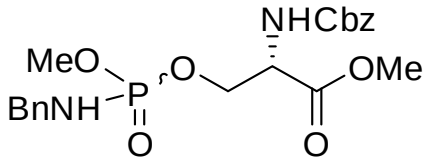


Current Data Parameters
NAME GPM_169-all
EXPNO 1
PROCNO 1

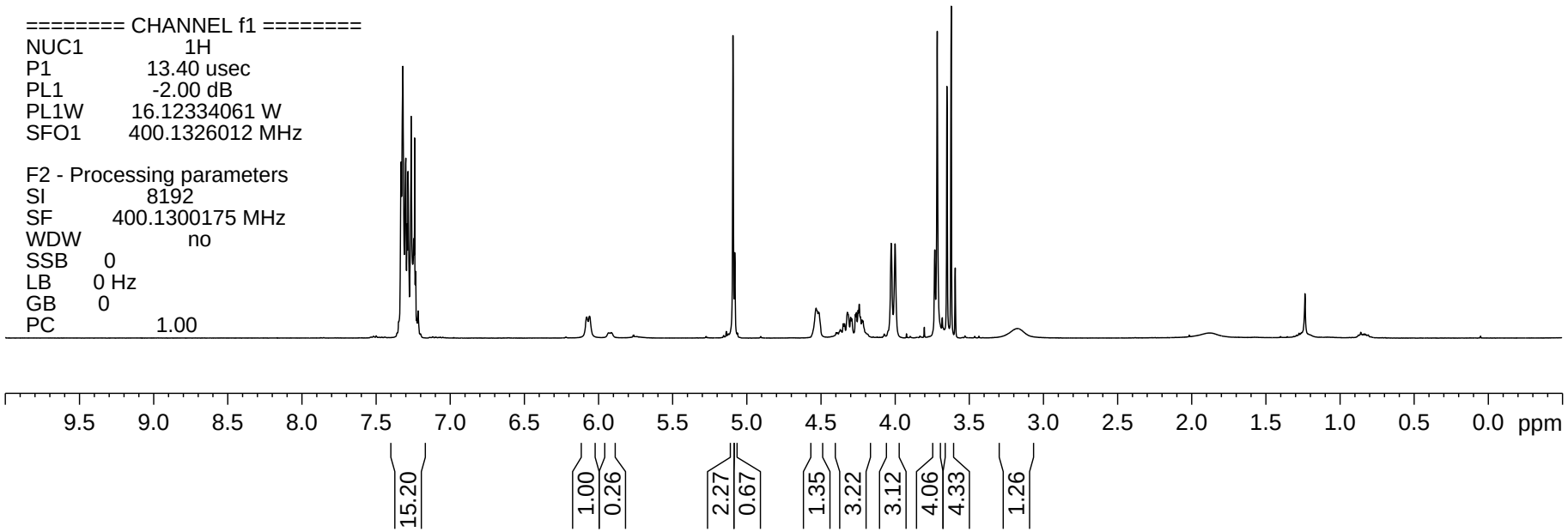
F2 - Acquisition Parameters
Date_ 20140824
Time 19.29
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 5597.015 Hz
FIDRES 0.341615 Hz
AQ 1.4636873 sec
RG 114
DW 89.333 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

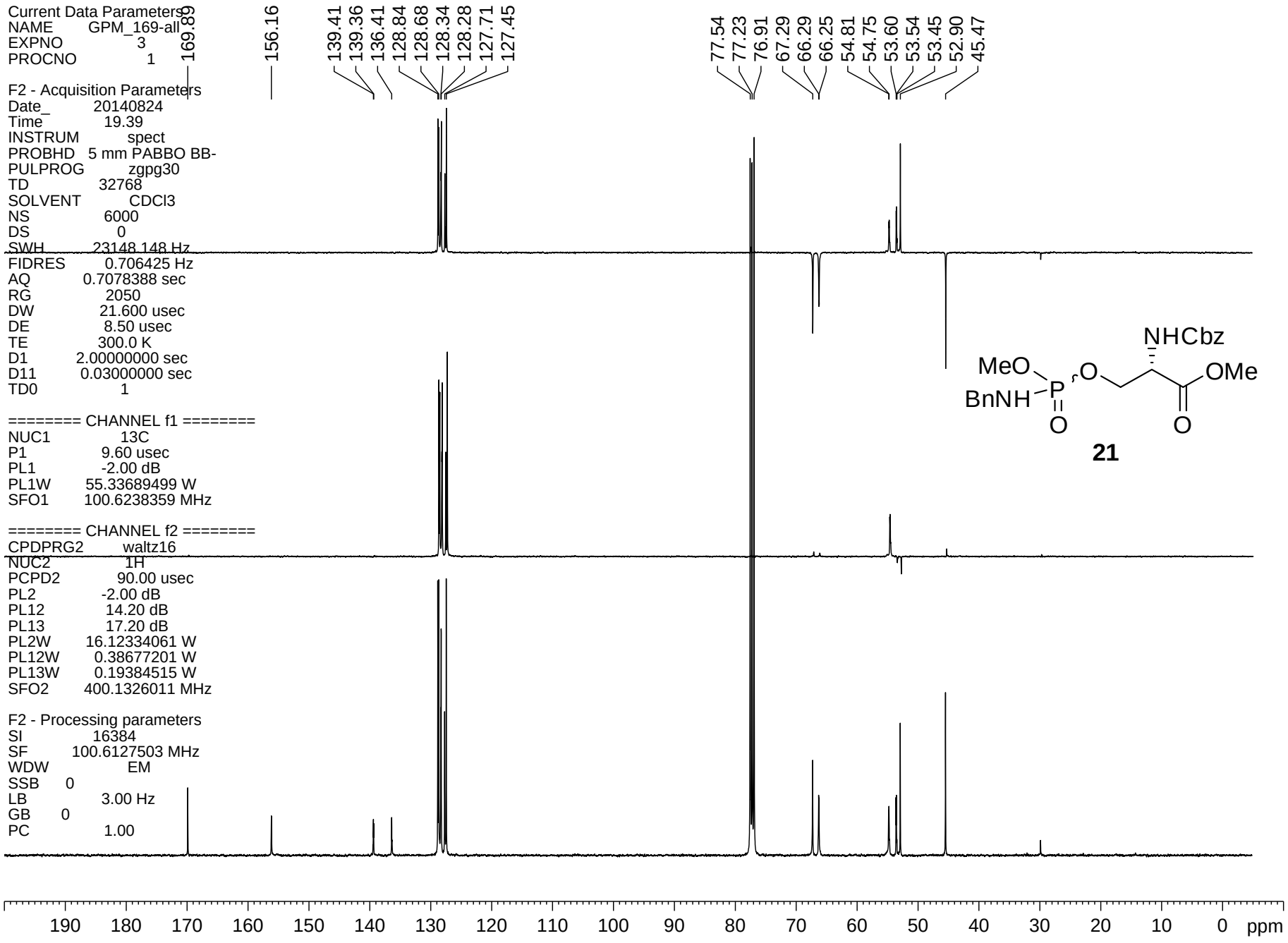
======== CHANNEL f1 ========
NUC1 1H
P1 13.40 usec
PL1 -2.00 dB
PL1W 16.12334061 W
SFO1 400.1326012 MHz

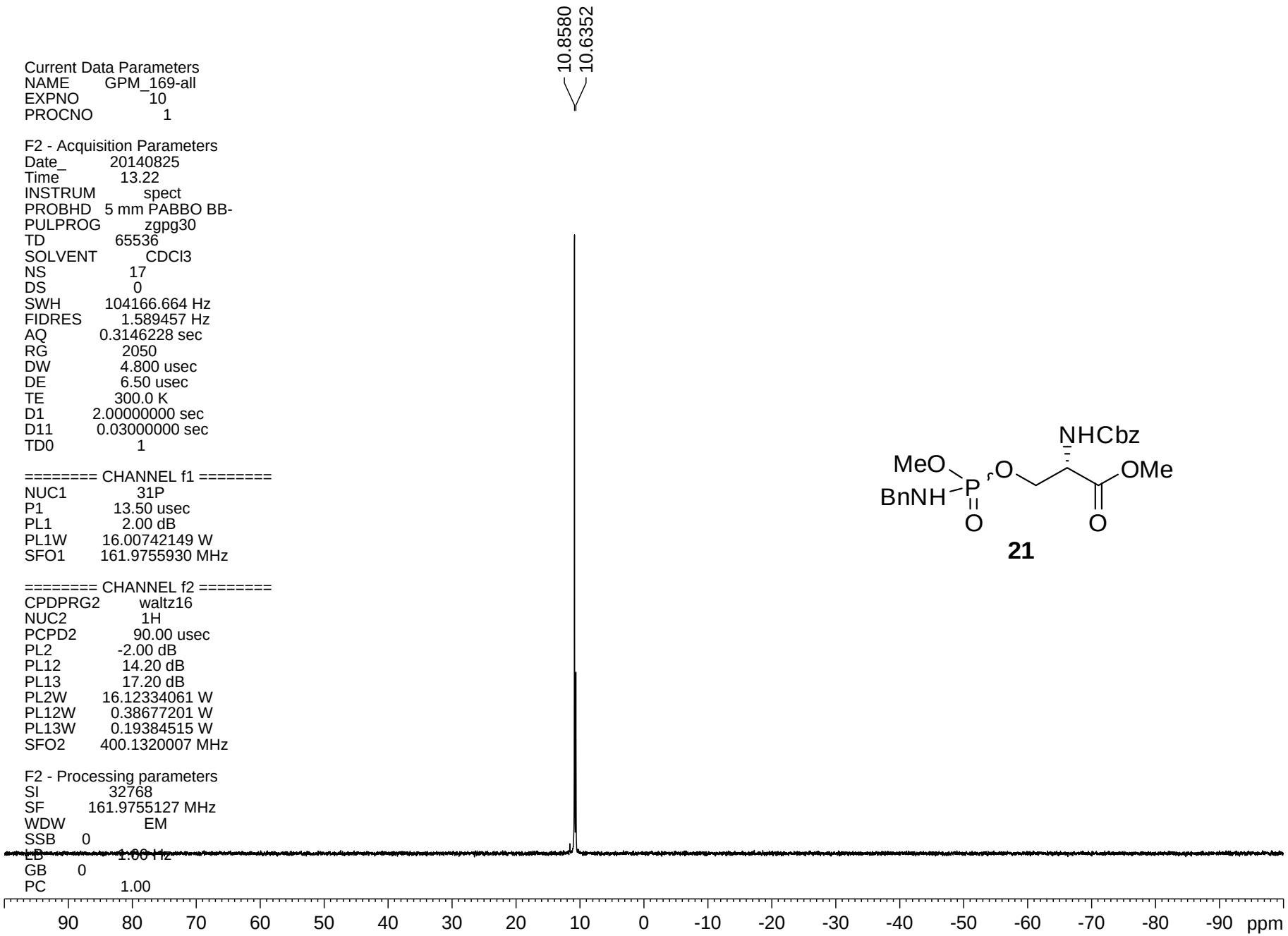
F2 - Processing parameters
SI 8192
SF 400.1300175 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

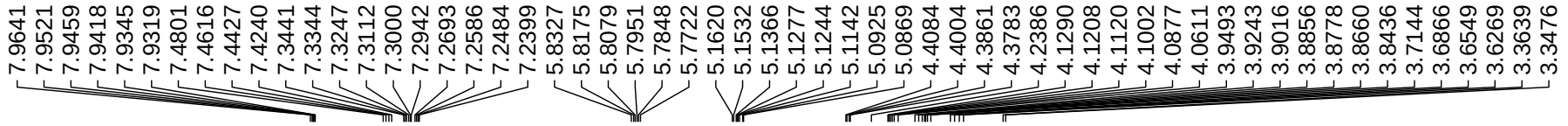


21




Current Data Parameters
NAME GPM_169-all
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140824
Time 19.39
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 6000
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 9.60 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 14.20 dB
PL13 17.20 dB
PL2W 16.12334061 W
PL12W 0.38677201 W
PL13W 0.19384515 W
SFO2 400.1326011 MHz
F2 - Processing parameters
SI 16384
SF 100.6127503 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
169.89
156.16
139.41
139.36
136.41
128.84
128.68
128.34
128.28
127.71
127.45
77.54
77.23
76.91
67.29
66.29
66.25
54.81
54.75
53.60
53.54
53.45
52.90
45.47
NHCbz
MeO
BnNH
P
O
O
O
OMe
O
21
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


10.8580
10.6352
Current Data Parameters
NAME GPM_169-all
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140825
Time 13.22
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 17
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
======== 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
F2 - Processing parameters
SI 32768
SF 161.9755127 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
NHCbz
MeO
BnNH
P
O
O
O
OMe
21
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 ppm

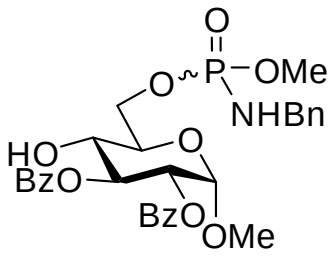


Current Data Parameters
NAME GPM-177-all
EXPNO 1
PROCNO 1

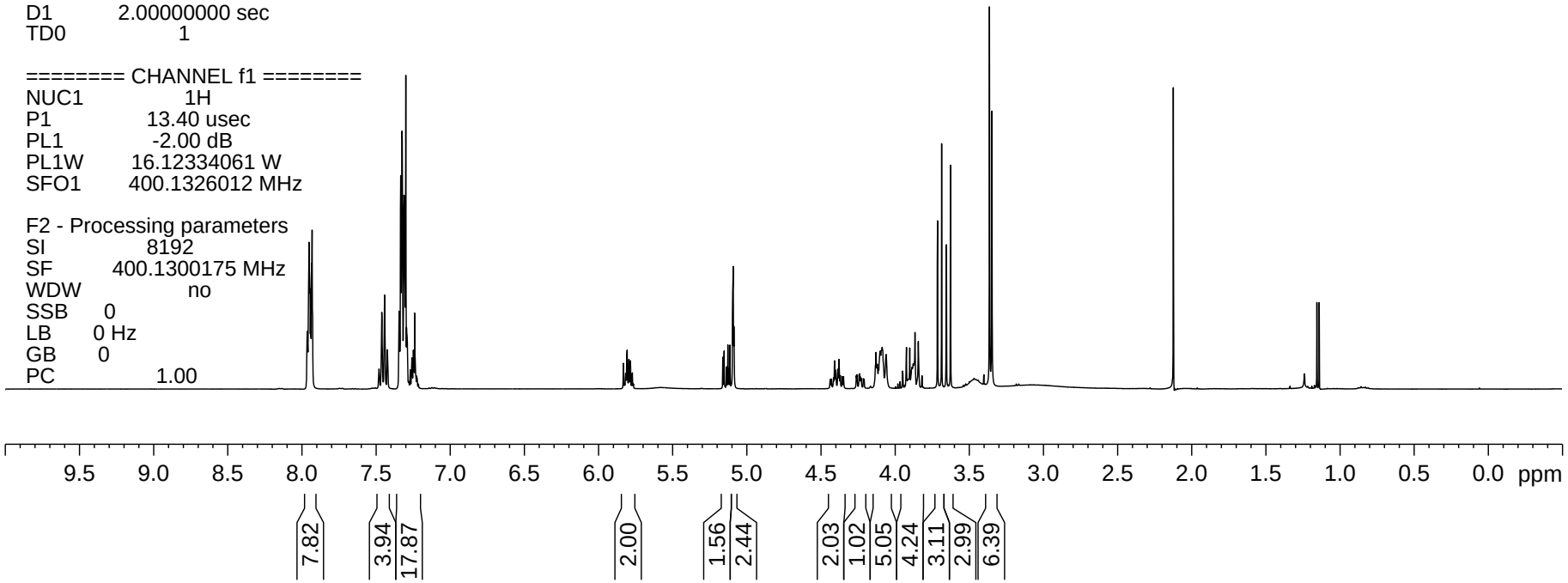
F2 - Acquisition Parameters
Date_ 20140907
Time 23.46
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 5597.015 Hz
FIDRES 0.341615 Hz
AQ 1.4636873 sec
RG 71.8
DW 89.333 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

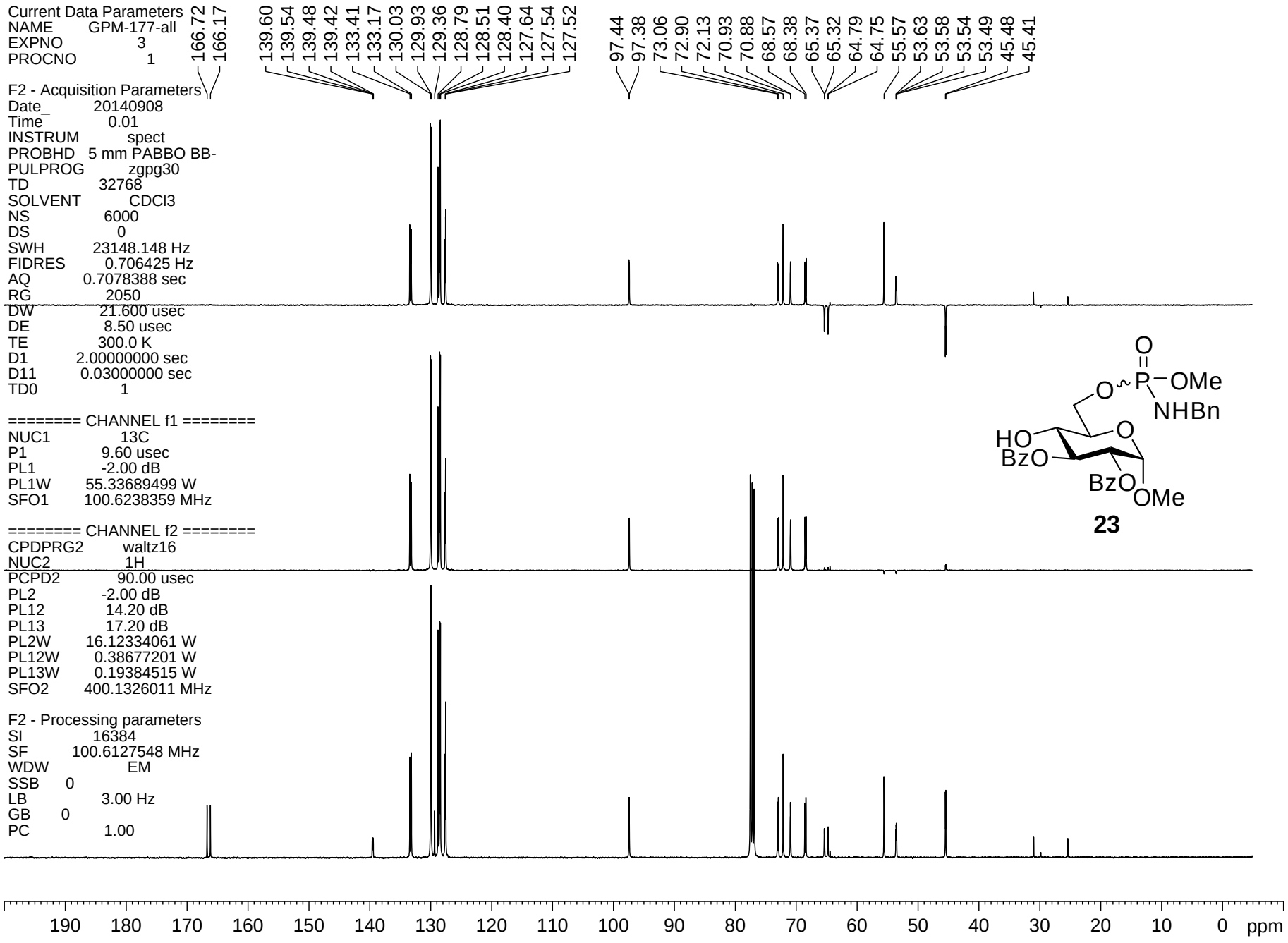
======== CHANNEL f1 ========
NUC1 1H
P1 13.40 usec
PL1 -2.00 dB
PL1W 16.12334061 W
SFO1 400.1326012 MHz

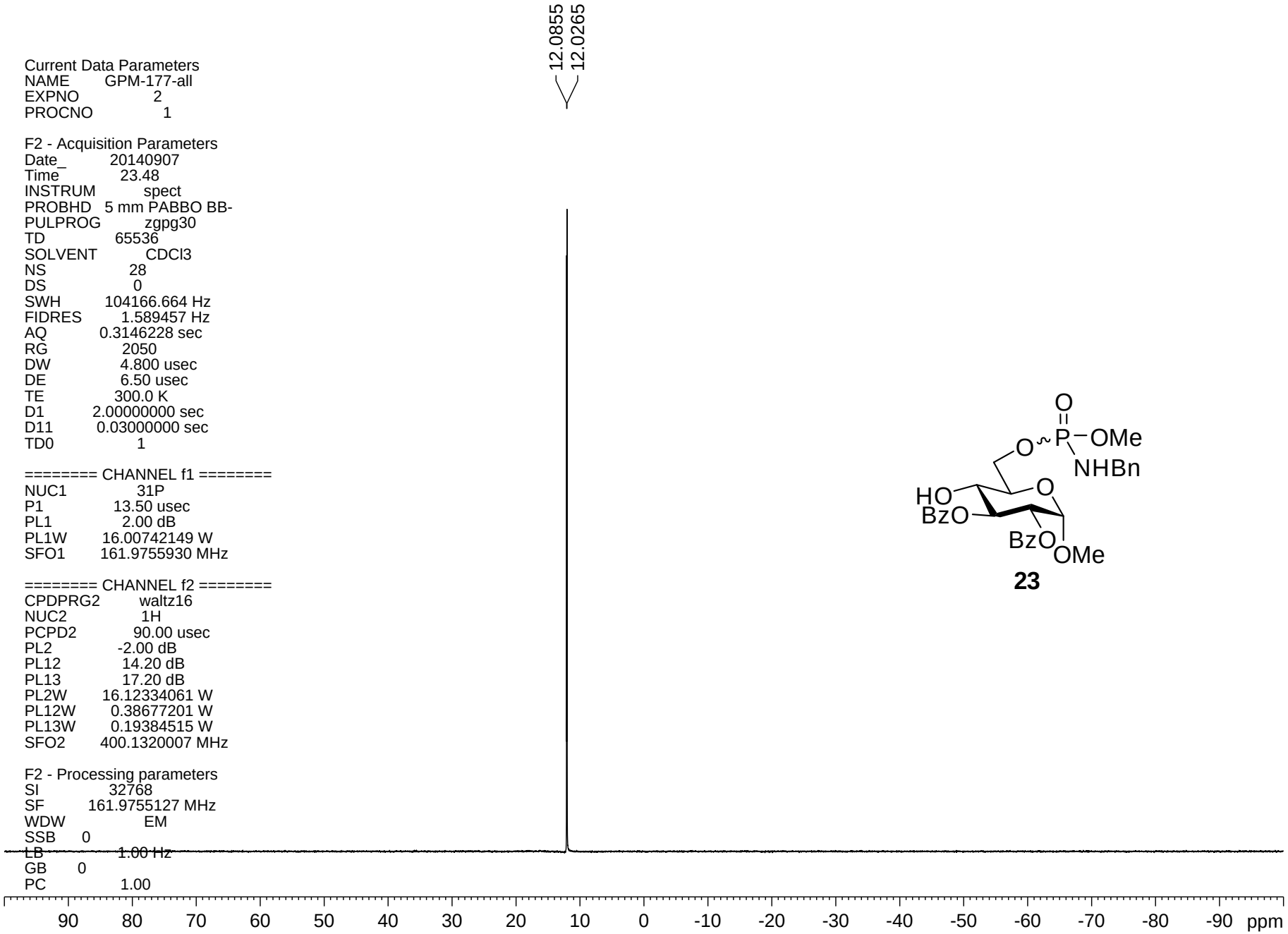
F2 - Processing parameters
SI 8192
SF 400.1300175 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

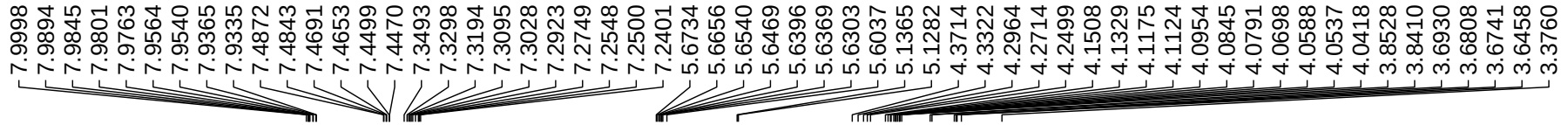


23




Current Data Parameters
NAME GPM-177-all
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140908
Time 0.01
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 6000
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 9.60 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 14.20 dB
PL13 17.20 dB
PL2W 16.12334061 W
PL12W 0.38677201 W
PL13W 0.19384515 W
SFO2 400.1326011 MHz
F2 - Processing parameters
SI 16384
SF 100.6127548 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
166.72
166.17
139.60
139.54
139.48
139.42
133.41
133.17
130.03
129.93
129.36
128.79
128.51
128.40
127.64
127.54
127.52
97.44
97.38
73.06
72.90
72.13
70.93
70.88
68.57
68.38
65.37
65.32
64.79
64.75
55.57
53.63
53.58
53.54
53.49
45.48
45.41
O
O
P
OMe
NHBn
HO
BzO
O
BzO
OMe
23
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


12.0855
12.0265
Current Data Parameters
NAME GPM-177-all
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140907
Time 23.48
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 28
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
======== 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
F2 - Processing parameters
SI 32768
SF 161.9755127 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
O
O
P
OMe
NHBn
HO
O
BzO
BzO
OMe
23
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 ppm



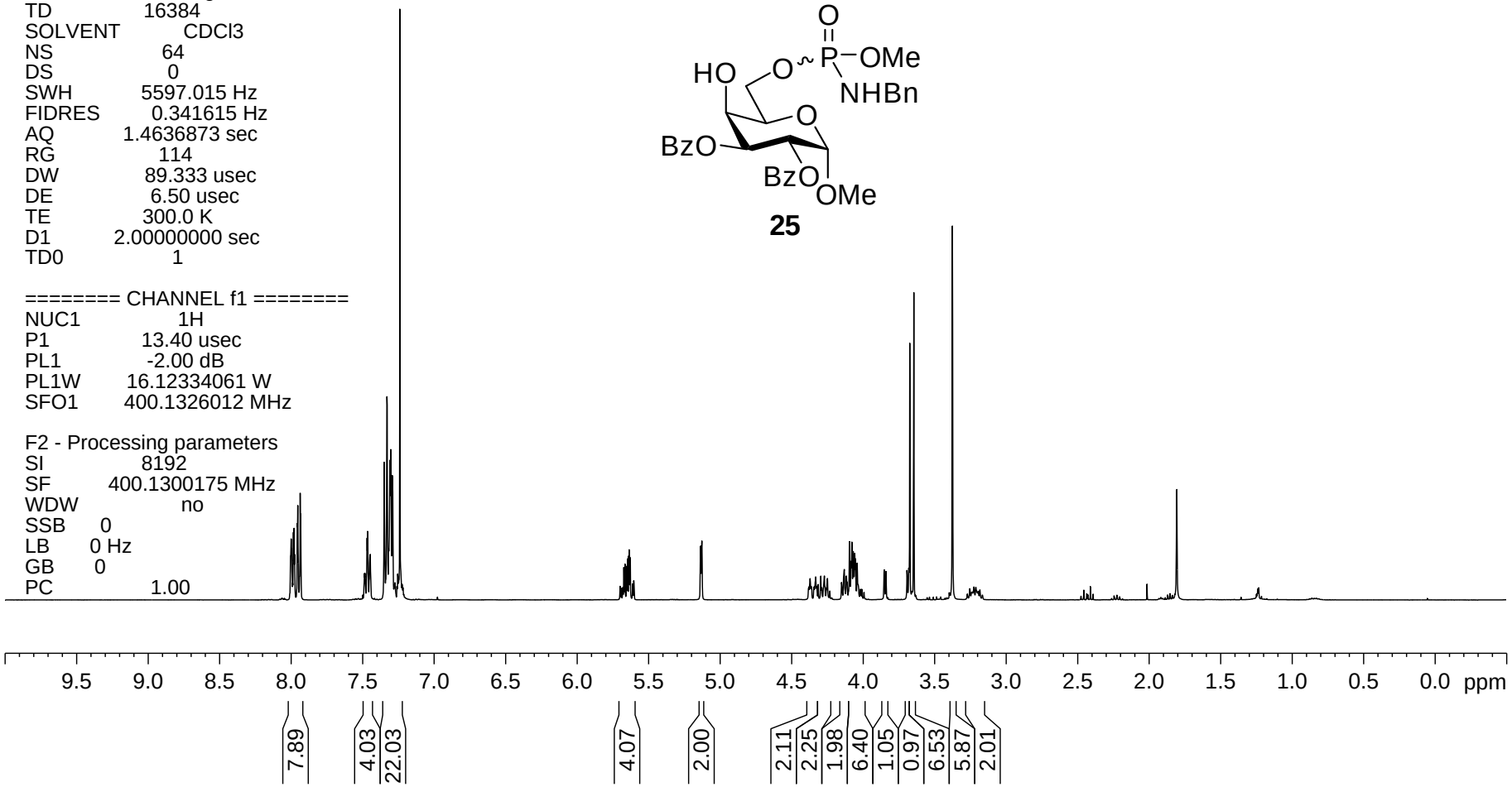
Current Data Parameters
NAME vd-728 all
EXPNO 1
PROCNO 1

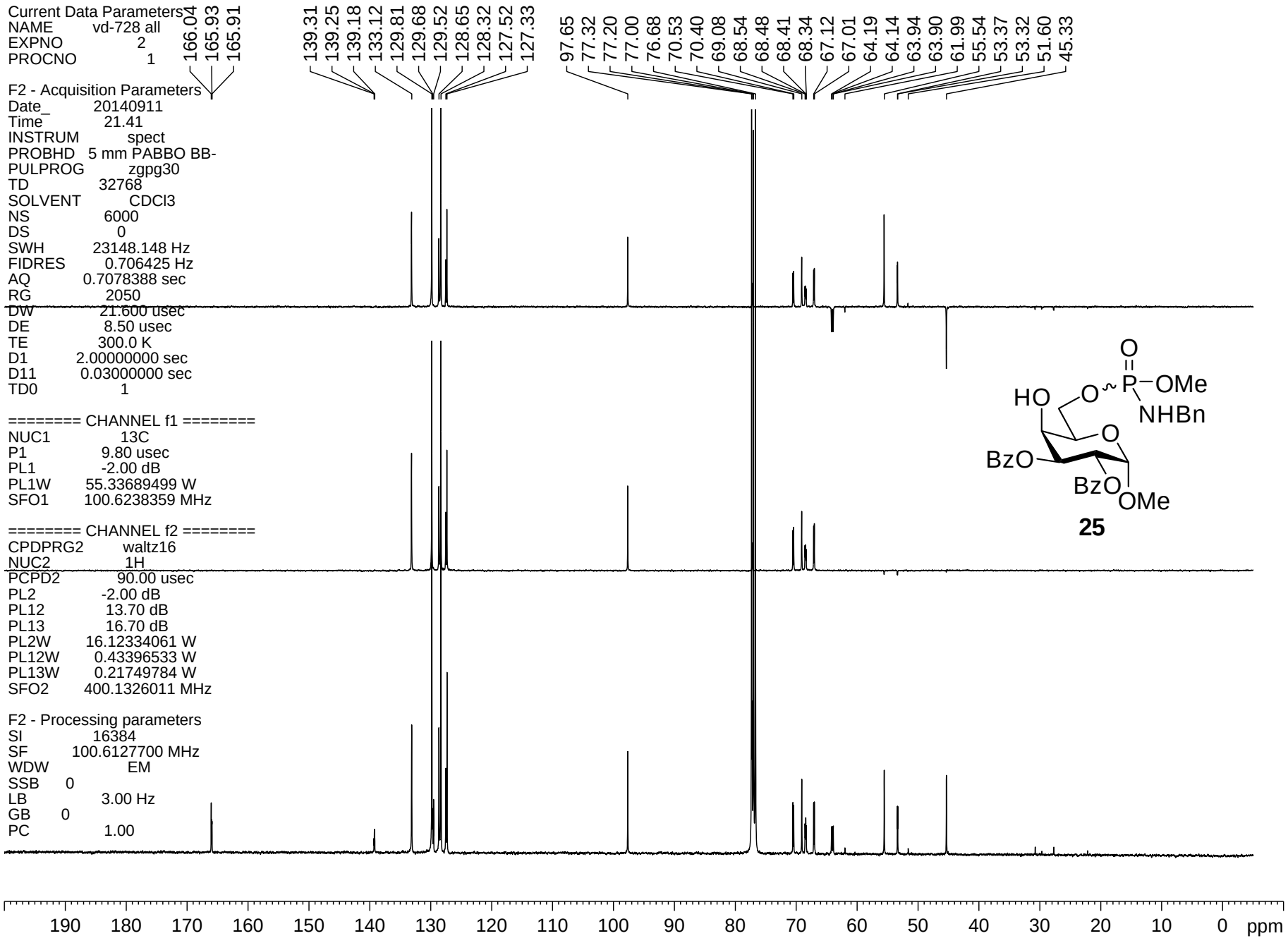
F2 - Acquisition Parameters
Date_ 20140911
Time 21.36
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 64
DS 0
SWH 5597.015 Hz
FIDRES 0.341615 Hz
AQ 1.4636873 sec
RG 114
DW 89.333 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

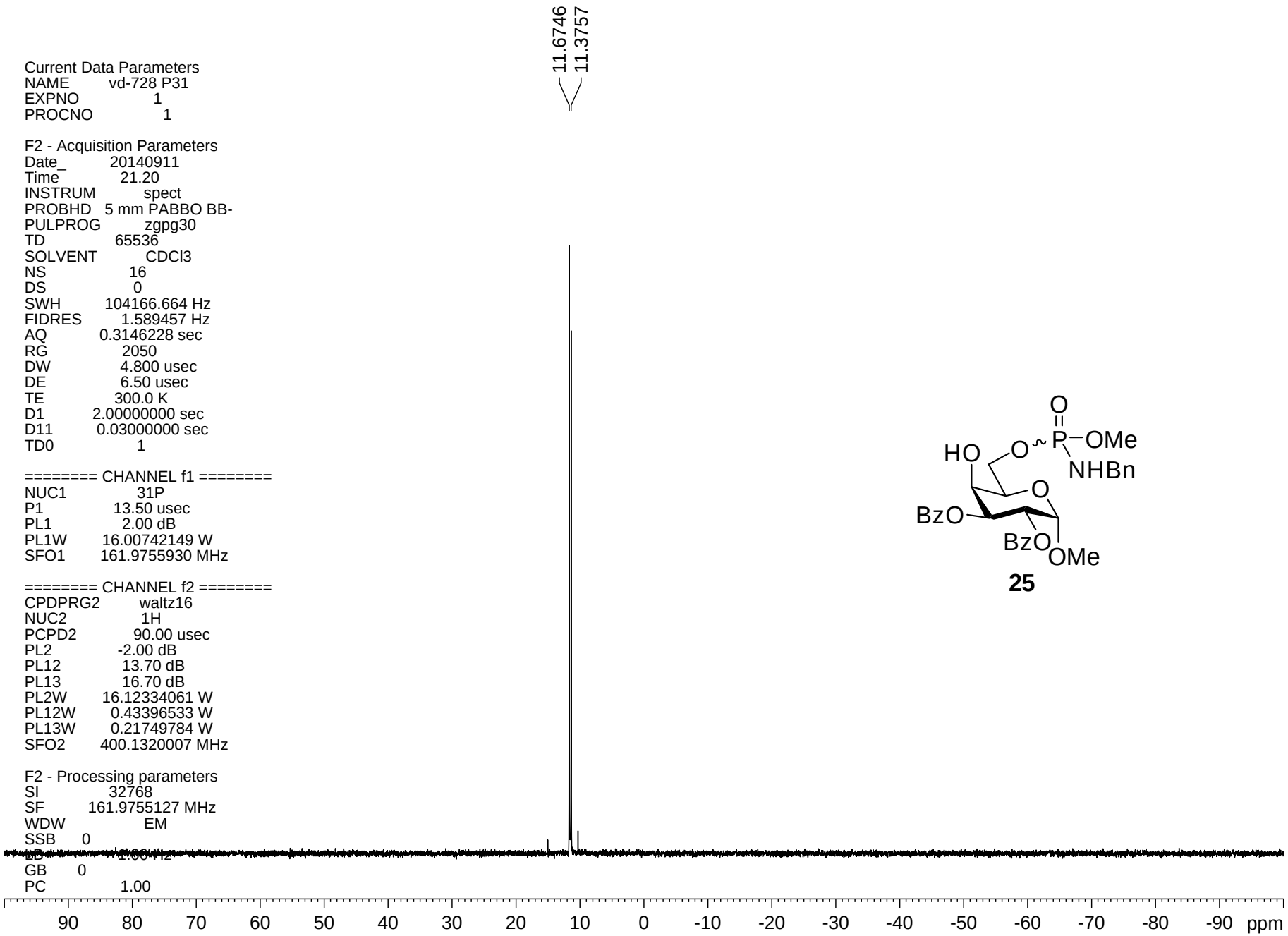
======== CHANNEL f1 ========
NUC1 1H
P1 13.40 usec
PL1 -2.00 dB
PL1W 16.12334061 W
SFO1 400.1326012 MHz

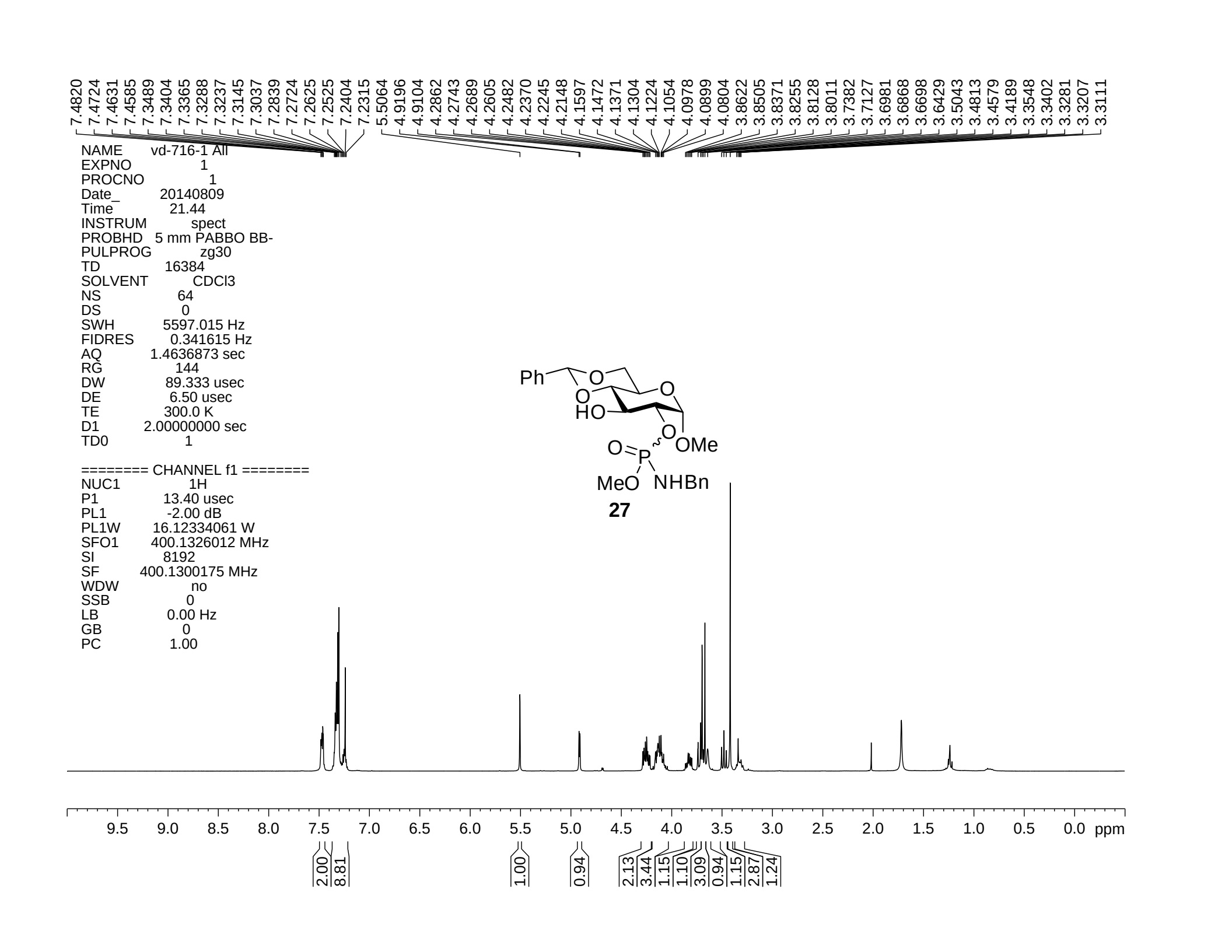
F2 - Processing parameters
SI 8192
SF 400.1300175 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

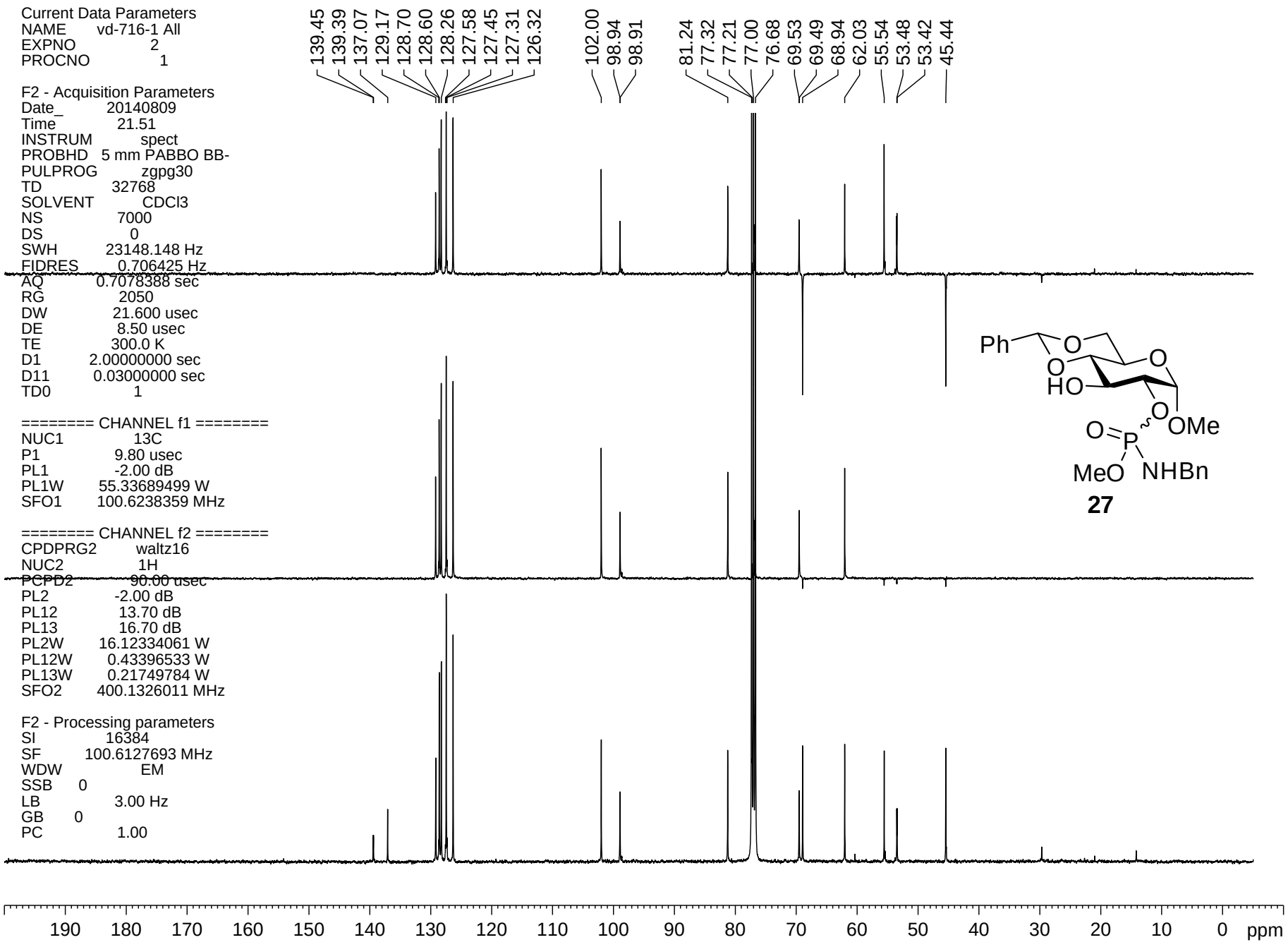
25

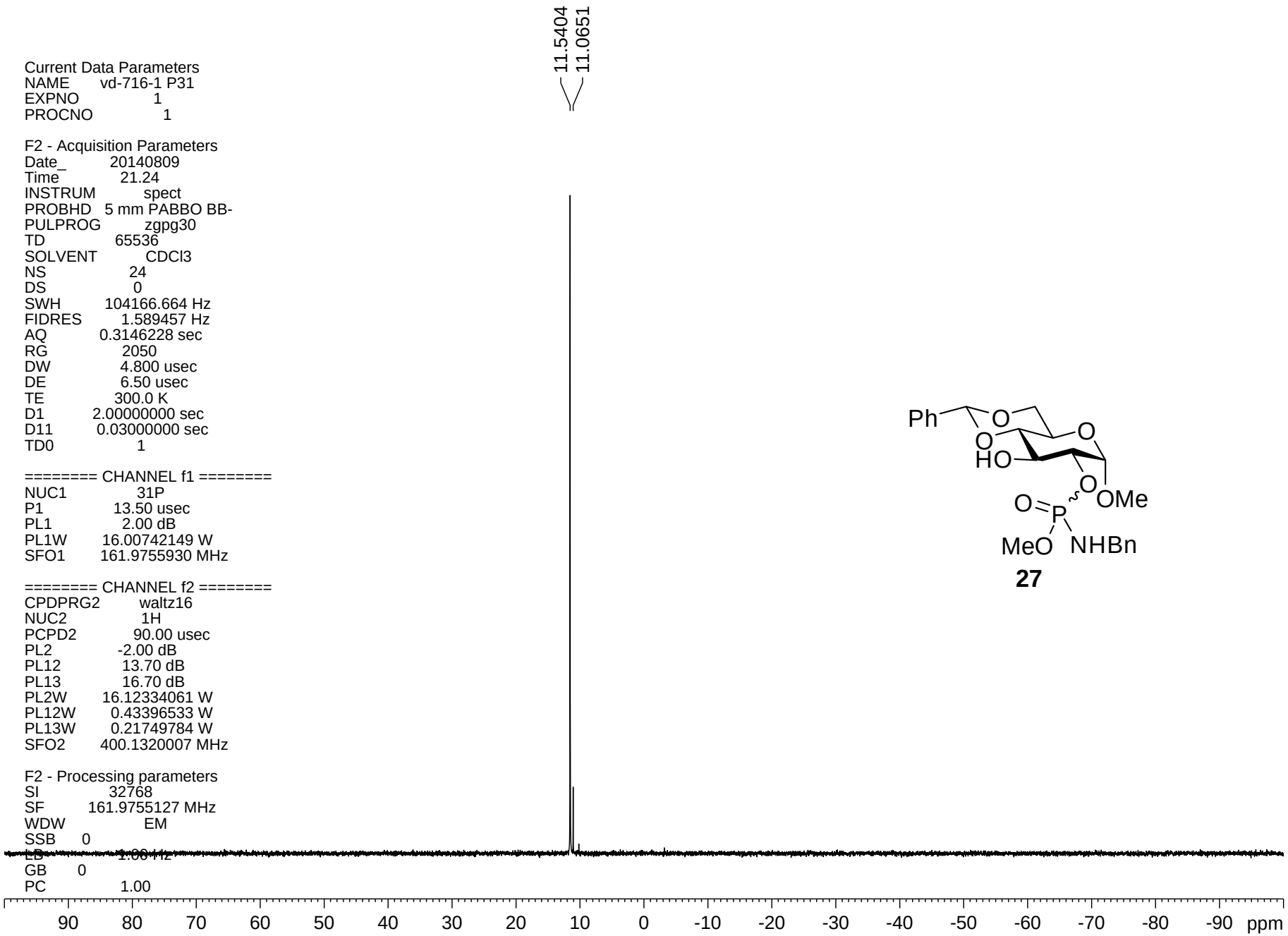


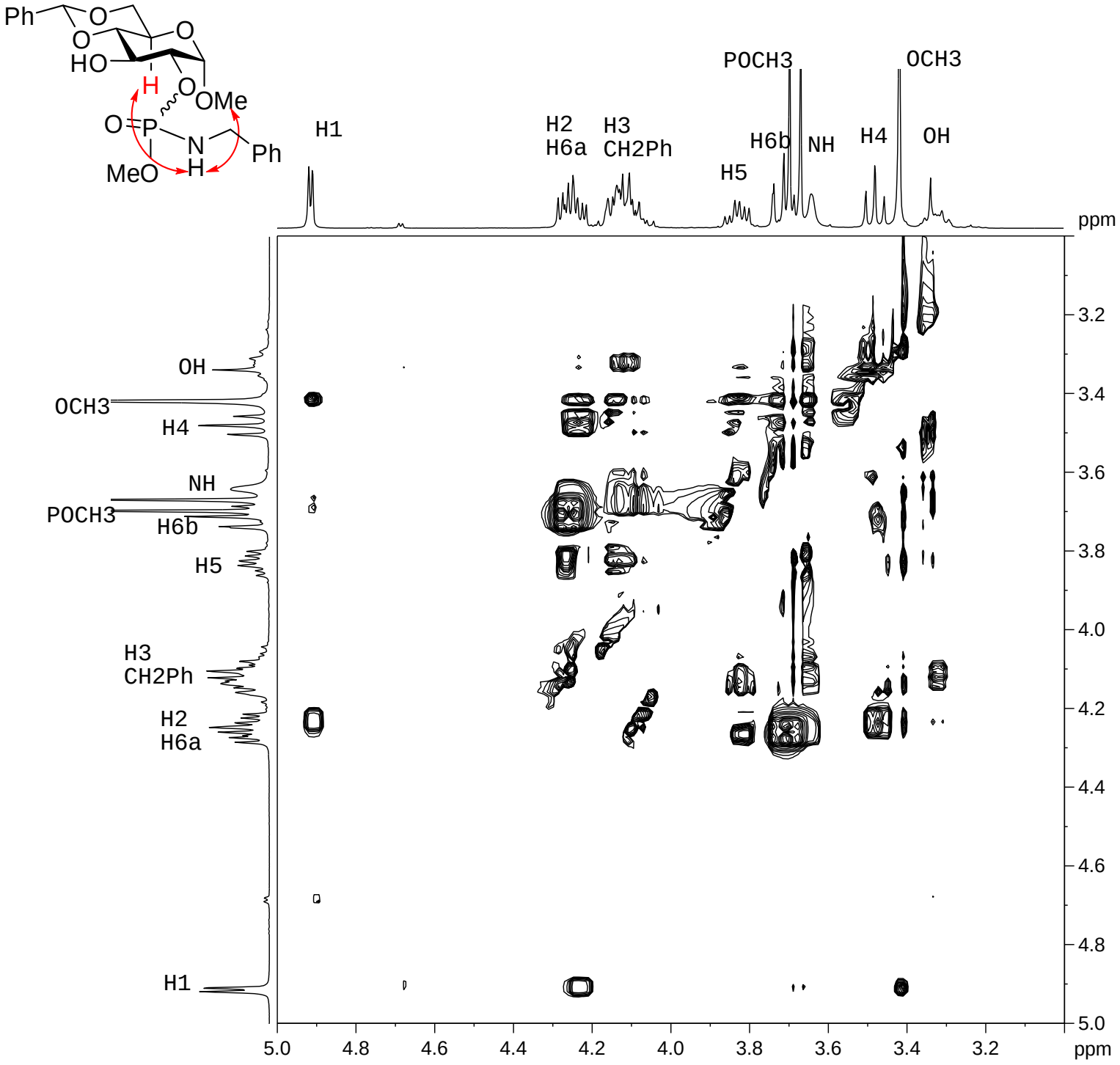

Current Data Parameters
NAME vd-728 all
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140911
Time 21.41
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 6000
DS 0
SWH 23148.148 Hz
FIDRES 0.706425 Hz
AQ 0.7078388 sec
RG 2050
DW 21.600 usec
DE 8.50 usec
TE 300.0 K
D1 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 100.6127700 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
166.04
165.93
165.91
139.31
139.25
139.18
133.12
129.81
129.68
129.52
128.65
128.32
127.52
127.33
97.65
77.32
77.20
77.00
76.68
70.53
70.40
69.08
68.54
68.48
68.41
68.34
67.12
67.01
64.19
64.14
63.94
63.90
61.99
55.54
53.37
53.32
51.60
45.33
HO
O
O
P
OMe
NHBn
O
BzO
BzO
OMe
25
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


11.6746
11.3757
Current Data Parameters
NAME vd-728 P31
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140911
Time 21.20
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
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
======== 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 13.70 dB
PL13 16.70 dB
PL2W 16.12334061 W
PL12W 0.43396533 W
PL13W 0.21749784 W
SFO2 400.1320007 MHz
F2 - Processing parameters
SI 32768
SF 161.9755127 MHz
WDW EM
SSB 0
GB 0
PC 1.00
HO
O
P
OMe
NHBn
O
BzO
BzO
OMe
25
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 ppm


7.4820 7.4724 7.4631 7.4585 7.3489 7.3404 7.3365 7.3288 7.3237 7.3145 7.3037 7.2839 7.2724 7.2625 7.2525 7.2404 7.2315 5.5064 4.9196 4.9104 4.2862 4.2743 4.2689 4.2605 4.2482 4.2370 4.2245 4.2148 4.1597 4.1472 4.1371 4.1304 4.1224 4.1054 4.0978 4.0899 4.0804 3.8622 3.8505 3.8371 3.8255 3.8128 3.8011 3.7382 3.7127 3.6981 3.6868 3.6698 3.6429 3.5043 3.4813 3.4579 3.4189 3.3548 3.3402 3.3281 3.3207 3.3111
NAME vd-716-1 All
EXPNO 1
PROCNO 1
Date_ 20140809
Time 21.44
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 64
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
SI 8192
SF 400.1300175 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
Ph
O
O
HO
O
O
O
OMe
P
MeO
NHBn
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
2.00 8.81 1.00 0.94 2.13 3.44 1.15 1.10 3.09 0.94 1.15 2.87 1.24


Current Data Parameters
NAME vd-716-1 All
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140809
Time 21.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 7000
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 100.6127693 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
139.45
139.39
137.07
129.17
128.70
128.60
128.26
127.58
127.45
127.31
126.32
102.00
98.94
98.91
81.24
77.32
77.21
77.00
76.68
69.53
69.49
68.94
62.03
55.54
53.48
53.42
45.44
Ph
O
O
HO
O
O
OMe
O
P
MeO
NHBn
27
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


11.5404
11.0651
Current Data Parameters
NAME vd-716-1 P31
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140809
Time 21.24
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 24
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
======== 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 13.70 dB
PL13 16.70 dB
PL2W 16.12334061 W
PL12W 0.43396533 W
PL13W 0.21749784 W
SFO2 400.1320007 MHz
F2 - Processing parameters
SI 32768
SF 161.9755127 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
Ph
O
O
O
HO
O
OMe
O
P
MeO
NHBn
27
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
ppm

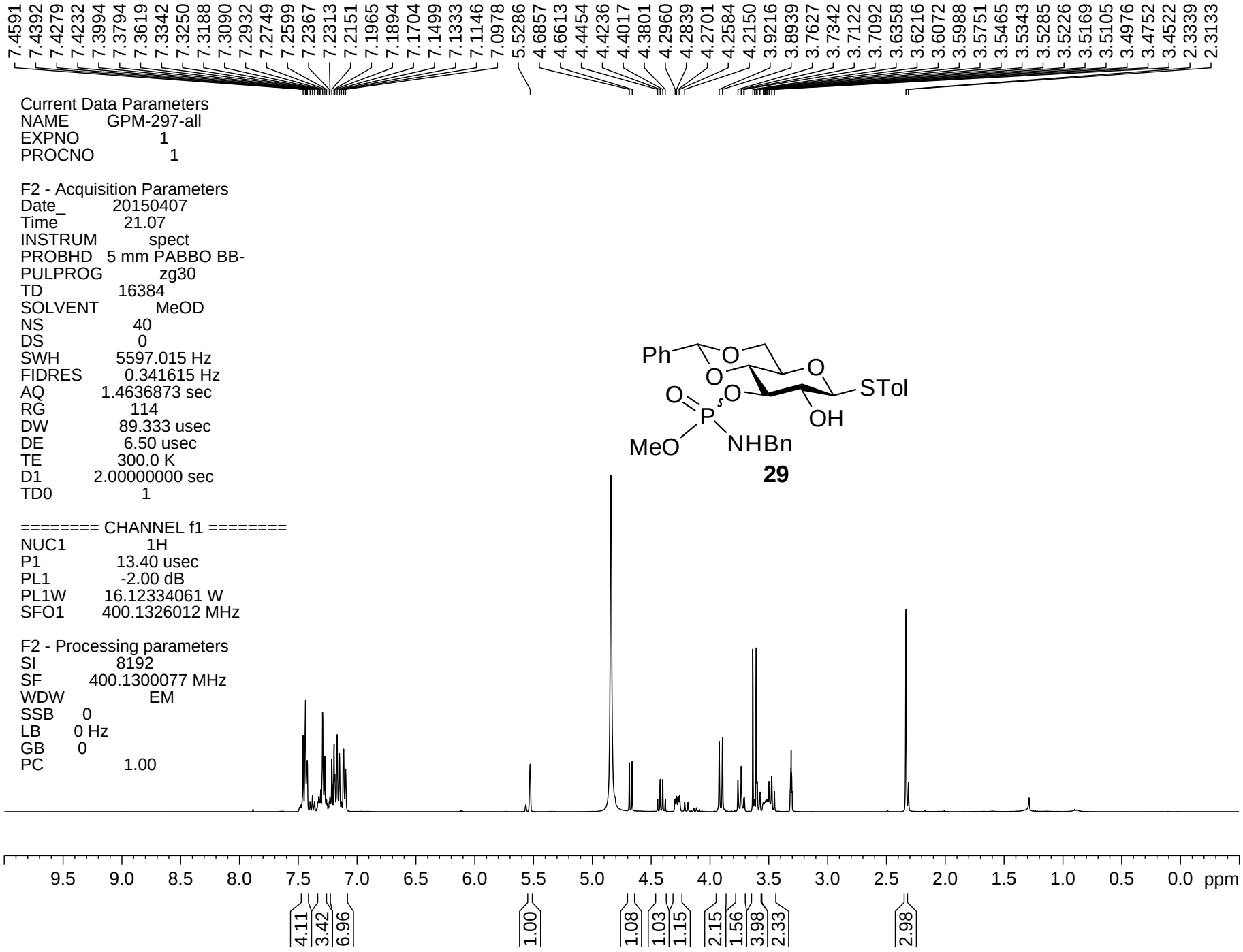


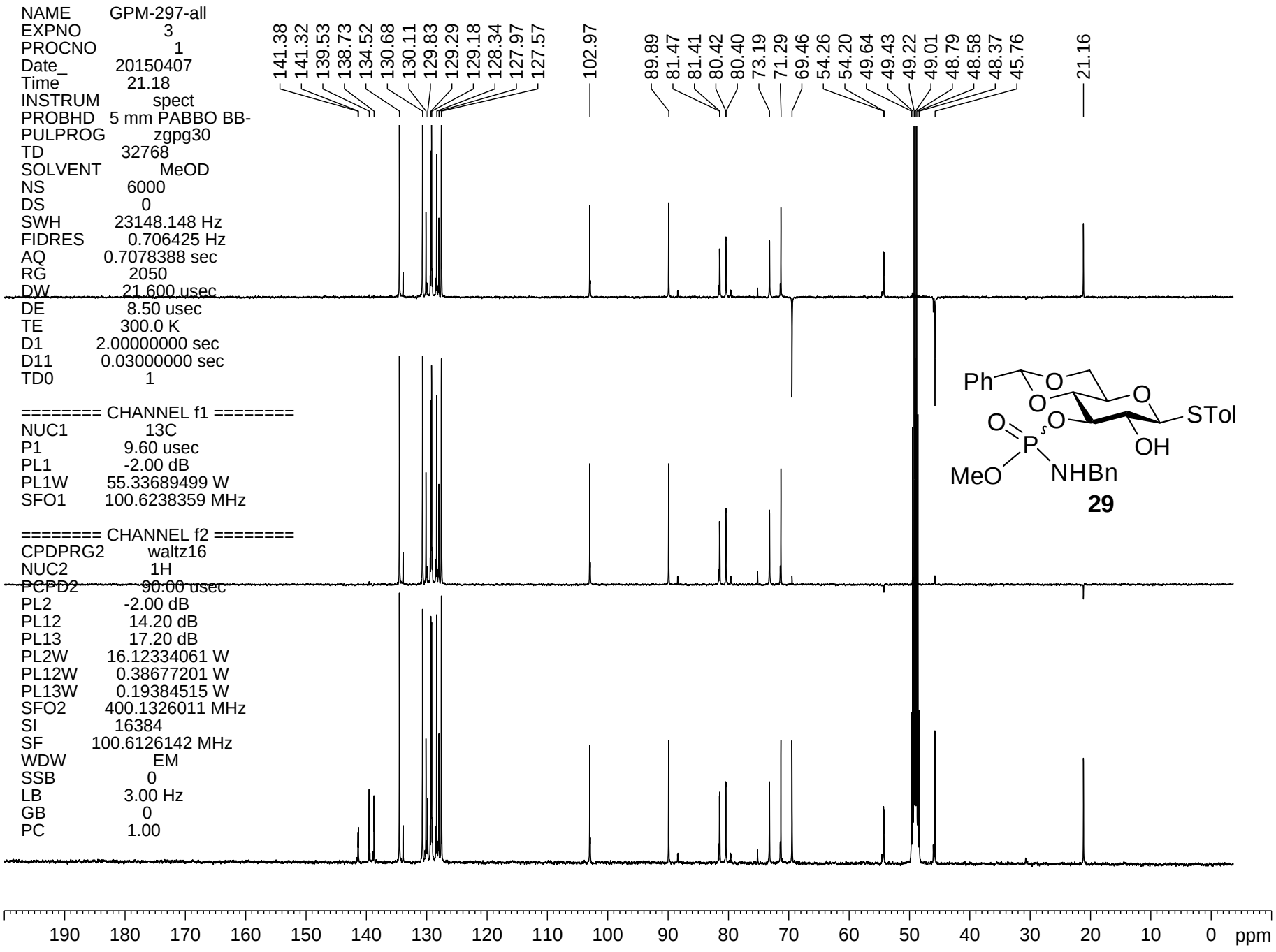
Current Data Parameters
NAME vd-716-1 All
EXPNO 7
PROCNO 1

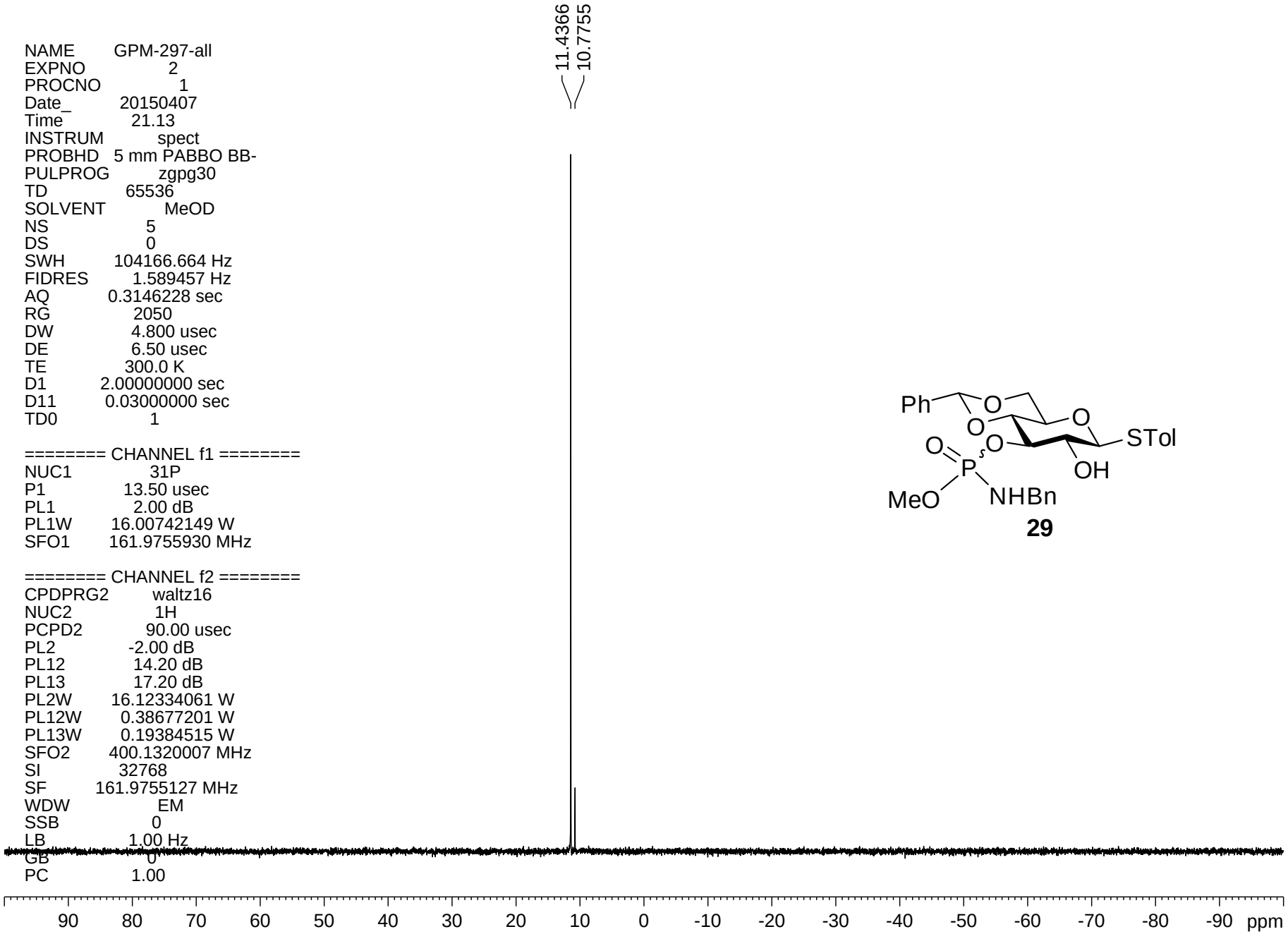
F2 - Acquisition Parameters
Date_ 20140810
Time 8.49
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG noesygpphpp
TD 2048
SOLVENT CDCl3
NS 4
DS 16
SWH 5197.505 Hz
FIDRES 2.537844 Hz
AQ 0.1970676 sec
RG 256
DW 96.200 usec
DE 6.50 usec
TE 300.0 K
D0 0.00007914 sec
D1 2.00000000 sec
D8 0.40000001 sec
D11 0.03000000 sec
D12 0.00002000 sec
D16 0.00020000 sec
IN0 0.00019240 sec

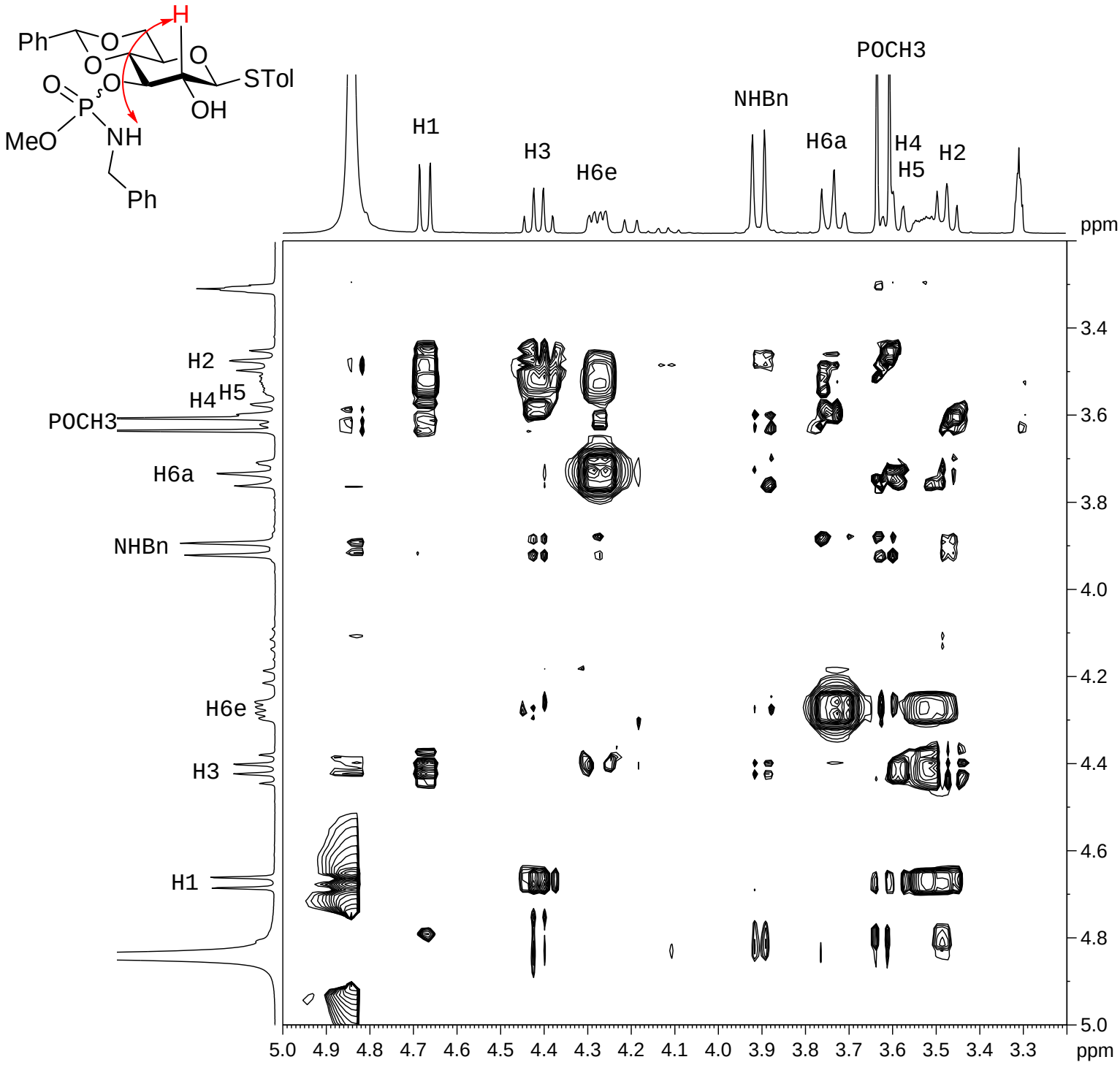
======== CHANNEL f1 ======:
NUC1 1H
P1 13.40 usec
P2 26.80 usec
P17 2500.00 usec
PL1 -2.00 dB
PL10 3.30 dB
PL1W 16.12334061 W
PL10W 4.75833511 W
SFO1 400.1318006 MHz

====== GRADIENT CHANNEL =
GPNAM1 SMSQ10.100
GPNAM2 SMSQ10.100
GPZ1 40.00 %


7.4591 7.4392 7.4279 7.4232 7.3994 7.3794 7.3619 7.3342 7.3250 7.3188 7.3090 7.2932 7.2749 7.2599 7.2367 7.2313 7.2151 7.1965 7.1894 7.1704 7.1499 7.1333 7.1146 7.0978 5.5286 4.6857 4.6613 4.4454 4.4236 4.4017 4.3801 4.2960 4.2839 4.2701 4.2584 4.2150 3.9216 3.8939 3.7627 3.7342 3.7122 3.7092 3.6358 3.6216 3.6072 3.5988 3.5751 3.5465 3.5343 3.5285 3.5226 3.5169 3.5105 3.4976 3.4752 3.4522 2.3339 2.3133
Current Data Parameters
NAME GPM-297-all
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150407
Time 21.07
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT MeOD
NS 40
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 13.40 usec
PL1 -2.00 dB
PL1W 16.12334061 W
SFO1 400.1326012 MHz
F2 - Processing parameters
SI 8192
SF 400.1300077 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
Ph
O
O
O
O
O
P
STol
OH
MeO
NHBn
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
4.11 3.42 6.96 1.00 1.08 1.03 1.15 2.15 1.56 3.98 2.33 2.98


NAME GPM-297-all
EXPNO 3
PROCNO 1
Date_ 20150407
Time 21.18
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT MeOD
NS 6000
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 9.60 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 14.20 dB
PL13 17.20 dB
PL2W 16.12334061 W
PL12W 0.38677201 W
PL13W 0.19384515 W
SFO2 400.1326011 MHz
SI 16384
SF 100.6126142 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
141.38
141.32
139.53
138.73
134.52
130.68
130.11
129.83
129.29
129.18
128.34
127.97
127.57
102.97
89.89
81.47
81.41
80.42
80.40
73.19
71.29
69.46
54.26
54.20
49.64
49.43
49.22
49.01
48.79
48.58
48.37
45.76
21.16
Ph
O
O
O
O
O
STol
OH
P
MeO
NHBn
29
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


11.4366
10.7755
NAME GPM-297-all
EXPNO 2
PROCNO 1
Date_ 20150407
Time 21.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 5
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
======== 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
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 ppm
Ph
O
O
O
O
O
STol
P
s
OH
MeO
NHBn
29

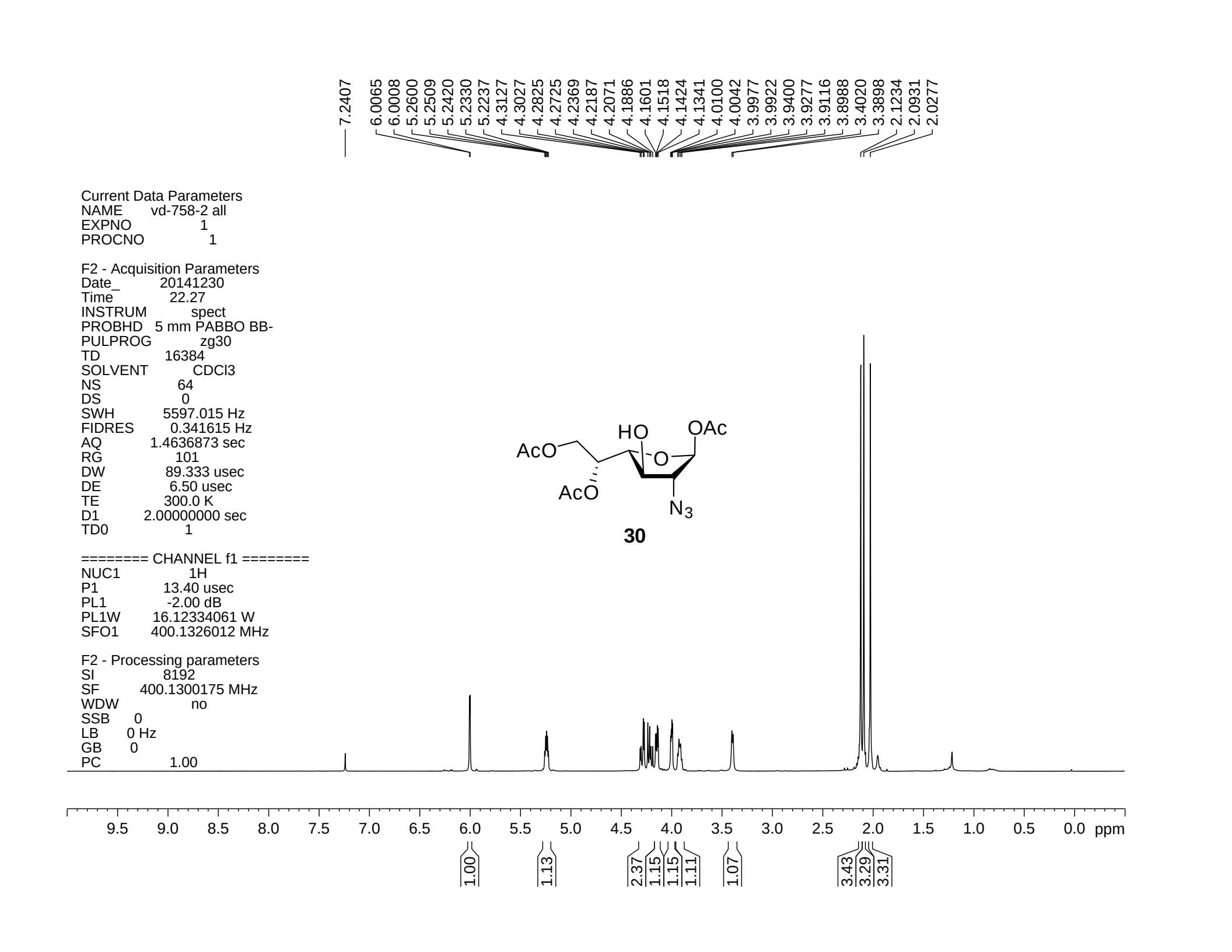


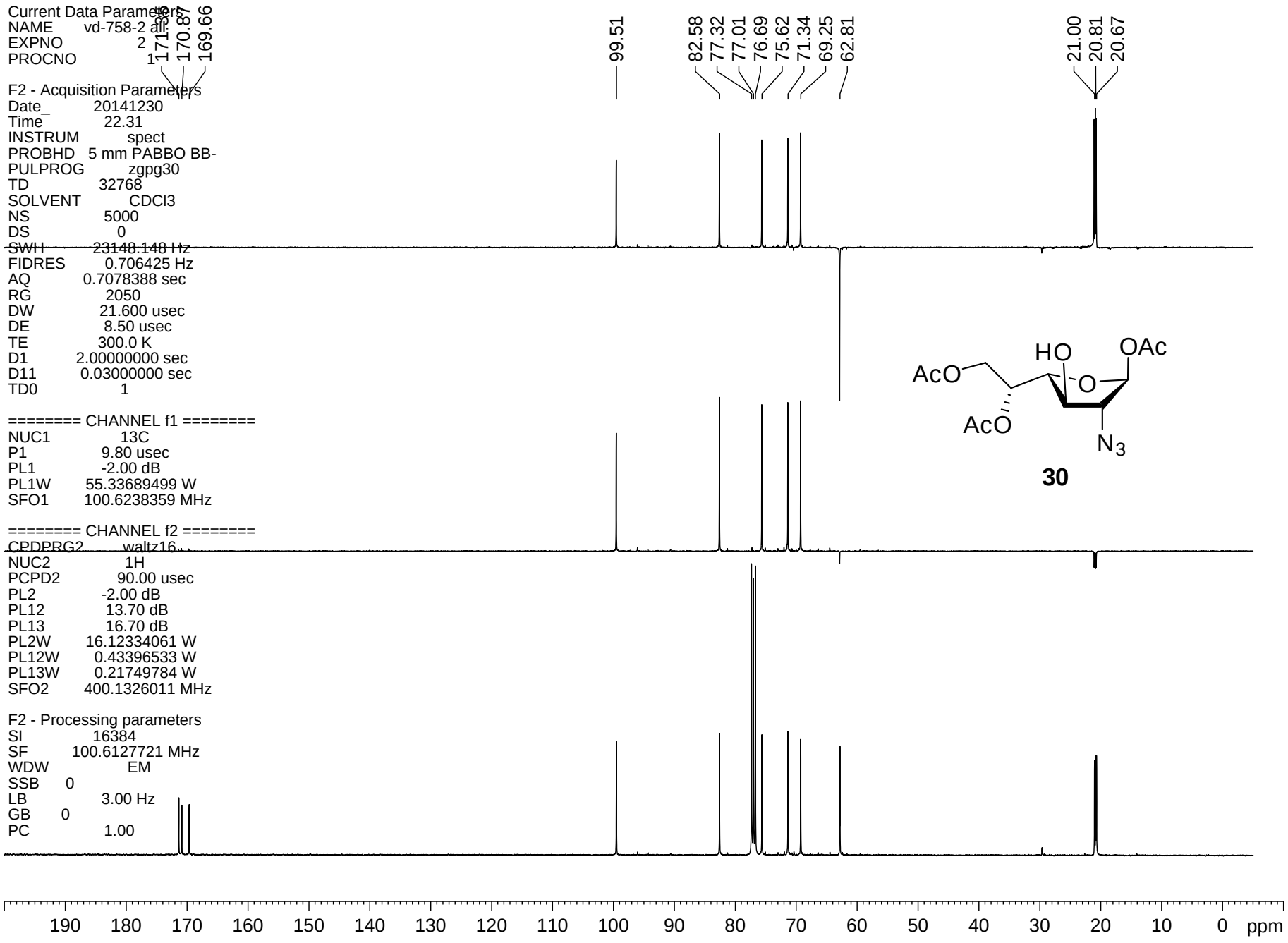
Current Data Parameters
NAME GPM-297-all
EXPNO 8
PROCNO 1

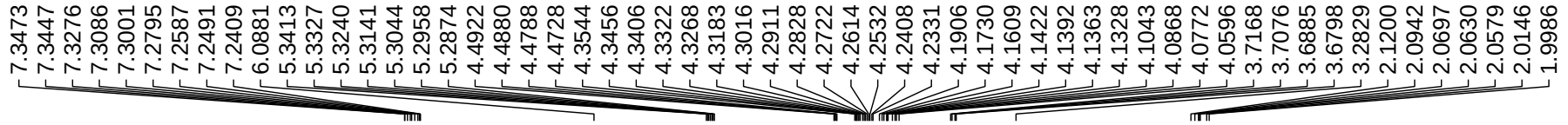
F2 - Acquisition Parameters
Date_ 20150408
Time 7.32
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG noesygpphpp
TD 2048
SOLVENT MeOD
NS 4
DS 16
SWH 5197.505 Hz
FIDRES 2.537844 Hz
AQ 0.1970676 sec
RG 287
DW 96.200 usec
DE 6.50 usec
TE 300.0 K
D0 0.00007914 sec
D1 2.00000000 sec
D8 0.40000001 sec
D11 0.03000000 sec
D12 0.00002000 sec
D16 0.00020000 sec
IN0 0.00019240 sec

======== CHANNEL f1 ======
NUC1 1H
P1 13.40 usec
P2 26.80 usec
P17 2500.00 usec
PL1 -2.00 dB
PL10 3.30 dB
PL1W 16.12334061 W
PL10W 4.75833511 W
SFO1 400.1318006 MHz

====== GRADIENT CHANNEL =
GPNAM1 SMSQ10.100
GPNAM2 SMSQ10.100
GPZ1 40.00 %


7.2407
6.0065
6.0008
5.2600
5.2509
5.2420
5.2330
5.2237
4.3127
4.3027
4.2825
4.2725
4.2369
4.2187
4.2071
4.1886
4.1601
4.1518
4.1424
4.1341
4.0100
4.0042
3.9977
3.9922
3.9400
3.9277
3.9116
3.8988
3.4020
3.3898
2.1234
2.0931
2.0277
Current Data Parameters
NAME vd-758-2 all
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141230
Time 22.27
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 64
DS 0
SWH 5597.015 Hz
FIDRES 0.341615 Hz
AQ 1.4636873 sec
RG 101
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
HO
OAc
AcO
O
AcO
N3
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
1.00
1.13
2.37
1.15
1.15
1.11
1.07
3.43
3.29
3.31


Current Data Parameters
NAME vd-758-2 all
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141230
Time 22.31
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 5000
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 100.6127721 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
171.38
170.87
169.66
99.51
82.58
77.32
77.01
76.69
75.62
71.34
69.25
62.81
21.00
20.81
20.67
HO
OAc
AcO
O
AcO
N3
30
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm



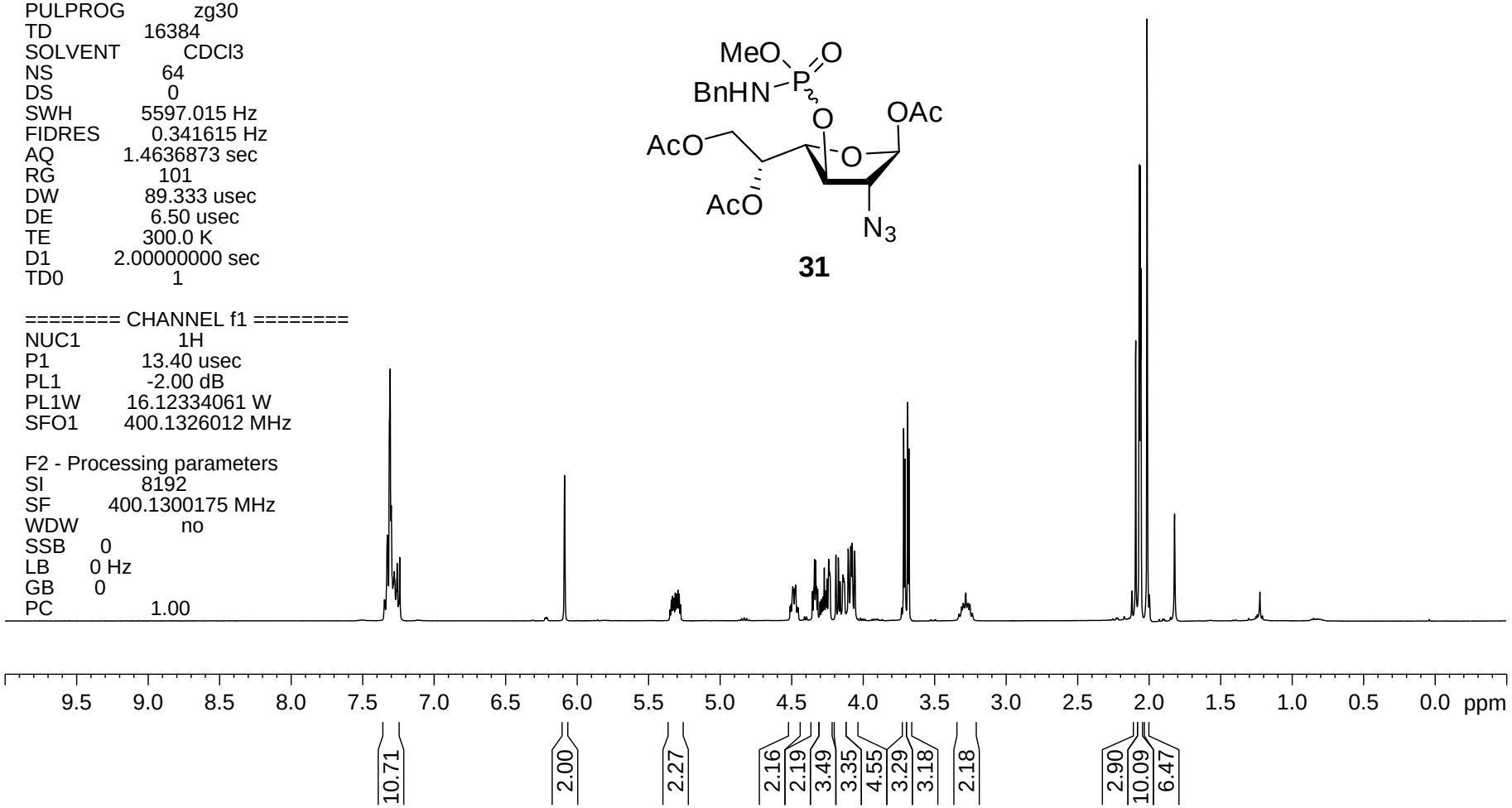
Current Data Parameters
NAME vd-759 all
EXPNO 1
PROCNO 1

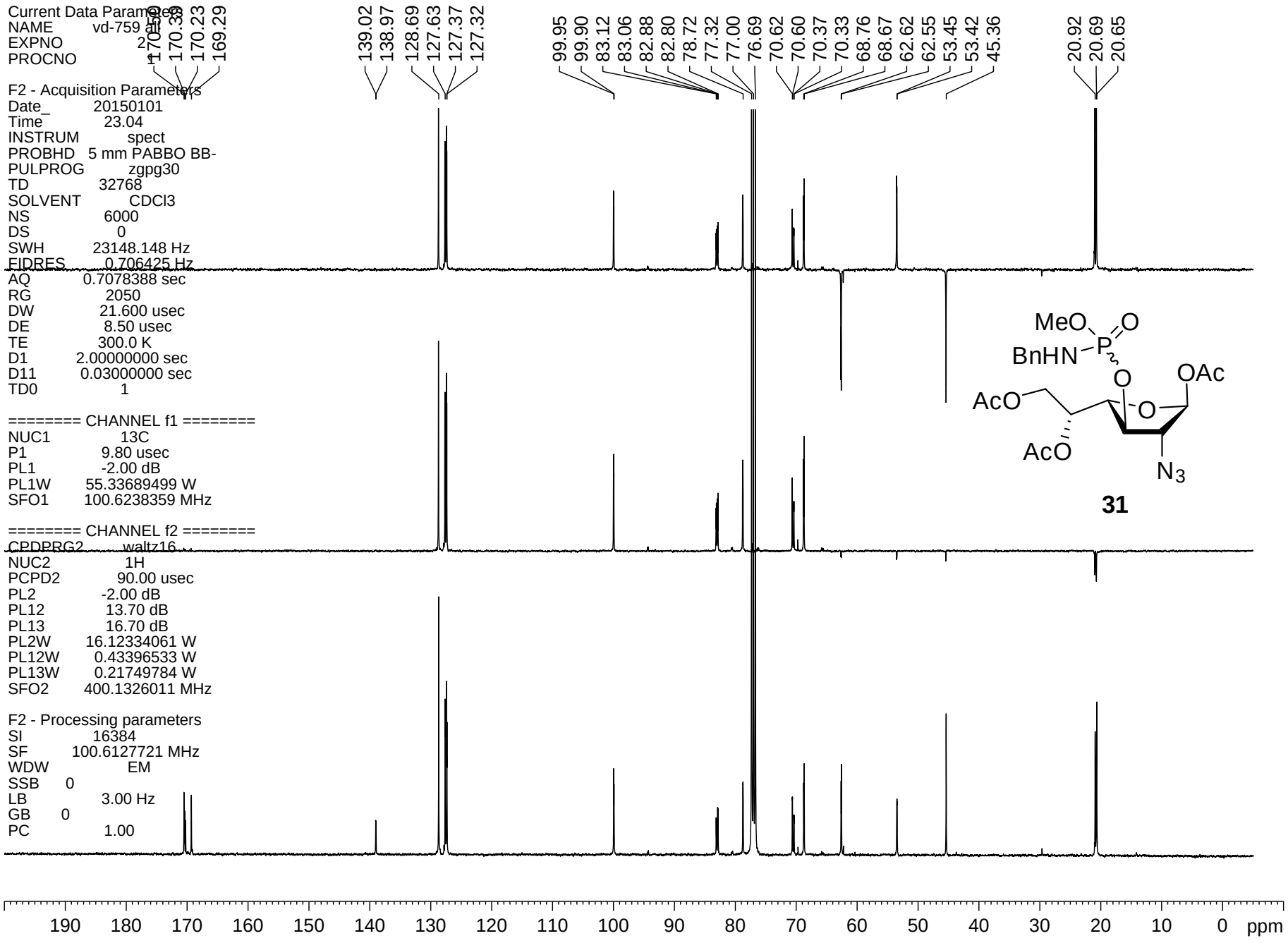
F2 - Acquisition Parameters
Date_ 20150101
Time 23.03
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 64
DS 0
SWH 5597.015 Hz
FIDRES 0.341615 Hz
AQ 1.4636873 sec
RG 101
DW 89.333 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

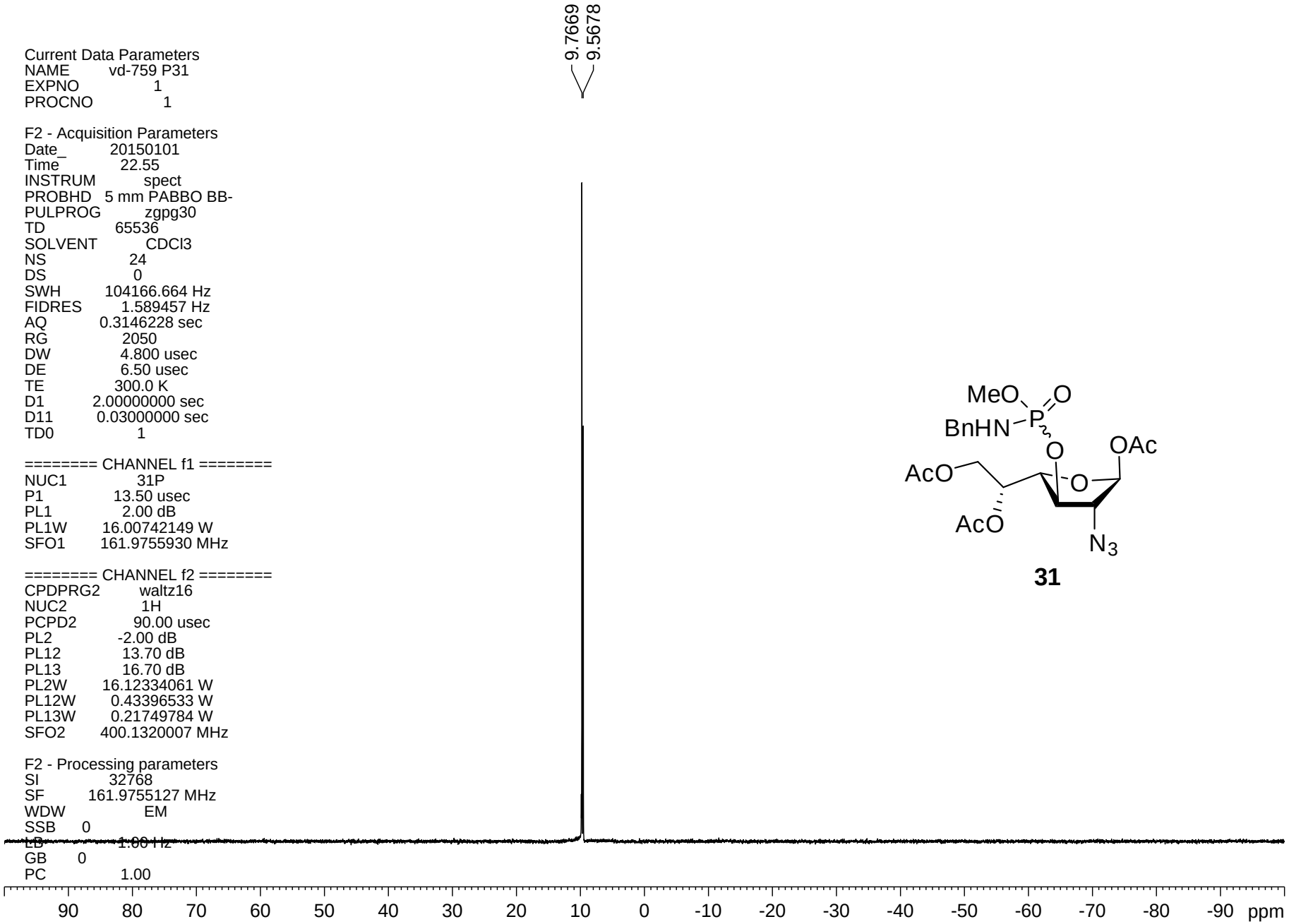
======== CHANNEL f1 ========
NUC1 1H
P1 13.40 usec
PL1 -2.00 dB
PL1W 16.12334061 W
SFO1 400.1326012 MHz

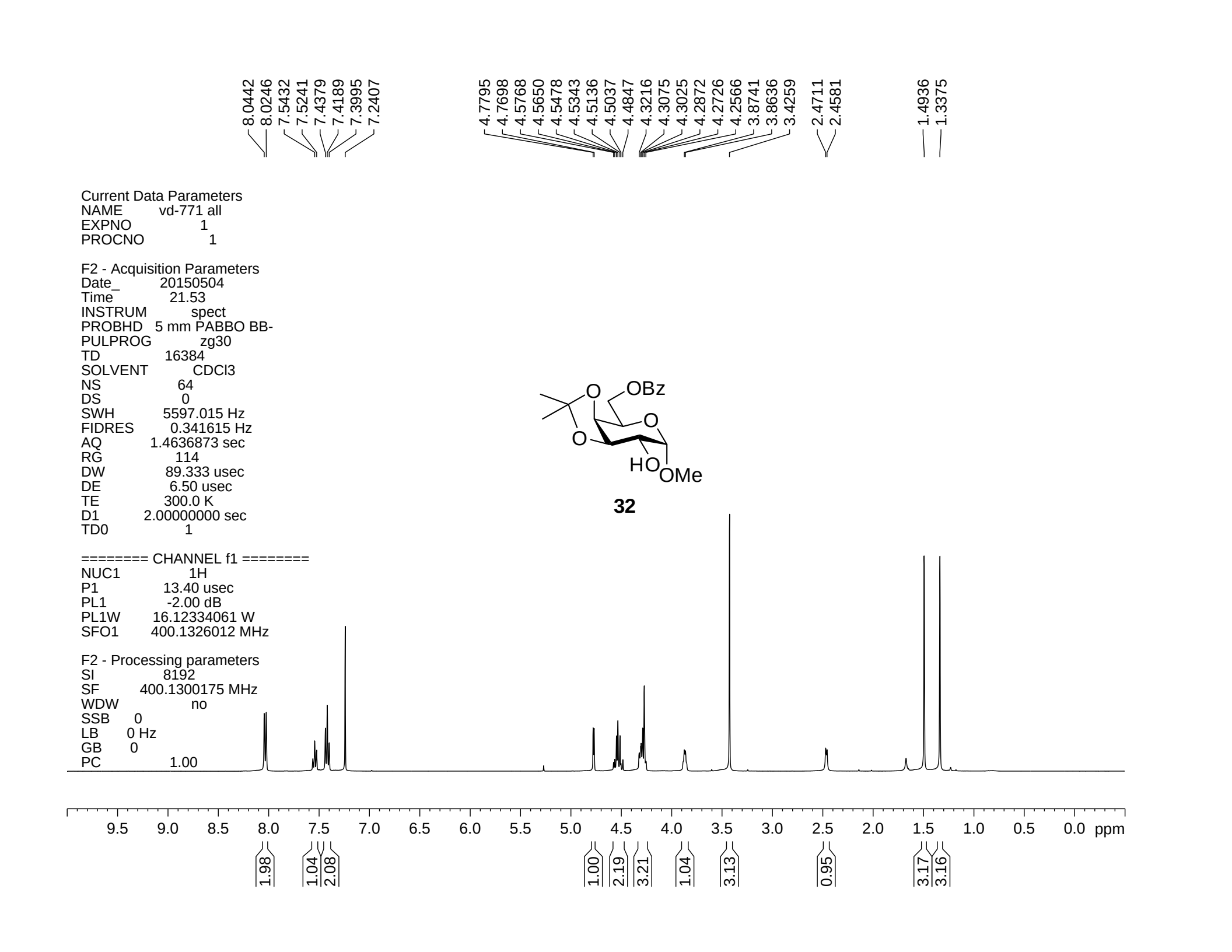
F2 - Processing parameters
SI 8192
SF 400.1300175 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

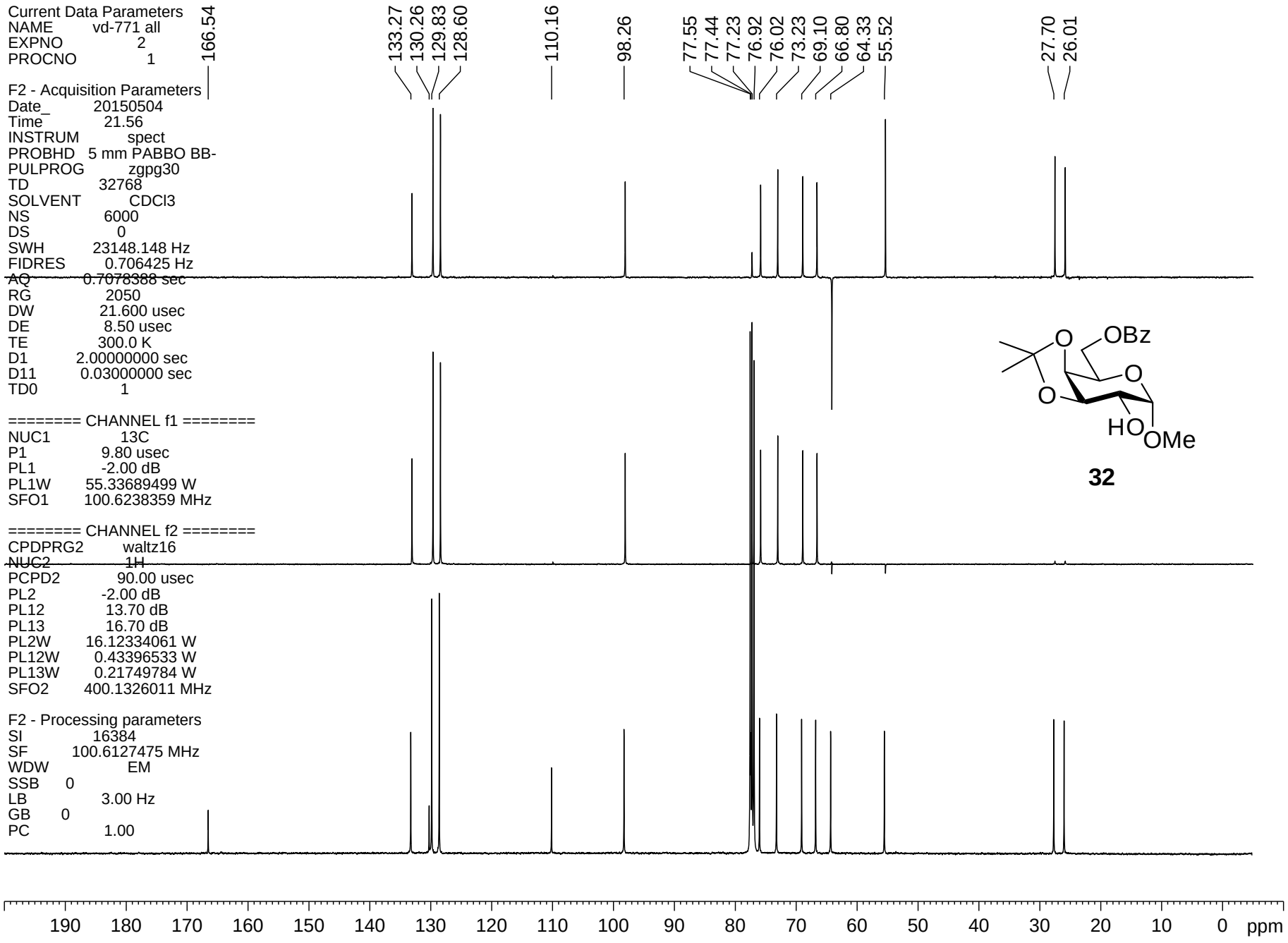
31

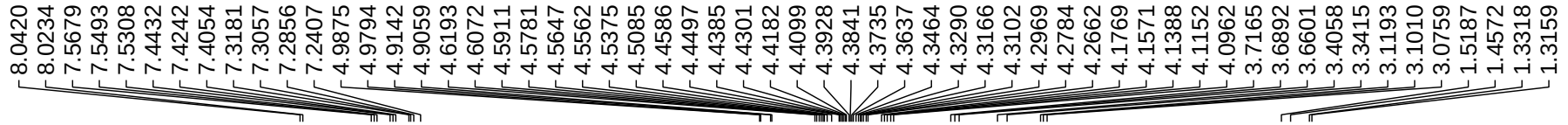



Current Data Parameters
NAME vd-759 al
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150101
Time 23.04
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 6000
DS 0
SWH 23148.148 Hz
FIDRES 0.706425 Hz
AQ 0.7078388 sec
RG 2050
DW 21.600 usec
DE 8.50 usec
TE 300.0 K
D1 2.00000000 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 100.6127721 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
170.45
170.38
170.23
169.29
139.02
138.97
128.69
127.63
127.37
127.32
99.95
99.90
83.12
83.06
82.88
82.80
78.72
77.32
77.00
76.69
70.62
70.60
70.37
70.33
68.76
68.67
62.62
62.55
53.45
53.42
45.36
20.92
20.69
20.65
MeO
O
P
BnHN
O
OAc
AcO
O
AcO
N3
31
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


9.7669
9.5678
Current Data Parameters
NAME vd-759 P31
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150101
Time 22.55
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 24
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
======== 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 13.70 dB
PL13 16.70 dB
PL2W 16.12334061 W
PL12W 0.43396533 W
PL13W 0.21749784 W
SFO2 400.1320007 MHz
F2 - Processing parameters
SI 32768
SF 161.9755127 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
MeO
O
P
BnHN
O
OAc
AcO
O
AcO
N3
31
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 ppm


8.0442
8.0246
7.5432
7.5241
7.4379
7.4189
7.3995
7.2407
4.7795
4.7698
4.5768
4.5650
4.5478
4.5343
4.5136
4.5037
4.4847
4.3216
4.3075
4.3025
4.2872
4.2726
4.2566
3.8741
3.8636
3.4259
2.4711
2.4581
1.4936
1.3375
Current Data Parameters
NAME vd-771 all
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150504
Time 21.53
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 64
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 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
O
OBz
O
O
HO
OMe
32
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
1.98
1.04
2.08
1.00
2.19
3.21
1.04
3.13
0.95
3.17
3.16


Current Data Parameters
NAME vd-771 all
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150504
Time 21.56
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 6000
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 100.6127475 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
166.54
133.27
130.26
129.83
128.60
110.16
98.26
77.55
77.44
77.23
76.92
76.02
73.23
69.10
66.80
64.33
55.52
27.70
26.01
O
OBz
O
O
HO
OMe
32
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

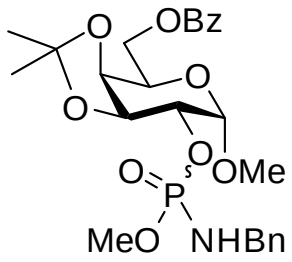


Current Data Parameters
NAME vd-772 all
EXPNO 1
PROCNO 1

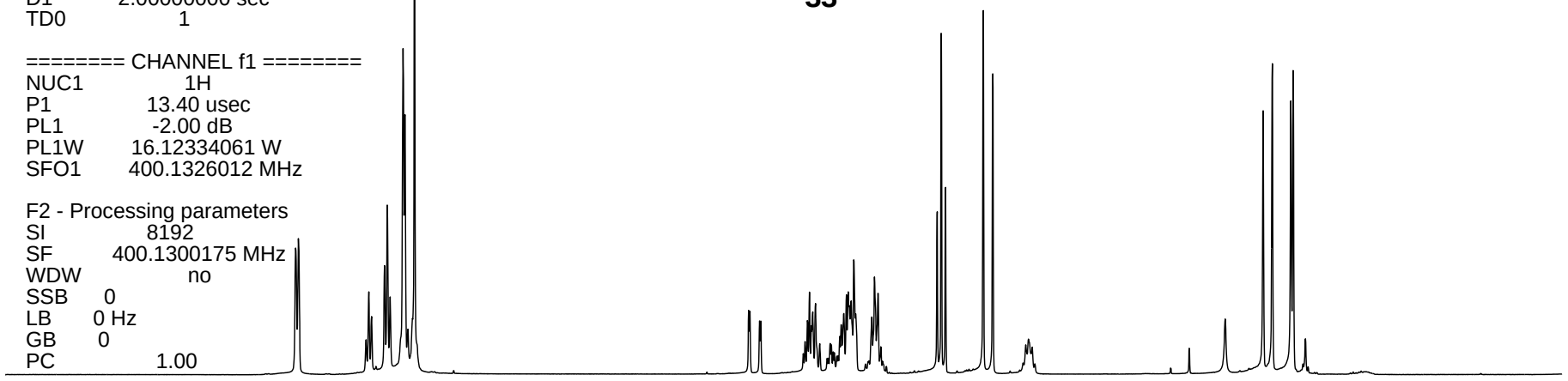
F2 - Acquisition Parameters
Date_ 20150430
Time 21.48
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 64
DS 0
SWH 5597.015 Hz
FIDRES 0.341615 Hz
AQ 1.4636873 sec
RG 114
DW 89.333 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

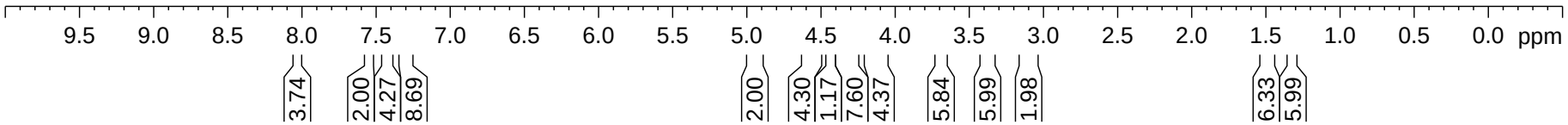
======== CHANNEL f1 ========
NUC1 1H
P1 13.40 usec
PL1 -2.00 dB
PL1W 16.12334061 W
SFO1 400.1326012 MHz

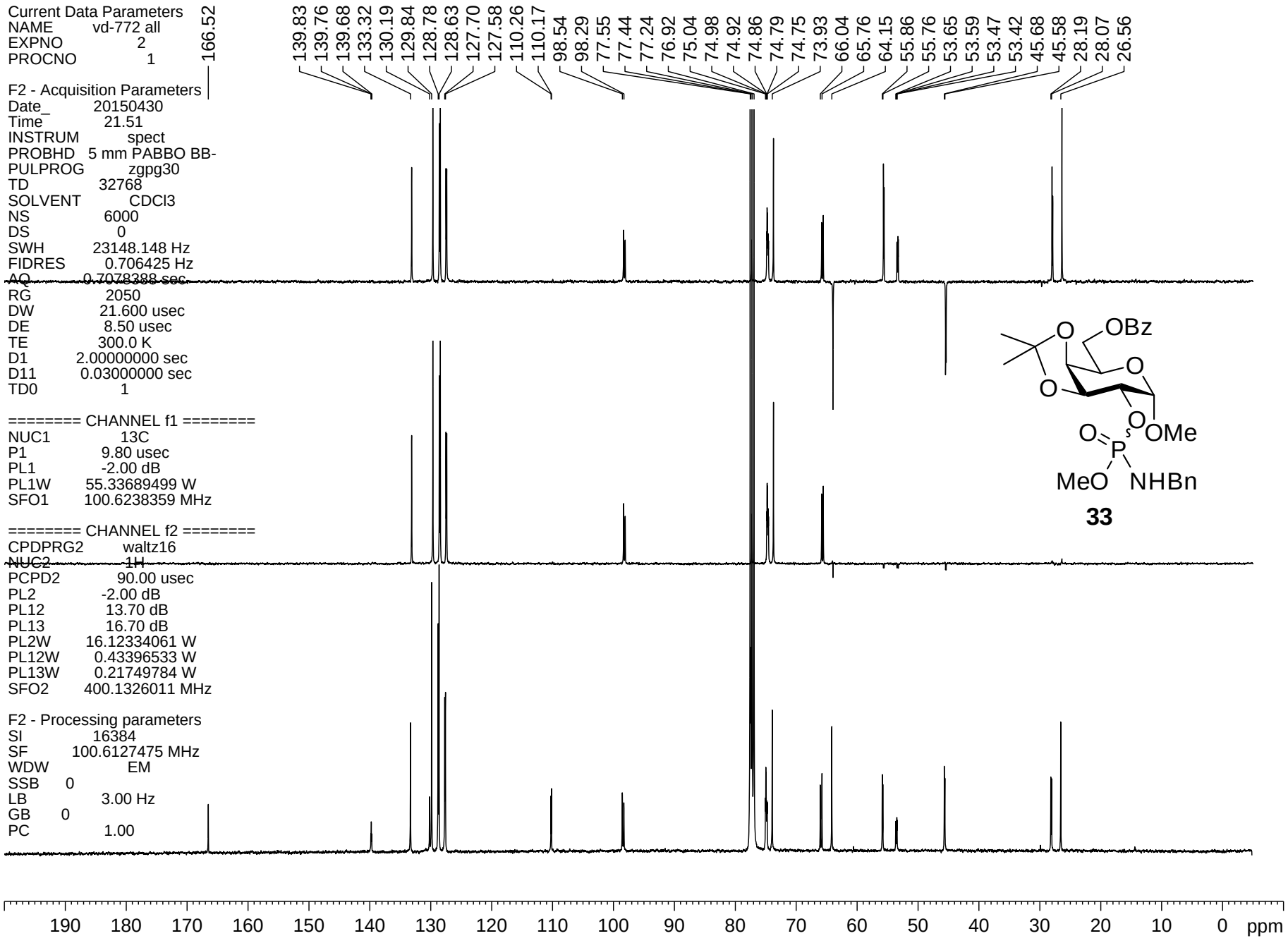
F2 - Processing parameters
SI 8192
SF 400.1300175 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

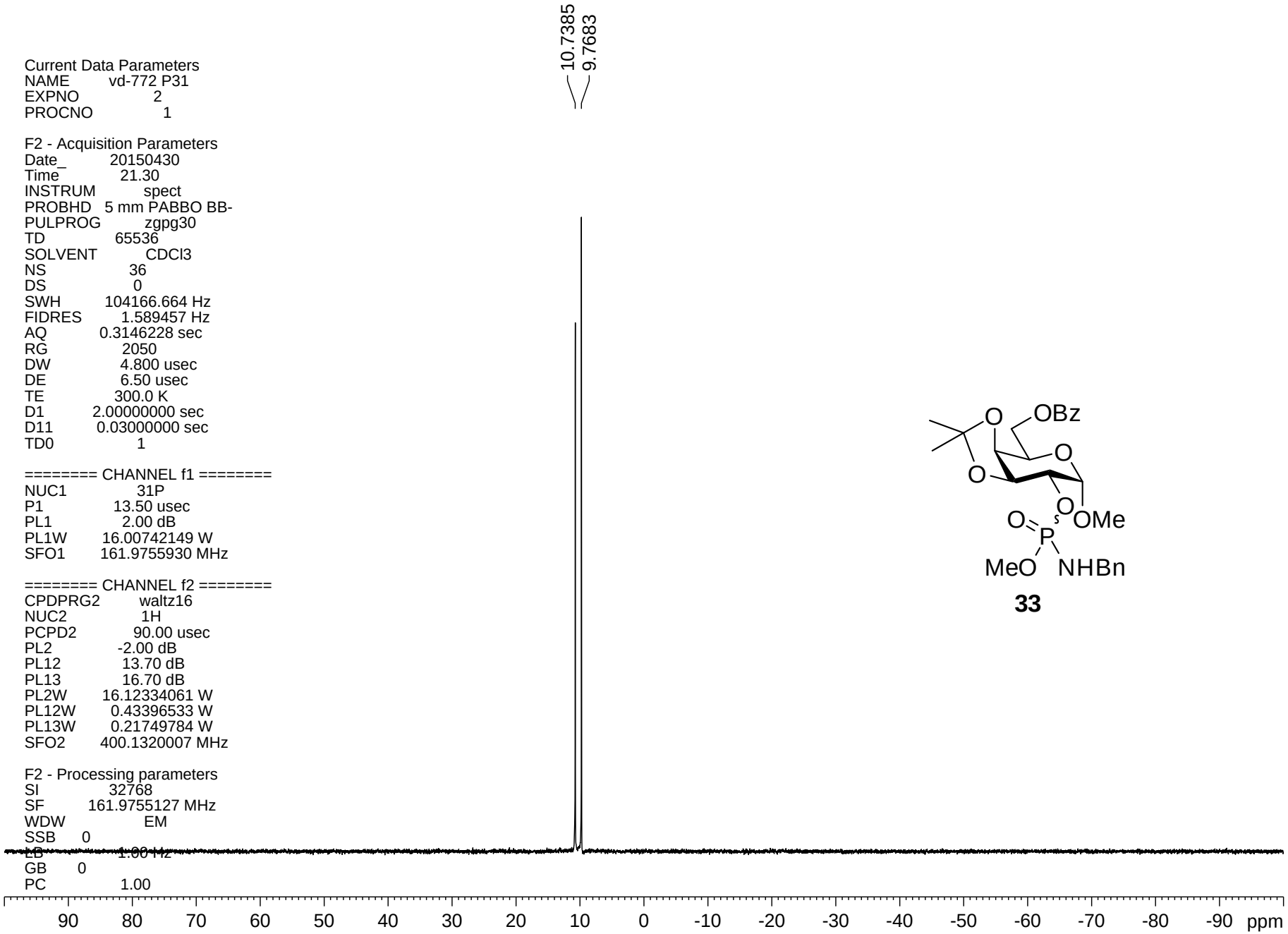


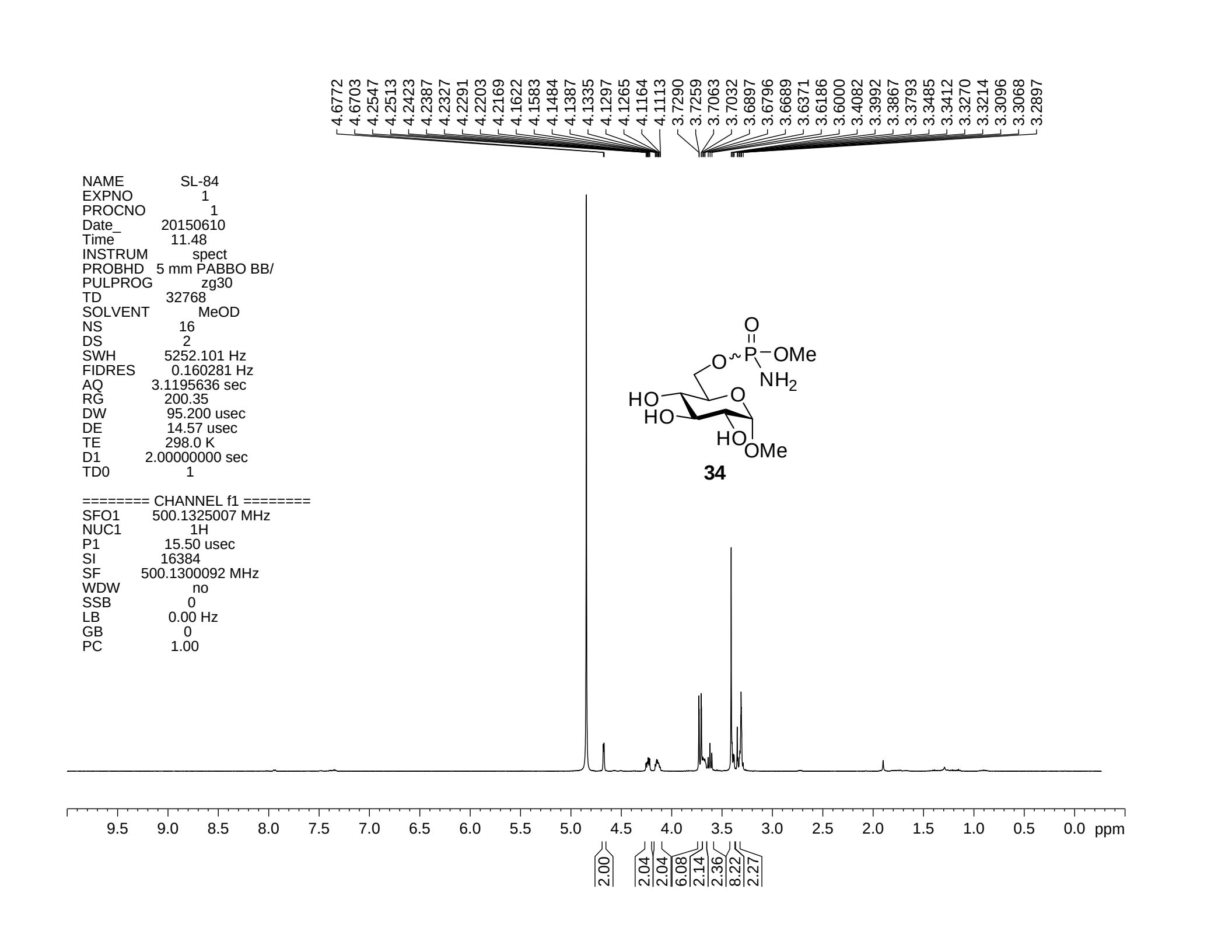
33

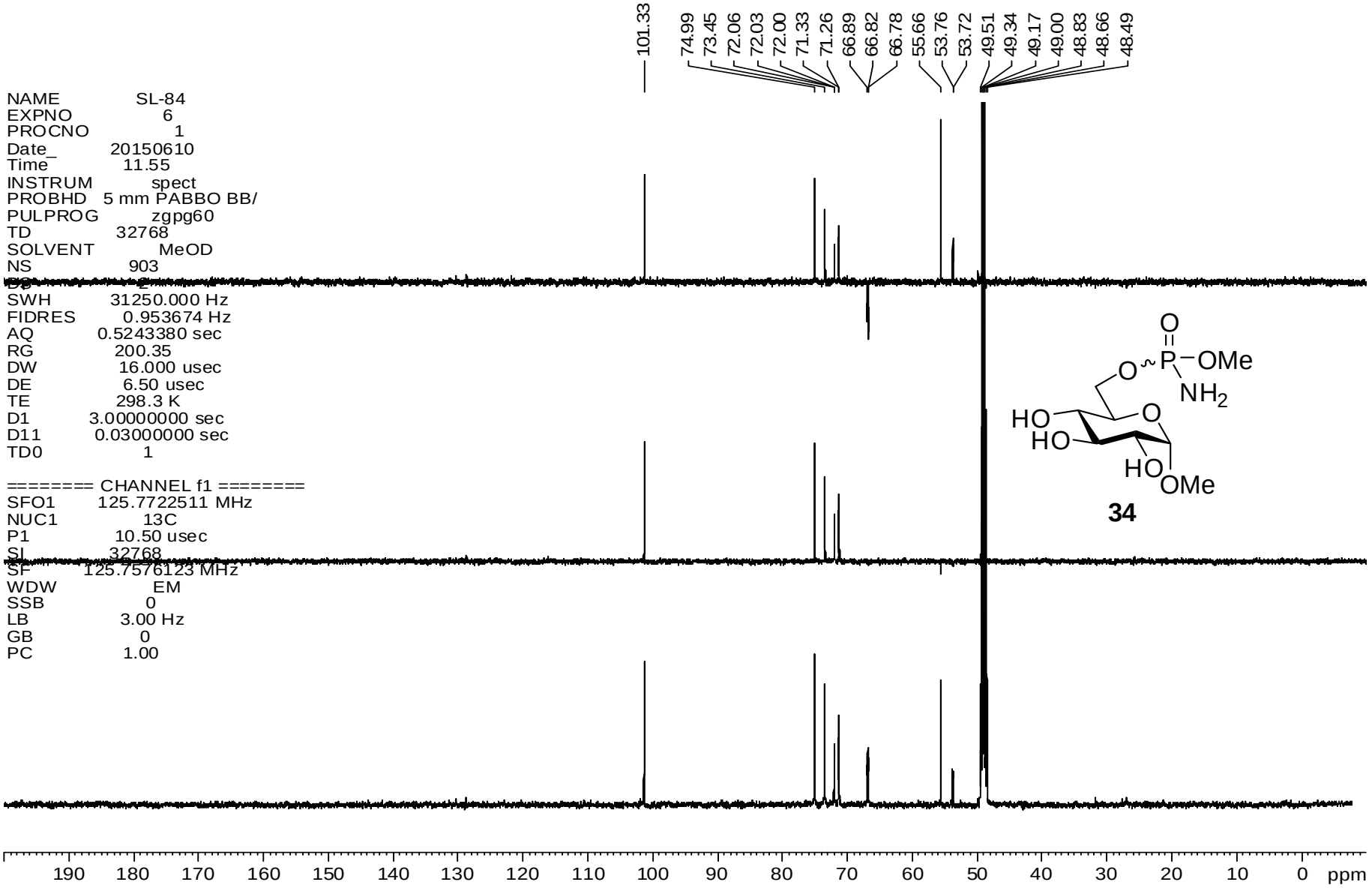





Current Data Parameters
NAME vd-772 all
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150430
Time 21.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 6000
DS 0
SWH 23148.148 Hz
FIDRES 0.706425 Hz
AQ 0.7078388 sec
RG 2050
DW 21.600 usec
DE 8.50 usec
TE 300.0 K
D1 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 100.6127475 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
166.52
139.83
139.76
139.68
133.32
130.19
129.84
128.78
128.63
127.70
127.58
110.26
110.17
98.54
98.29
77.55
77.44
77.24
76.92
75.04
74.98
74.92
74.86
74.79
74.75
73.93
66.04
65.76
64.15
55.86
55.76
53.65
53.59
53.47
53.42
45.68
45.58
28.19
28.07
26.56
O
OBz
O
O
O
O
S
OMe
O
P
MeO
NHBn
33
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

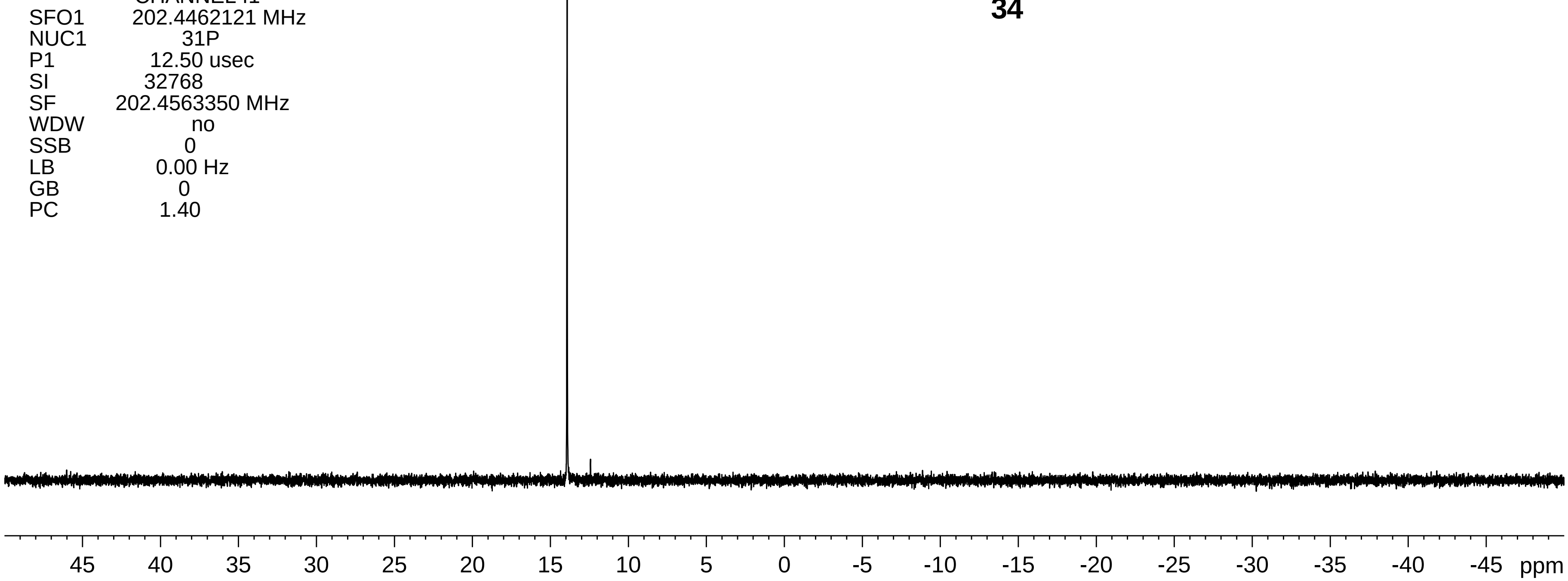

10.7385
9.7683
Current Data Parameters
NAME vd-772 P31
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150430
Time 21.30
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 36
DS 0
SWH 104166.664 Hz
FIDRES 1.589457 Hz
AQ 0.3146228 sec
RG 2050
DW 4.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== 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 13.70 dB
PL13 16.70 dB
PL2W 16.12334061 W
PL12W 0.43396533 W
PL13W 0.21749784 W
SFO2 400.1320007 MHz
F2 - Processing parameters
SI 32768
SF 161.9755127 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
O
OBz
O
O
O
O
OMe
P
MeO
NHBn
33
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
ppm

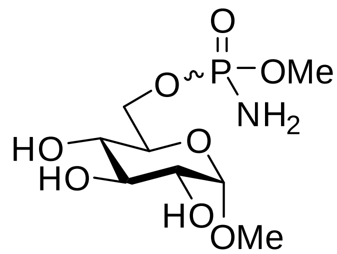

4.6772
4.6703
4.2547
4.2513
4.2423
4.2387
4.2327
4.2291
4.2203
4.2169
4.1622
4.1583
4.1484
4.1387
4.1335
4.1297
4.1265
4.1164
4.1113
3.7290
3.7259
3.7063
3.7032
3.6897
3.6796
3.6689
3.6371
3.6186
3.6000
3.4082
3.3992
3.3867
3.3793
3.3485
3.3412
3.3270
3.3214
3.3096
3.3068
3.2897
NAME SL-84
EXPNO 1
PROCNO 1
Date_ 20150610
Time 11.48
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT MeOD
NS 16
DS 2
SWH 5252.101 Hz
FIDRES 0.160281 Hz
AQ 3.1195636 sec
RG 200.35
DW 95.200 usec
DE 14.57 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1325007 MHz
NUC1 1H
P1 15.50 usec
SI 16384
SF 500.1300092 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
O
P
OMe
NH2
O
HO
HO
O
HO
OMe
34
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
2.00
2.04
2.04
6.08
2.14
2.36
8.22
2.27


101.33
74.99
73.45
72.06
72.03
72.00
71.33
71.26
66.89
66.82
66.78
55.66
53.76
53.72
49.51
49.34
49.17
49.00
48.83
48.66
48.49
NAME SL-84
EXPNO 6
PROCNO 1
Date_ 20150610
Time 11.55
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg60
TD 32768
SOLVENT MeOD
NS 903
SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 0.5243380 sec
RG 200.35
DW 16.000 usec
DE 6.50 usec
TE 298.3 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7722511 MHz
NUC1 13C
P1 10.50 usec
SI 32768
SF 125.7576123 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
O
O
P
OMe
NH2
HO
HO
O
HO
OMe
34
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

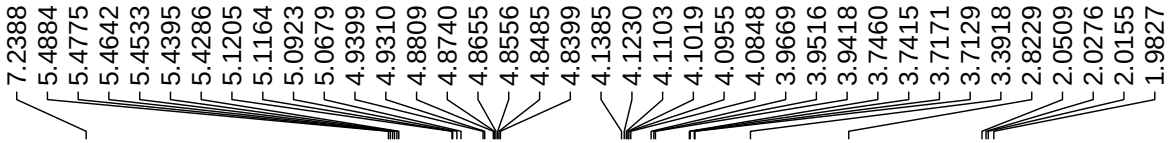
NAME SL-84
EXPNO 9
PROCNO 1
Date_ 20150610
Time 11.52
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 16
DS 4
SWH 40760.871 Hz
FIDRES 0.621962 Hz
AQ 0.8039582 sec
RG 200.35
DW 12.267 usec
DE 6.50 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 202.4462121 MHz
NUC1 31P
P1 12.50 usec
SI 32768
SF 202.4563350 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40





34

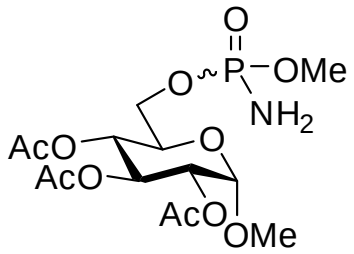


Current Data Parameters
NAME GPM-339
EXPNO 1
PROCNO 1

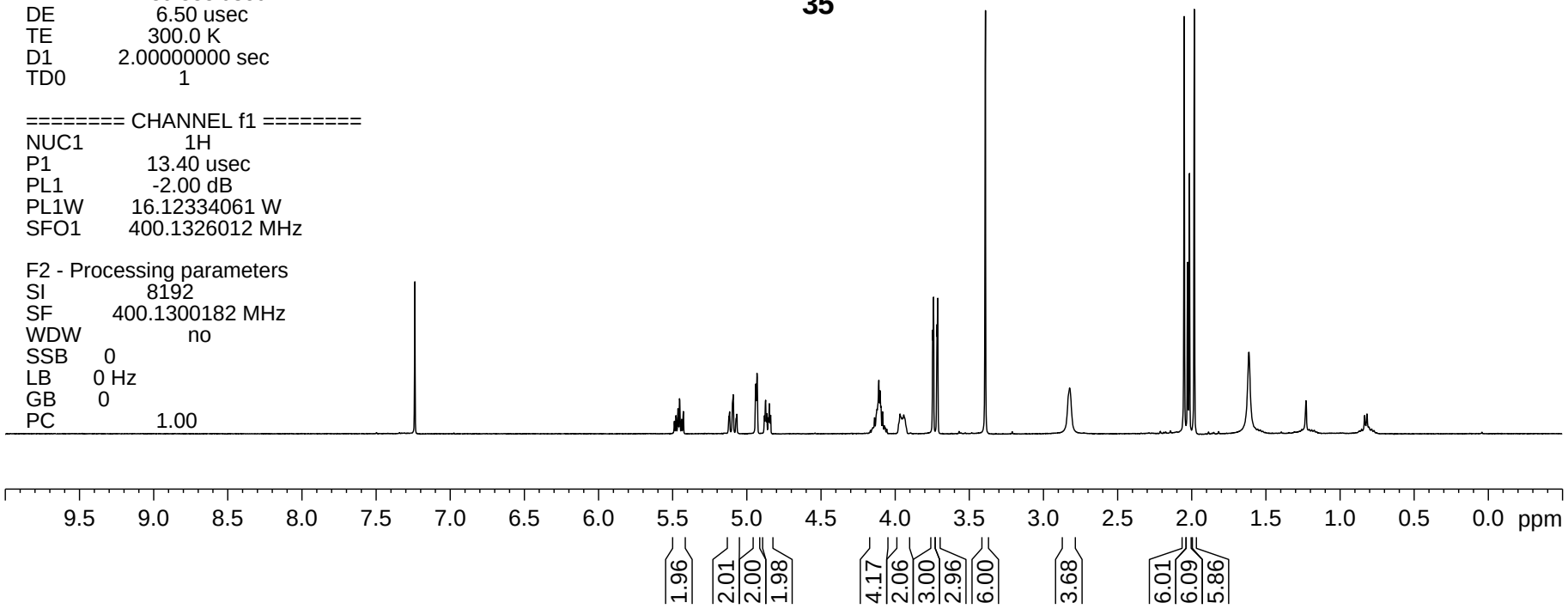
F2 - Acquisition Parameters
Date_ 20150605
Time 17.33
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 5597.015 Hz
FIDRES 0.341615 Hz
AQ 1.4636873 sec
RG 322
DW 89.333 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

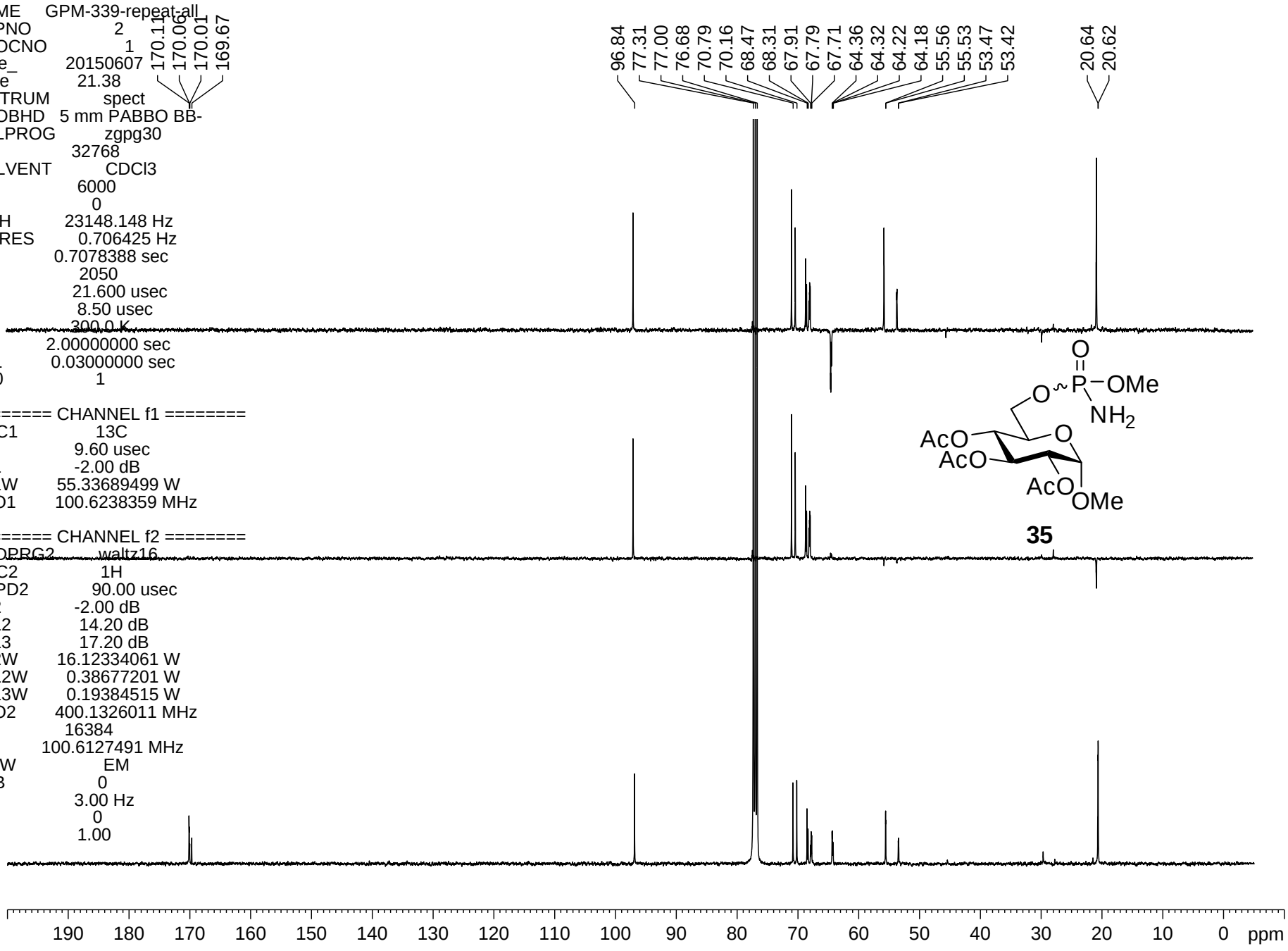
======== CHANNEL f1 ========
NUC1 1H
P1 13.40 usec
PL1 -2.00 dB
PL1W 16.12334061 W
SFO1 400.1326012 MHz

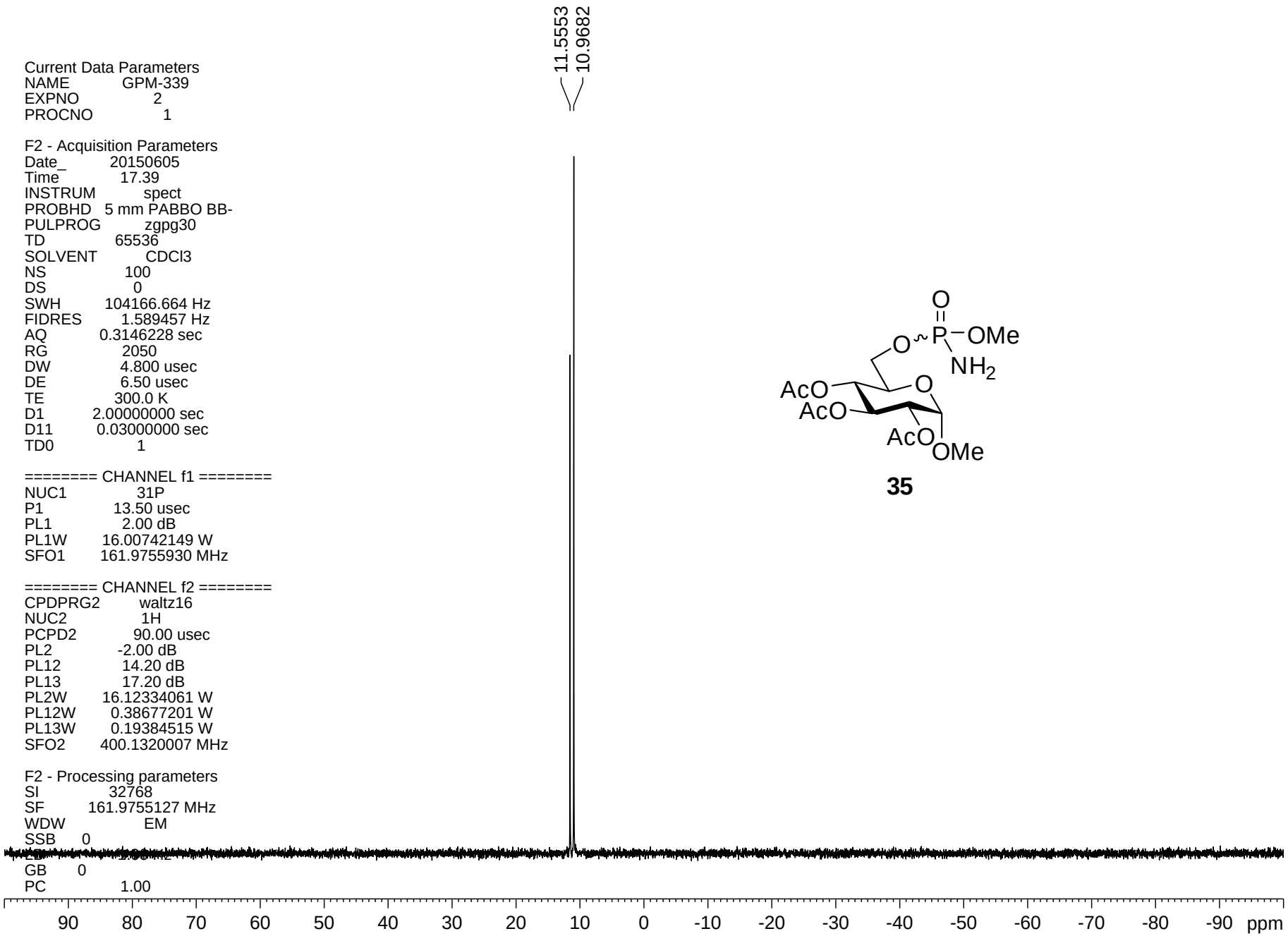
F2 - Processing parameters
SI 8192
SF 400.1300182 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00



35

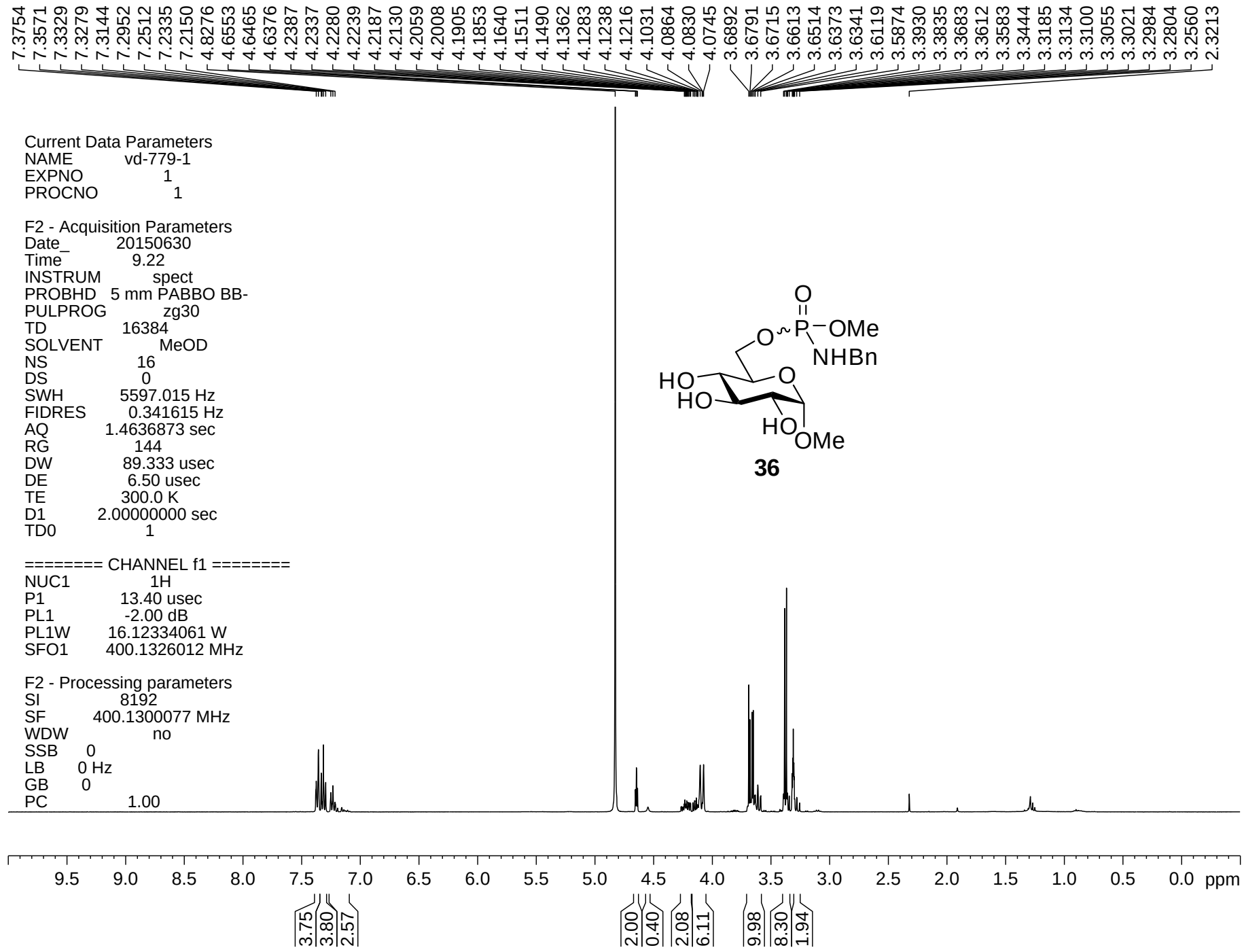


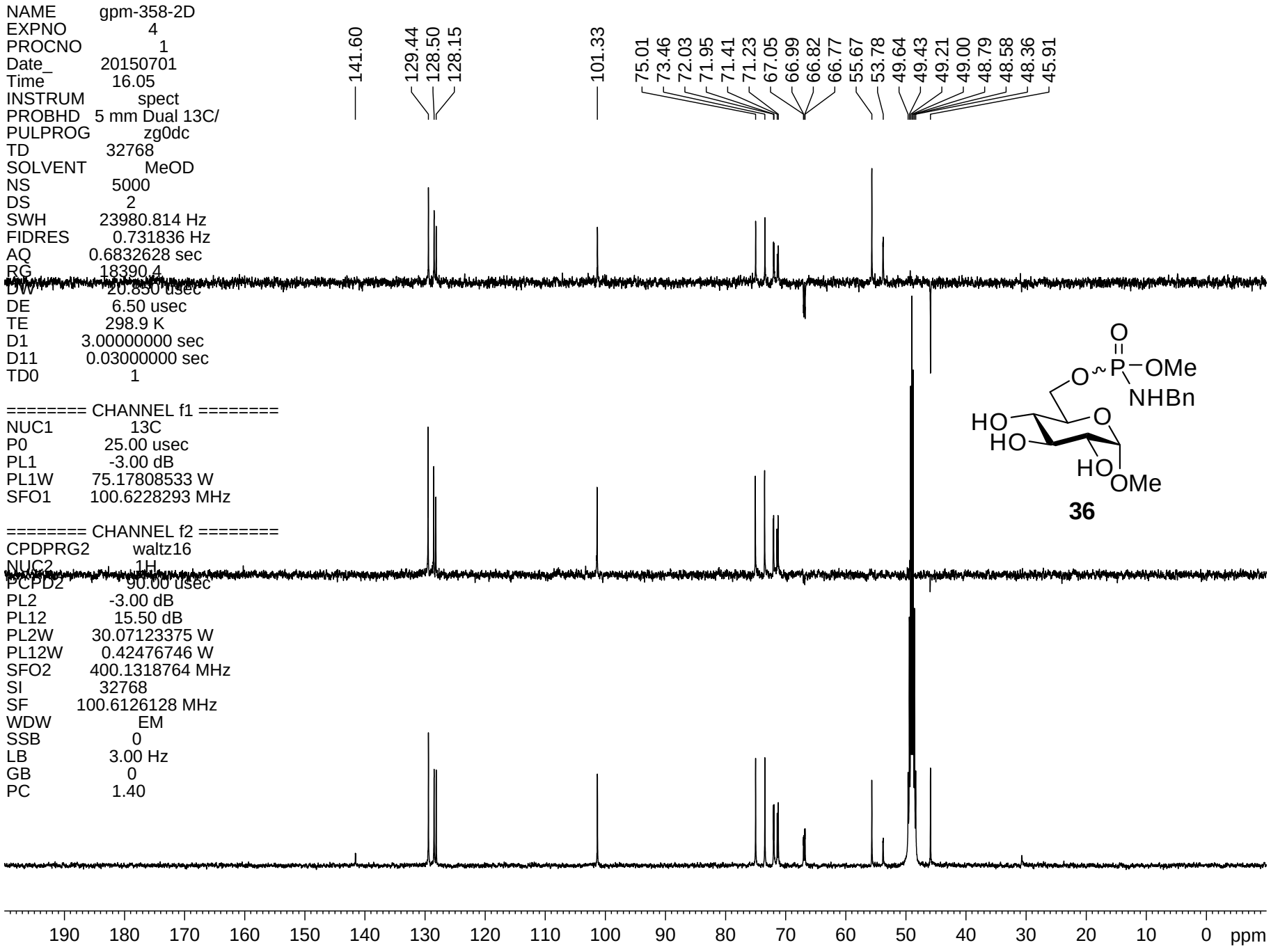

NAME GPM-339-repeat-all
EXPNO 2
PROCNO 1
Date_ 20150607
Time 21.38
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 6000
DS 0
SWH 23148.148 Hz
FIDRES 0.706425 Hz
AQ 0.7078388 sec
RG 2050
DW 21.600 usec
DE 8.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.60 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 14.20 dB
PL13 17.20 dB
PL2W 16.12334061 W
PL12W 0.38677201 W
PL13W 0.19384515 W
SFO2 400.1326011 MHz
SI 16384
SF 100.6127491 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
170.11
170.06
170.01
169.67
96.84
77.31
77.00
76.68
70.79
70.16
68.47
68.31
67.91
67.79
67.71
64.36
64.32
64.22
64.18
55.56
55.53
53.47
53.42
20.64
20.62
O
O
P
OMe
NH2
AcO
AcO
AcO
OMe
35
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


11.5553
10.9682
Current Data Parameters
NAME GPM-339
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150605
Time 17.39
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 100
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
======== 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
F2 - Processing parameters
SI 32768
SF 161.9755127 MHz
WDW EM
SSB 0
GB 0
PC 1.00
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 ppm

O
O
P
OMe
NH2
AcO
AcO
O
AcO
OMe

35


7.3754 7.3571 7.3329 7.3279 7.3144 7.2952 7.2512 7.2335 7.2150 4.8276 4.6553 4.6465 4.6376 4.2387 4.2337 4.2280 4.2239 4.2187 4.2130 4.2059 4.2008 4.1905 4.1853 4.1640 4.1511 4.1490 4.1362 4.1283 4.1238 4.1216 4.1031 4.0864 4.0830 4.0745 3.6892 3.6791 3.6715 3.6613 3.6514 3.6373 3.6341 3.6119 3.5874 3.3930 3.3835 3.3683 3.3612 3.3583 3.3444 3.3185 3.3134 3.3100 3.3055 3.3021 3.2984 3.2804 3.2560 2.3213
Current Data Parameters
NAME vd-779-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150630
Time 9.22
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT MeOD
NS 16
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 400.1300077 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
O
P
OMe
NHBn
O
HO
HO
O
HO
OMe
36
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
3.75 3.80 2.57
2.00 0.40 2.08 6.11 9.98 8.30 1.94


NAME gpm-358-2D
EXPNO 4
PROCNO 1
Date_ 20150701
Time 16.05
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg0dc
TD 32768
SOLVENT MeOD
NS 5000
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 298.9 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P0 25.00 usec
PL1 -3.00 dB
PL1W 75.17808533 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 15.50 dB
PL2W 30.07123375 W
PL12W 0.42476746 W
SFO2 400.1318764 MHz
SI 32768
SF 100.6126128 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
141.60
129.44
128.50
128.15
101.33
75.01
73.46
72.03
71.95
71.41
71.23
67.05
66.99
66.82
66.77
55.67
53.78
49.64
49.43
49.21
49.00
48.79
48.58
48.36
45.91
O
P
OMe
NHBn
HO
HO
HO
OMe
36
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

12.1944

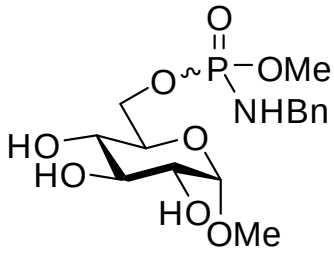
Current Data Parameters
NAME vd-779-1
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150630
Time 9.25
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 24
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

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

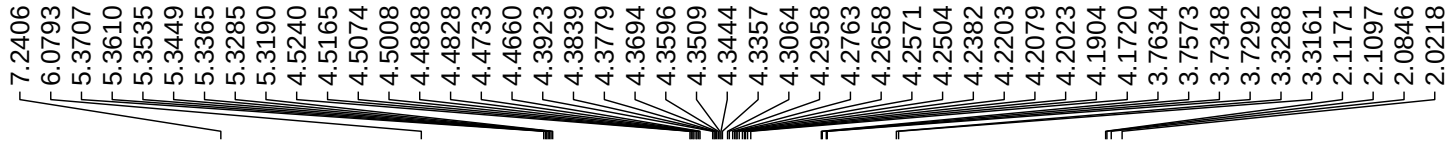
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.00 dB
PL12 13.70 dB
PL13 16.70 dB
PL2W 16.12334061 W
PL12W 0.43396533 W
PL13W 0.21749784 W
SFO2 400.1320007 MHz

F2 - Processing parameters
SI 32768
SF 161.9755127 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00



36

90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 ppm

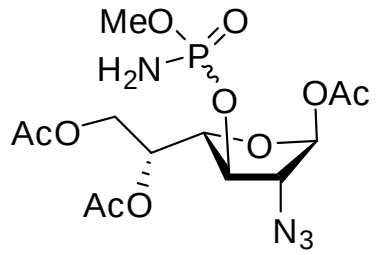


Current Data Parameters
NAME vd-775 All
EXPNO 1
PROCNO 1

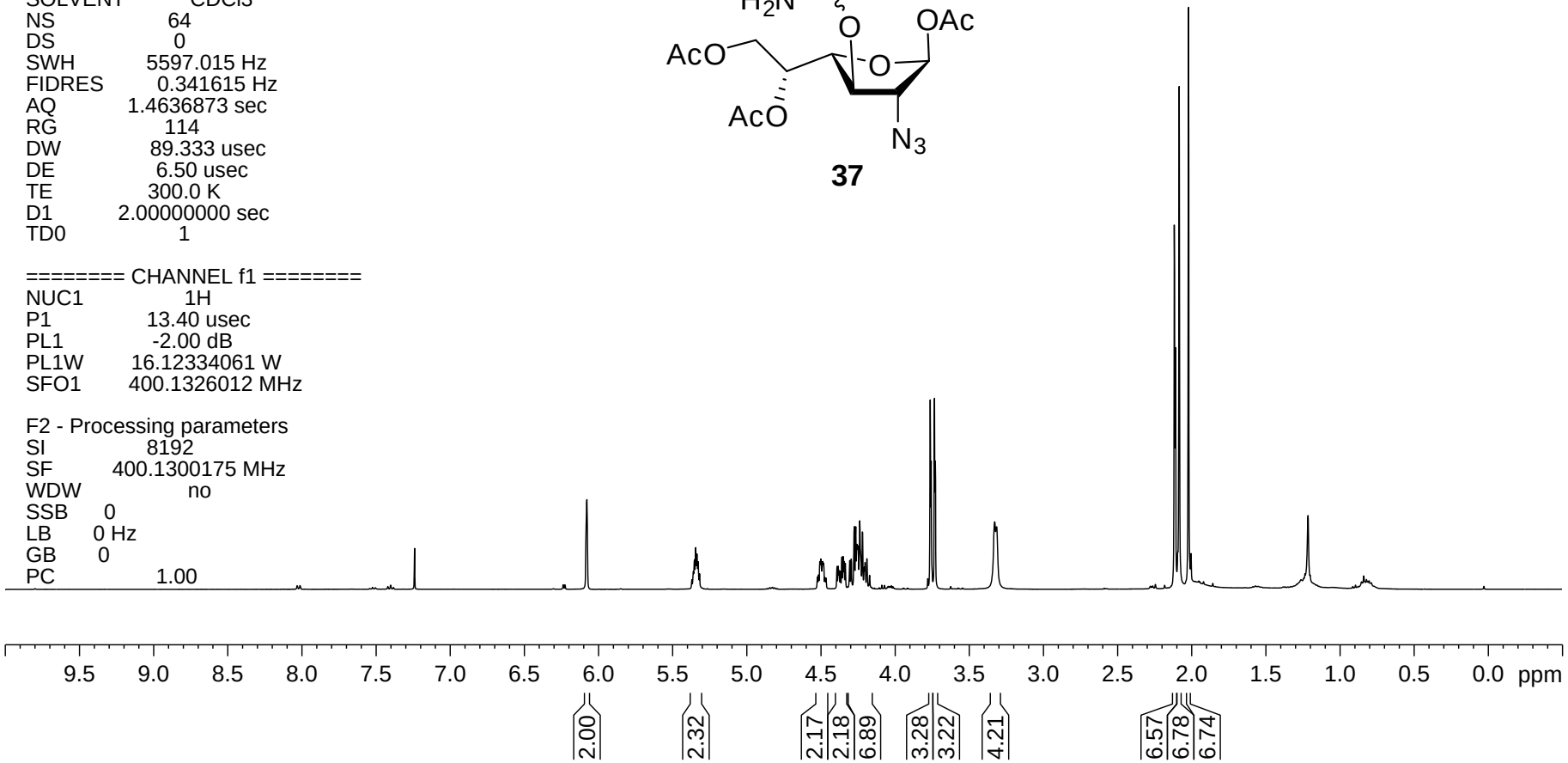
F2 - Acquisition Parameters
Date_ 20150604
Time 21.46
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 64
DS 0
SWH 5597.015 Hz
FIDRES 0.341615 Hz
AQ 1.4636873 sec
RG 114
DW 89.333 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

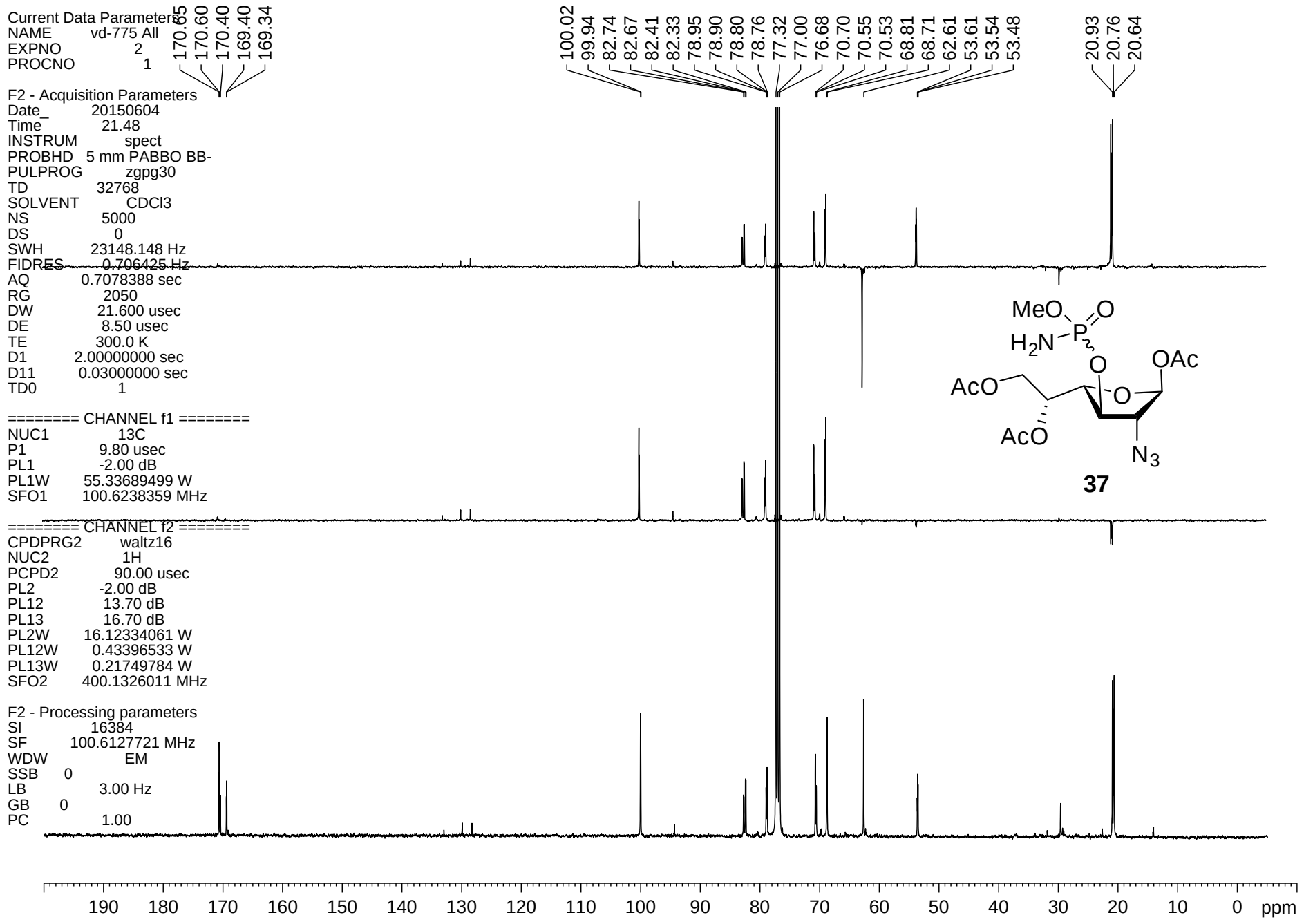
======== CHANNEL f1 ========
NUC1 1H
P1 13.40 usec
PL1 -2.00 dB
PL1W 16.12334061 W
SFO1 400.1326012 MHz

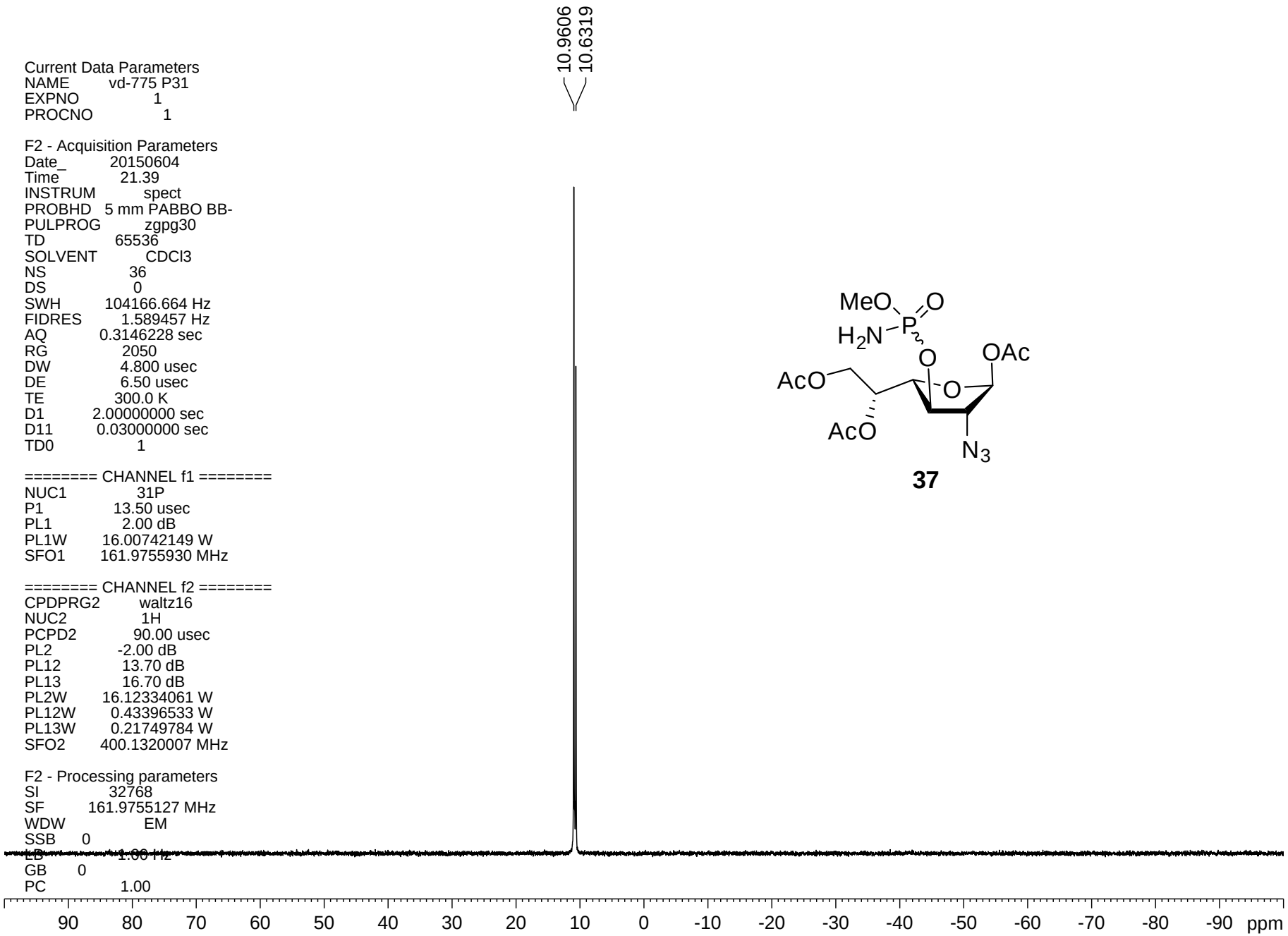
F2 - Processing parameters
SI 8192
SF 400.1300175 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

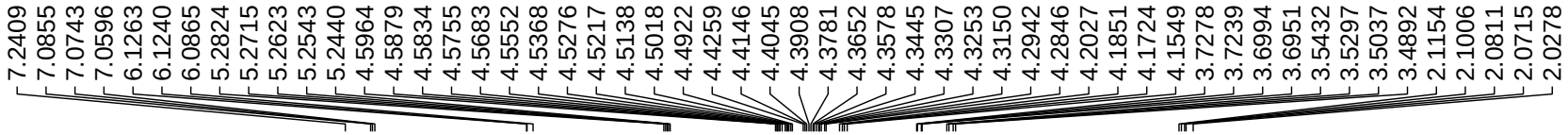


37



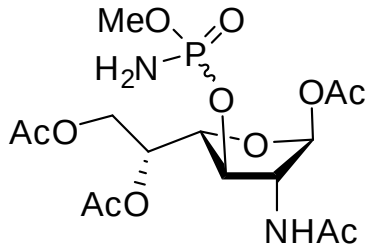

Current Data Parameters
NAME vd-775 All
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150604
Time 21.48
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 5000
DS 0
SWH 23148.148 Hz
FIDRES 0.706425 Hz
AQ 0.7078388 sec
RG 2050
DW 21.600 usec
DE 8.50 usec
TE 300.0 K
D1 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 100.6127721 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
170.85
170.60
170.40
169.40
169.34
100.02
99.94
82.74
82.67
82.41
82.33
78.95
78.90
78.80
78.76
77.32
77.00
76.68
70.70
70.55
70.53
68.81
68.71
62.61
53.61
53.54
53.48
20.93
20.76
20.64
MeO
O
H2N
P
O
OAc
AcO
O
AcO
N3
37
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


10.9606
10.6319
Current Data Parameters
NAME vd-775 P31
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150604
Time 21.39
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 36
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
======== 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 13.70 dB
PL13 16.70 dB
PL2W 16.12334061 W
PL12W 0.43396533 W
PL13W 0.21749784 W
SFO2 400.1320007 MHz
F2 - Processing parameters
SI 32768
SF 161.9755127 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
MeO
O
H2N
P
O
OAc
AcO
O
AcO
N3
37
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 ppm



Current Data Parameters
NAME vd-776 NHAc all
EXPNO 1
PROCNO 1

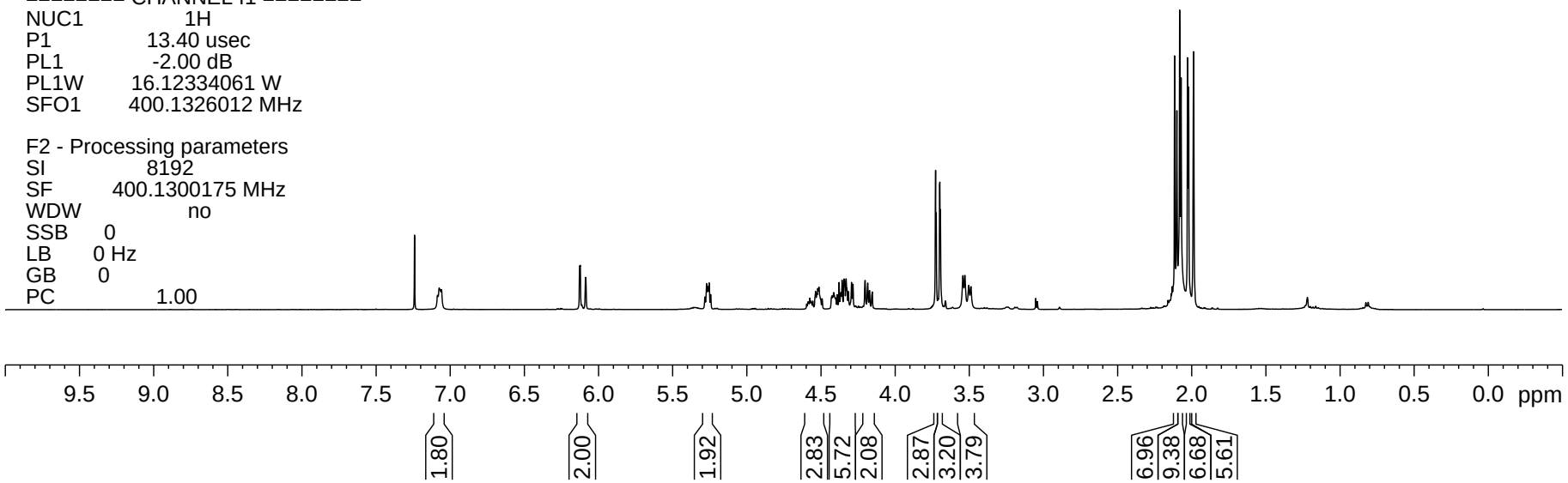
F2 - Acquisition Parameters
Date_ 20150608
Time 21.32
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 64
DS 0
SWH 5597.015 Hz
FIDRES 0.341615 Hz
AQ 1.4636873 sec
RG 144
DW 89.333 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

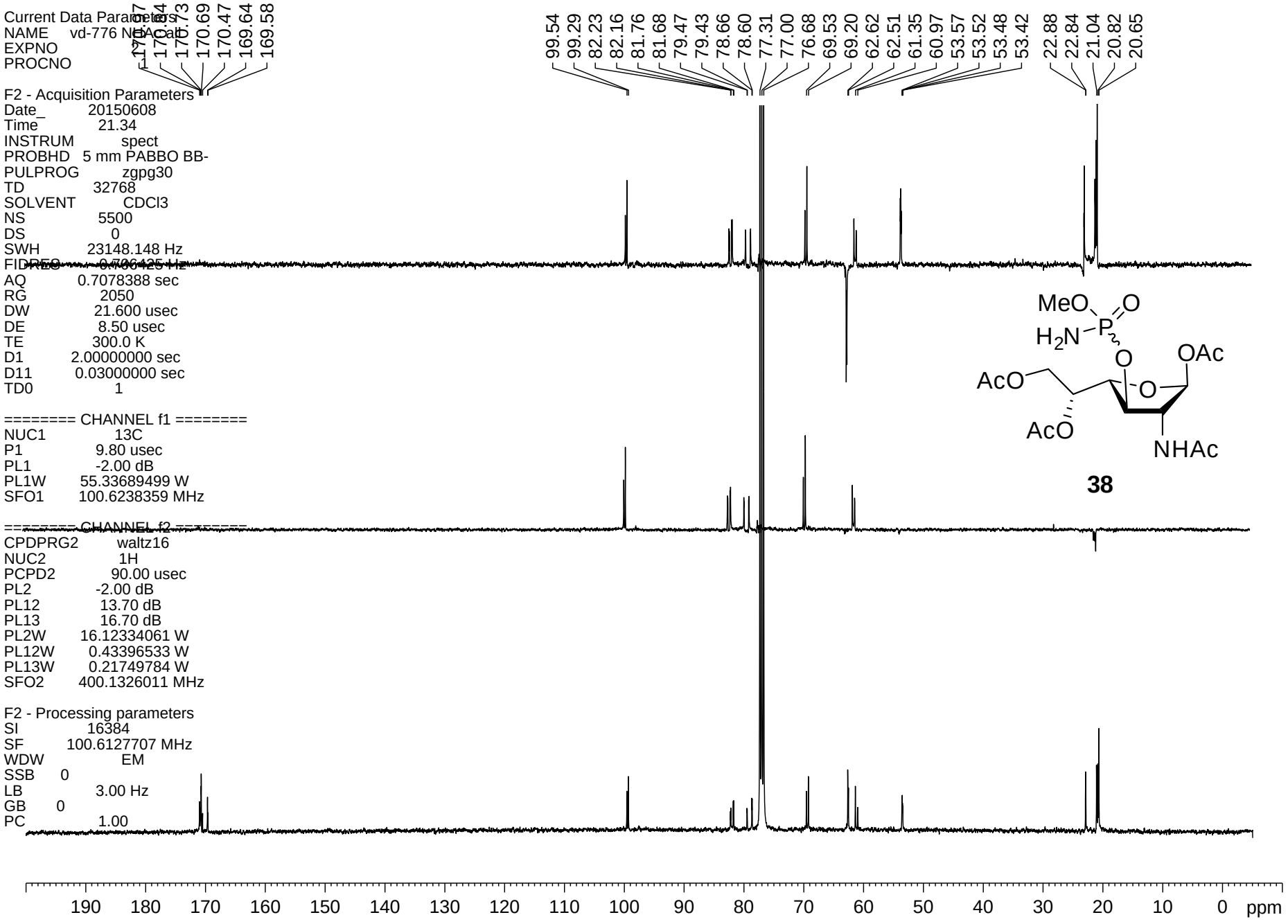


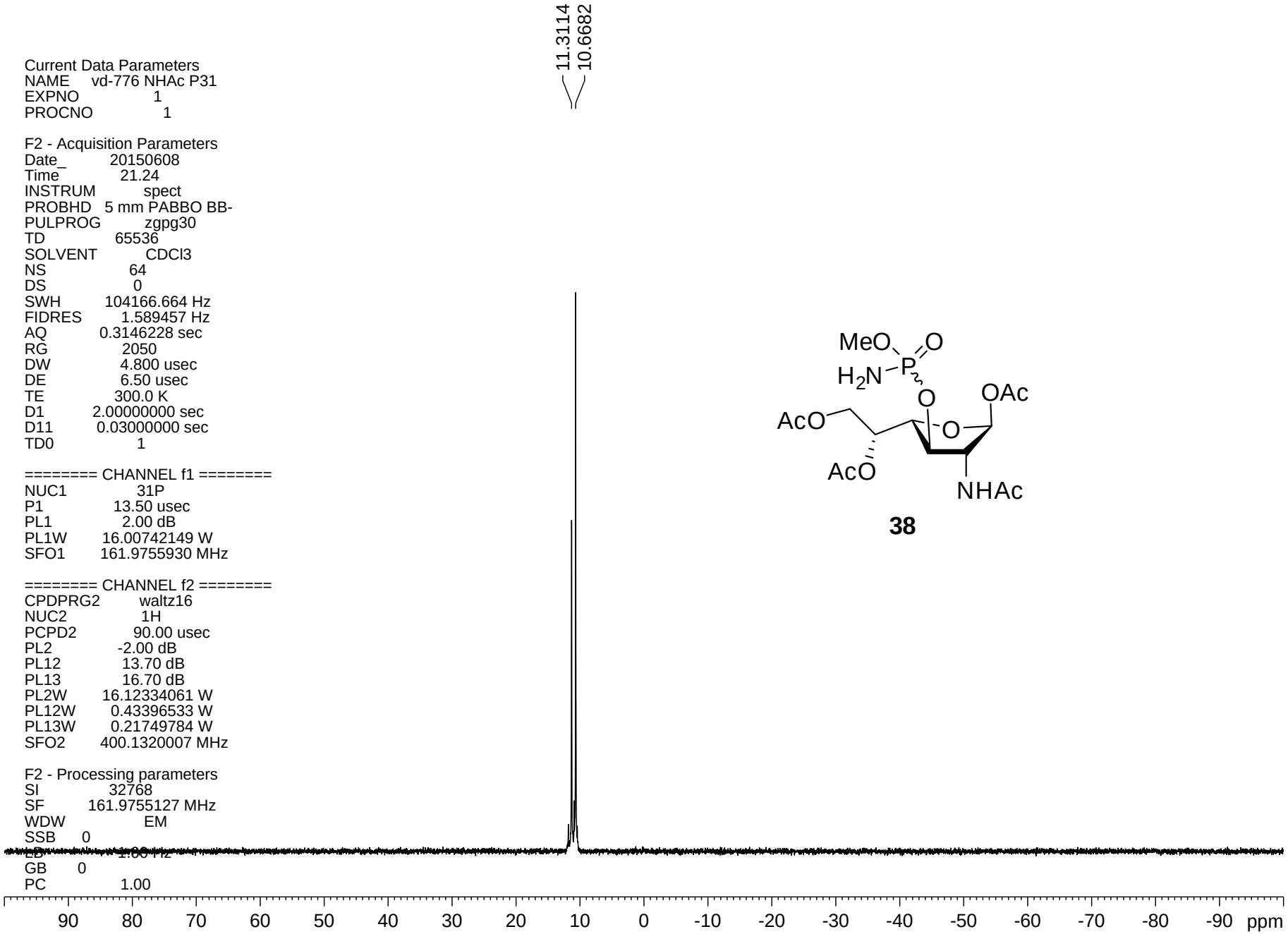
38

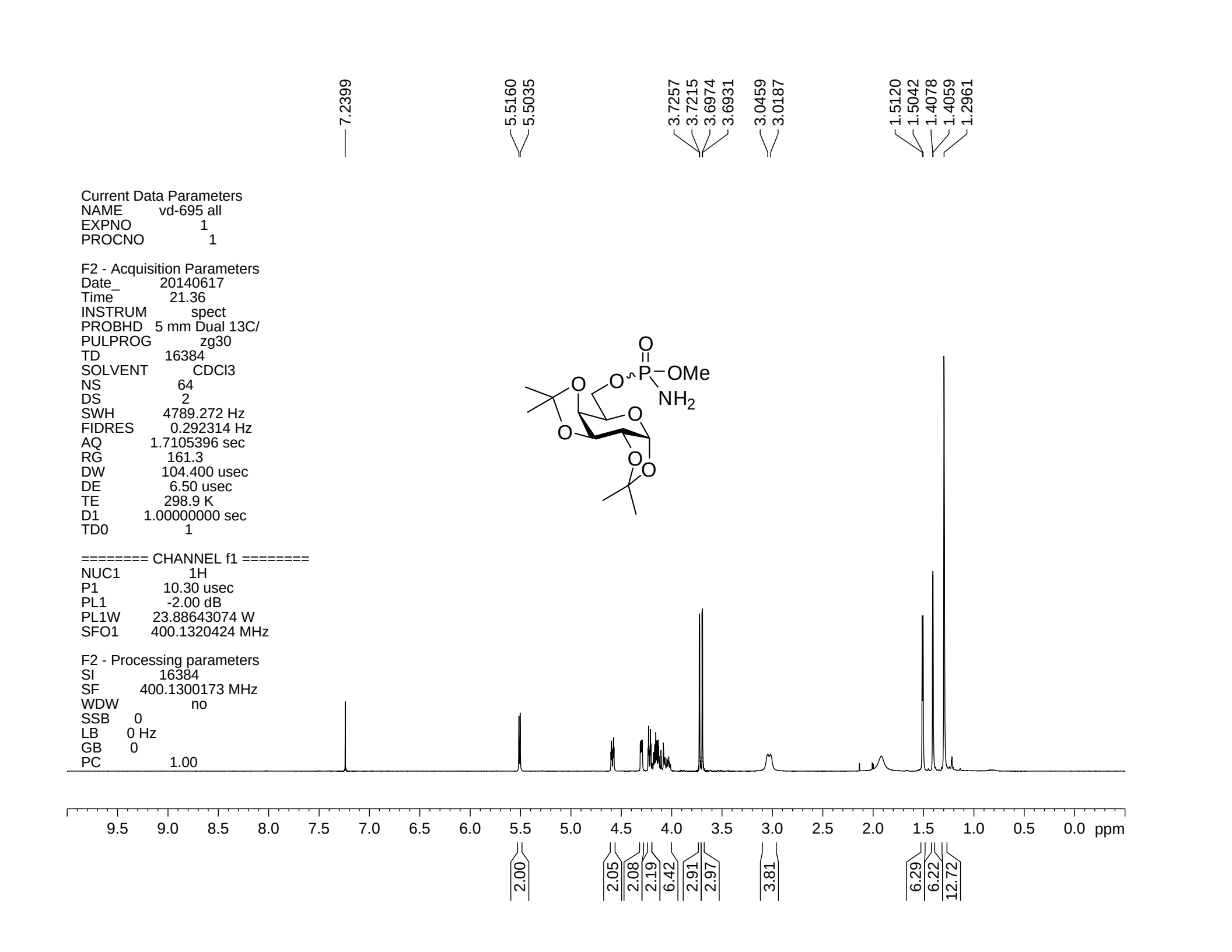
======== CHANNEL f1 ========
NUC1 1H
P1 13.40 usec
PL1 -2.00 dB
PL1W 16.12334061 W
SFO1 400.1326012 MHz

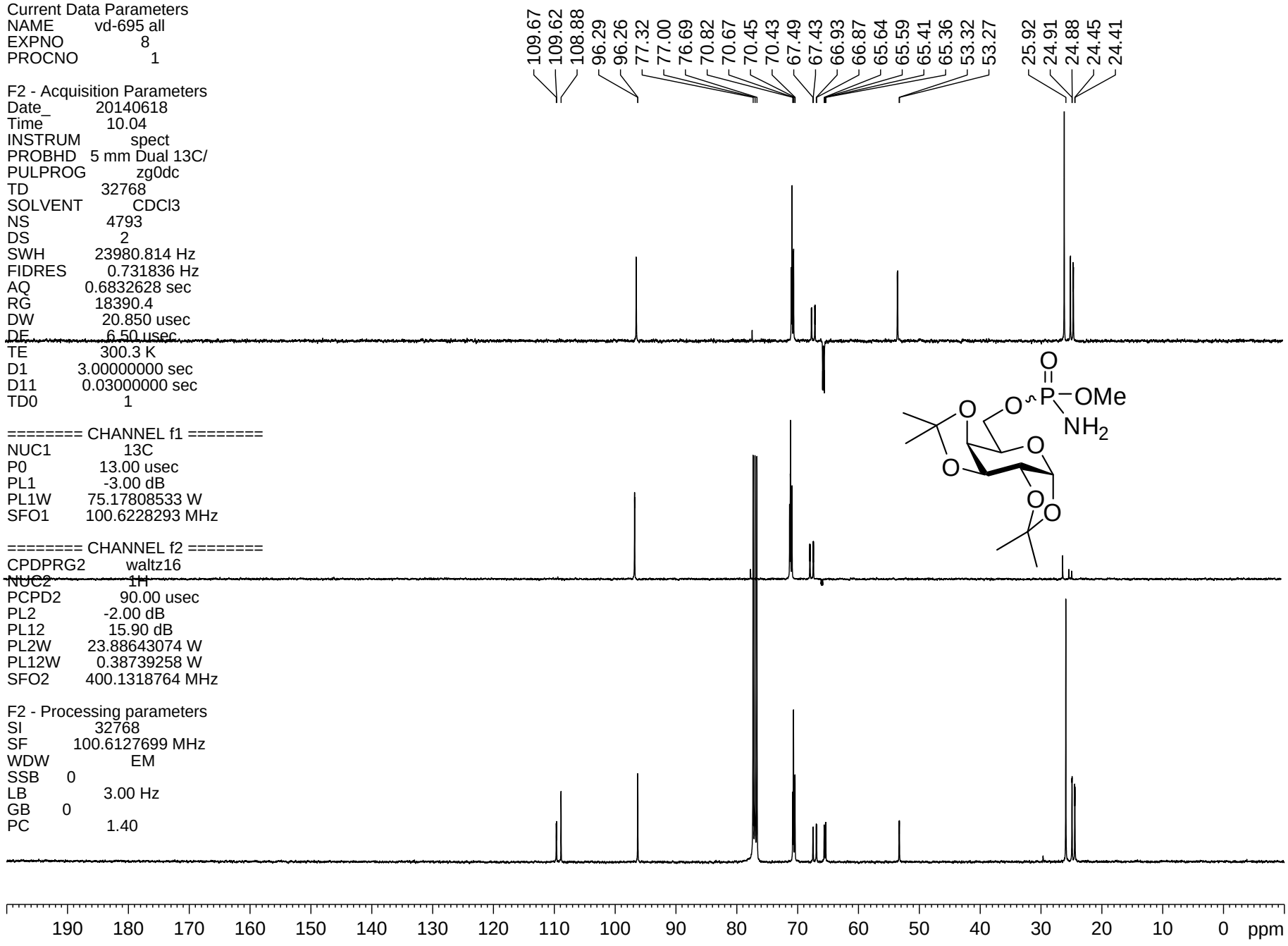
F2 - Processing parameters
SI 8192
SF 400.1300175 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

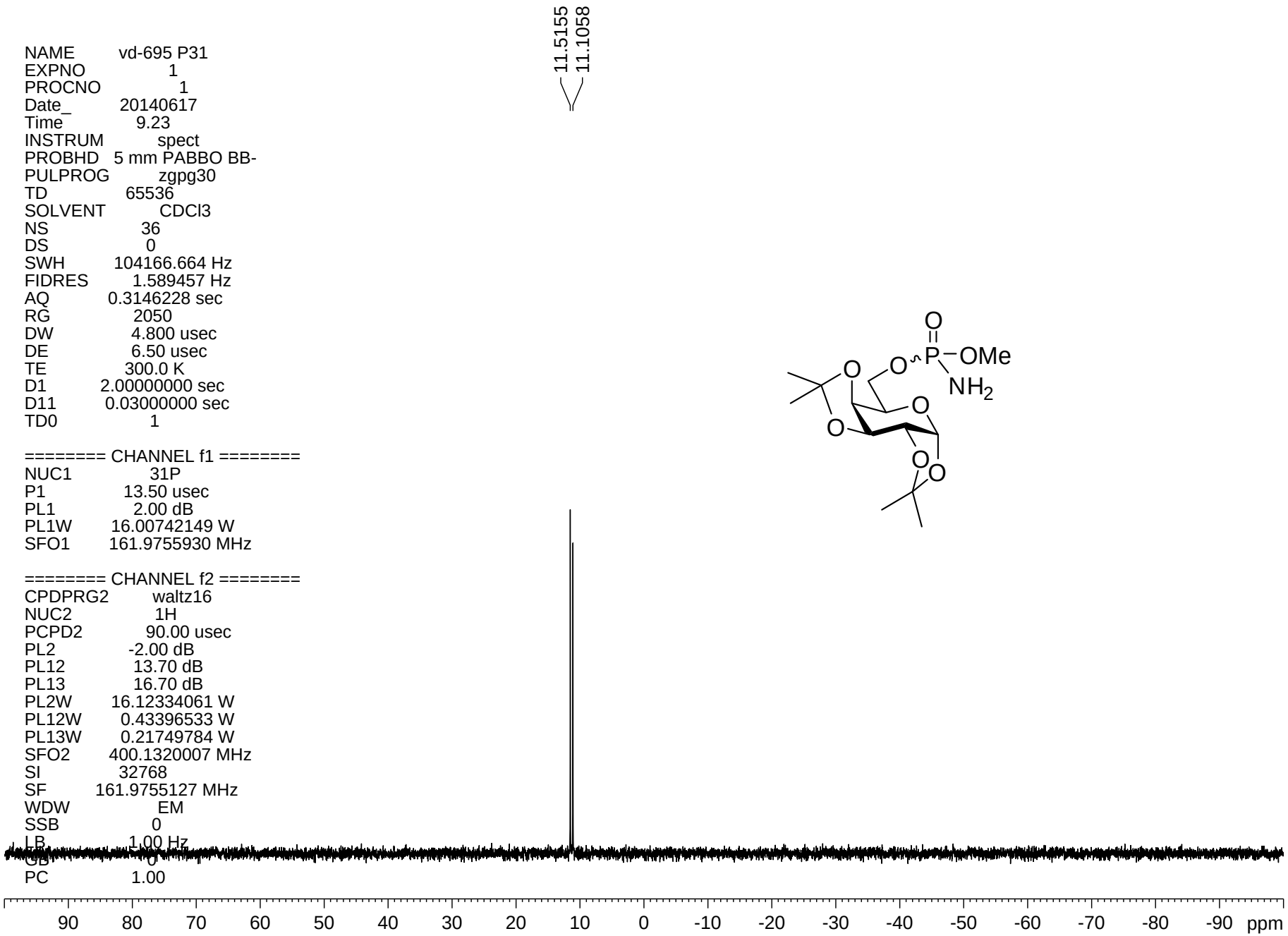



Current Data Parameters
NAME vd-776 NHAc.cab
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150608
Time 21.34
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 5500
DS 0
SWH 23148.148 Hz
FIDRES 0.706425 Hz
AQ 0.7078388 sec
RG 2050
DW 21.600 usec
DE 8.50 usec
TE 300.0 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 100.6127707 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
170.97
170.64
170.73
170.69
170.47
169.64
169.58
99.54
99.29
82.23
82.16
81.76
81.68
79.47
79.43
78.66
78.60
77.31
77.00
76.68
69.53
69.20
62.62
62.51
61.35
60.97
53.57
53.52
53.48
53.42
22.88
22.84
21.04
20.82
20.65
MeO
O
H2N
P
O
OAc
AcO
O
AcO
NHAc
38
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


11.3114
10.6682
Current Data Parameters
NAME vd-776 NHAc P31
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150608
Time 21.24
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
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
======== 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 13.70 dB
PL13 16.70 dB
PL2W 16.12334061 W
PL12W 0.43396533 W
PL13W 0.21749784 W
SFO2 400.1320007 MHz
F2 - Processing parameters
SI 32768
SF 161.9755127 MHz
WDW EM
SSB 0
GB 0
PC 1.00
MeO
O
H2N
P
O
OAc
AcO
O
AcO
NHAc
38
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 ppm


7.2399
5.5160
5.5035
3.7257
3.7215
3.6974
3.6931
3.0459
3.0187
1.5120
1.5042
1.4078
1.4059
1.2961
Current Data Parameters
NAME vd-695 all
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140617
Time 21.36
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 64
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7105396 sec
RG 161.3
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
F2 - Processing parameters
SI 16384
SF 400.1300173 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00
O
P
OMe
NH2
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
2.00
2.05
2.08
2.19
6.42
2.91
2.97
3.81
6.29
6.22
12.72


Current Data Parameters
NAME vd-695 all
EXPNO 8
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140618
Time 10.04
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg0dc
TD 32768
SOLVENT CDCl3
NS 4793
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 300.3 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 100.6127699 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
109.67
109.62
108.88
96.29
96.26
77.32
77.00
76.69
70.82
70.67
70.45
70.43
67.49
67.43
66.93
66.87
65.64
65.59
65.41
65.36
53.32
53.27
25.92
24.91
24.88
24.45
24.41
O
O
P
OMe
NH2
O
O
O
O
O
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


11.5155
11.1058
NAME vd-695 P31
EXPNO 1
PROCNO 1
Date_ 20140617
Time 9.23
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 36
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
======== 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 13.70 dB
PL13 16.70 dB
PL2W 16.12334061 W
PL12W 0.43396533 W
PL13W 0.21749784 W
SFO2 400.1320007 MHz
SI 32768
SF 161.9755127 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
O
P
OMe
NH2
O
O
O
O
O
O
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 ppm