Supplementary Material (ESI) for Chemical Communications

Supplementary Information

Iodine catalyzed one-pot diamination of glycals with chloramine-T: a new approach to 2-amino-β-glycosylamines for applications in *N*-glycopeptide synthesis

Vipin Kumar and Namakkal G. Ramesh*
Department of Chemistry, Indian Institute of Technology - Delhi, Hauz Khas, New Delhi, India 110016.
E-mail: ramesh@chemistry.iitd.ac.in

General consideration:

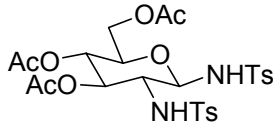
All solvents were purified using standard procedures. Chloramine-T purchased from Aldrich or Fluka Chemicals only was used for consistency of results. Thin-layer chromatography (TLC) was performed on Merck silica gel pre-coated on aluminium plates. Flash column chromatography was performed on 230-400 mesh silica gel. Optical rotations were recorded on an Autopol II or Autopol V (Rudolph Research Flanders, New Jersey) instrument. All the rotations were measured at 589 nm (sodium D' line). Melting points of the compounds are uncorrected. IR spectra were taken within the range 4000-400 cm^{-1} as KBr pellets on a Nicolet (Madison, USA) FT-IR spectrophotometer (Model Protege 460). All the ^{1}H and ^{13}C NMR spectra were recorded on a 300 MHz Bruker Spectrospin DPX FT-NMR. Chemical shifts are reported as δ values (ppm) relative to internal standard Me_4Si. Elemental analyses were performed on a Perkin Elmer 2400 series II analyzer. Mass spectra were recorded using Waters Micro Mass Q-TOF instrument.

General procedure for disulfonamidation of Glycals:

To a 0 °C stirred suspension of glycal (1 equiv.), chloramine-T (2.3 equiv.) in acetonitrile taken in a dried 100 mL round-bottomed flask, was added catalytic amount of iodine (15 mol%) and the reaction mixture was allowed to stir at 0°C until the reaction was complete (as indicated by tlc). The reaction mixture was diluted with $CHCl_3$ and stirred for additional 5 min. It was then transferred into a separating funnel containing aq.

sodium thiosulfate solution and shaken vigorously. The organic layer was separated and the remaining aqueous layer was washed with more amount of $CHCl_3$. The combined organic layer was then washed with brine solution and dried over anhydrous sodium sulfate and concentrated. The product was purified by flash chromatography.

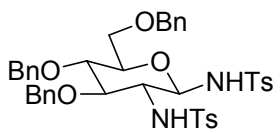
3,4,6-Tri-*O*-acetyl-1,2-dideoxy-1,2-di-(*p*-toluenesulfonamido)-β-D-glucopyranose 7.



7

7.76 g of **7** was obtained from the reaction of tri-*O*-acetyl-D-glucal **6** (5.00 g, 18.38 mmol) with chloramine-T (11.91 g, 42.27 mmol) and iodine (0.701 g, 2.76 mmol) in CH_3CN (50 mL) in 14 h as per the general procedure. Flash chromatography of the crude reaction mixture was performed with Hexane: Ethyl acetate (2:1). Yield 69%; white solid; mp 191-192 ºC (recrystallized from hot benzene); $[\alpha]_D^{28}$ +27.2 (*c* 1.12 in $CHCl_3$); Anal. Calcd. for $C_{26}H_{32}N_2O_{11}S_2$: Found: C, 50.97; H, 5.24; N, 4.78. requires C, 50.97; H, 5.26; N, 4.57; IR (KBr): ν 3286.1, 2927.4, 1744.9, 1599.1, 1456.8, 1368.1, 1329.4, 1242.2, 1162.4, 1090.8, 1071.9, 1044.3, 817.2, 672.5 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 7.82 (2 H, d, J = 8.2 Hz), 7.71 (2 H, d, J = 8.2 Hz), 7.32 (2 H, d, J = 8.0 Hz), 7.29 (2 H, d, J = 8.0 Hz), 6.35 (1 H, d, J = 7.4 Hz, N*H*, exchangeable with D_2O), 5.34 (1 H, d, J = 8.1 Hz, N*H*, exchangeable with D_2O), 4.97-4.87 (2 H, m), 4.67 (1 H, dd, J = 8.7, 7.8 Hz), 4.14 (1 H, dd, J = 12.3, 4.8 Hz), 3.96 (1 H, dd, J = 12.3, 1.8 Hz), 3.67-3.63 (1 H, m), 3.40 (1 H, q, J = 9.2 Hz), 2.43 (3 H, s), 2.42 (3 H, s), 2.06 (3 H, s), 1.97 (3 H, s), 1.48 (3 H, s); ^{13}C NMR (75 MHz, $CDCl_3$): δ 171.11 (s), 170.51 (s), 169.39 (s), 143.81 (s), 143.56 (s), 138.15 (s), 137.64 (s), 129.83 (d), 129.38 (d), 127.23 (d), 127.08 (d), 83.59 (d), 72.88 (2 x d), 68.26 (d), 61.72 (t), 56.64 (d), 21.44 (q), 20.61 (q), 20.45 (q), 19.99 (q).

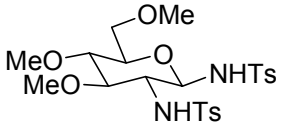
3,4,6-Tri-*O*-benzyl-1,2-dideoxy-1,2-di-(*p*-toluenesulfonamido)-β-D-glucopyranose 9.



9

5.18 g of **9** was obtained from the reaction of tri-*O*-benzyl-D-glucal **8** (5.00 g, 12.02 mmol) with chloramine-T (7.79 g, 27.65 mmol) and iodine (0.457 g, 1.80 mmol) in CH_3CN (40 mL) in 13 h as per the general procedure. Flash chromatography of the crude reaction mixture was performed with Hexane: Ethyl acetate (3:1). Yield 57%; white solid; mp 124 ºC (recrystallized from hot benzene); $[\alpha]_D^{28}$ +18.3 (*c* 2.29 in acetone); Anal. Calcd. for $C_{41}H_{44}N_2O_8S_2$: Found: C, 65.18; H, 5.79; N, 4.01. requires C, 65.08; H, 5.82; N, 3.70; IR (KBr): ν 3265.9, 2869.1, 2361.5, 1456.3, 1326.6, 1160.2, 1089.8, 1062.6, 698.1, 675.9 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 7.80 (2 H, d, *J* = 8.1 Hz), 7.68 (2 H, d, *J* = 8.1 Hz), 7.29-7.18 (13 H, m), 7.08 (2 H, d, *J* = 8.1 Hz), 7.04-6.98 (4 H, m), 6.21 (1 H, d, *J* = 7.8 Hz, N*H*, exchangeable with D_2O), 4.86 (1 H, d, *J* = 7.7 Hz, N*H*, exchangeable with D_2O), 4.63-4.58 (3 H, m), 4.52 (1 H, d, *J* = 11.4 Hz), 4.47-4.41 (2 H, m), 4.30 (1 H, d, *J* = 12.1 Hz), 3.59 (2 H, dd, *J* = 9.1, 8.7 Hz), 3.43-3.38 (3 H, m), 3.30 (1 H, ddd, *J* = 9.3, 8.7, 8.4 Hz), 2.36 (3 H, s), 2.30 (3 H, s); ^{13}C NMR (75 MHz, $CDCl_3$): δ 143.82 (s), 143.59 (s), 139.18 (s), 138.37 (s), 138.19 (s), 137.91 (s), 129.95 (d), 129.69 (d), 129.30 (d), 128.77 (d), 128.71 (d), 128.5 (d), 128.09 (d), 127.86 (d), 127.52 (d), 84.36 (d), 82.51 (d), 78.73 (d), 76.57 (d), 75.48 (t), 75.04 (t), 73.97 (t), 68.72 (t), 58.40 (d), 21.91 (q); HRMS (ESI): $[M+Na]^+$, Found: 779.2439, $C_{41}H_{44}N_2O_8S_2Na$ requires 779.2437.

1,2-Dideoxy-3,4,6-tri-*O*-methyl-1,2-di-(*p*-toluenesulfonamido)-β-D-glucopyranose 11.

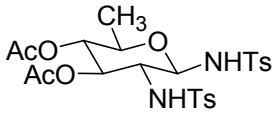


11

4.38 g of **11** was obtained from the reaction of tri-*O*-methyl-D-glucal **10** (2.60 g, 13.83 mmol) with chloramine-T (8.96 g, 31.81 mmol) and iodine (0.526 g, 2.07 mmol) in CH_3CN (40 mL) in 13 h as per the general procedure. Flash chromatography of the crude reaction mixture was performed with Hexane: Ethyl acetate (2:1). Yield 60%; white solid; mp 180-181 ºC (recrystallized from hot benzene); $[\alpha]_D^{28}$ +38.3 (*c* 1.62 in $CHCl_3$); Anal. Calcd. for $C_{23}H_{32}N_2O_8S_2$: Found: C, 52.08; H, 6.13; N, 5.34. requires C, 52.26; H, 6.10; N, 5.30; IR (KBr): ν 3303.2, 3282.5, 2986.8, 2934.3, 2837.2, 1458.4, 1330.6,

1314.8, 1159.4, 1090.7, 1068.1, 895.9, 813.7, 675.8 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 7.82 (2 H, d, J = 8.2 Hz), 7.76 (2 H, d, J = 8.2 Hz), 7.31-7.26 (4 H, m), 6.01 (1 H, d, J = 8.0 Hz, N*H*, exchangeable with D_2O), 5.11 (1 H, d, J = 8.0 Hz, N*H*, exchangeable with D_2O), 4.59 (1 H, t, J = 8.6 Hz), 3.45 (1 H, dd, J = 10.8, 3.0 Hz), 3.40 (3 H, s), 3.31 (1 H, dd, J = 10.8, 1.8 Hz), 3.23 (4 H, m, with one of the methyl protons superimposed), 3.15 (4 H, with one of the methyl protons superimposed), 3.11-2.98 (2 H, m), 2.42 (3 H, s), 2.41 (3 H, s); ^{13}C NMR (75 MHz, $CDCl_3$): δ 143.18 (s), 143.11 (s), 138.53 (s), 137.65 (s), 129.28 (d), 129.13 (d), 127.39 (d), 127.16 (d), 84.26 (d), 83.48 (d), 79.38 (d), 75.62 (d), 70.20 (t), 60.45 (q), 59.87 (q), 58.97 (q), 57.75 (d), 21.42 (q).

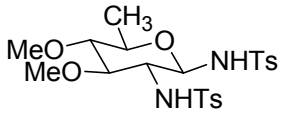
3,4-Di-*O*-acetyl-1,2-dideoxy-1,2-di-(*p*-toluenesulfonamido)-β-D-rhamnopyranose 13.



13

2.45 g of **13** was obtained from the reaction of di-*O*-acetyl-D-rhamnal **12** (1.50 g, 7.01 mmol) with chloramine-T (4.54 g, 16.12 mmol) and iodine (0.267 g, 1.05 mmol) in CH_3CN (20 mL) in 14 h as per the general procedure. Flash chromatography of the crude reaction mixture was performed with Hexane: Ethyl acetate (4:1). Yield 63%; white solid; mp 207 ºC (recrystallized from hot benzene); $[\alpha]_D^{28}$ +27.2 (*c* 1.80 in $CHCl_3$); Anal. Calcd. for $C_{24}H_{30}N_2O_9S_2$: Found: C, 52.06; H, 5.43; N, 4.64. requires C, 51.97; H, 5.45; N, 5.05; IR (KBr): ν 3276.9, 2929.4, 2361.7, 1746.1, 1456.3, 1337.3, 1330.0, 1244.0, 1218.8, 1160.9, 1089.8, 1045.5, 897.5, 815.8, 675.5 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 7.81 (2 H, d, J = 8.0 Hz), 7.70 (2 H, d, J = 8.0 Hz), 7.30 (4 H, m), 6.27 (1 H, d, J = 7.5 Hz, N*H*, exchangeable with D_2O), 5.00 (1 H, d, J = 7.0 Hz, N*H*, exchangeable with D_2O), 4.83 (1 H, dd, J = 10.0, 9.5 Hz), 4.66 (1 H, t, J = 9.4 Hz), 4.59 (1 H, m), 3.48 (1 H, dq, J = 9.6, 6.0 Hz), 3.35 (1 H, q, J = 9.3 Hz), 2.43 (3 H, s), 2.42 (3 H, s), 1.98 (3 H, s), 1.48 (3 H, s), 1.08 (3 H, d, J = 6.0 Hz); ^{13}C NMR (75 MHz, $CDCl_3$): δ 171.44 (s), 169.51(s), 143.98 (s), 143.55 (s), 138.15 (s), 137.41 (s), 129.91 (d), 129.41 (d), 127.30 (d), 127.18 (d), 83.61 (d), 72.99 (d), 72.88 (d), 71.34 (d), 57.01 (d), 21.51 (q), 21.45 (q), 20.56 (q), 20.05 (q), 17.15 (q).

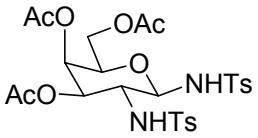
1,2-Dideoxy-3,4-di-*O*-methyl-1,2-di-(*p*-toluenesulfonamido)-β-D-rhamnopyranose 15.



15

1.32 g of **15** was obtained from the reaction of di-*O*-methyl-D-rhamnal **14** (0.700 g, 4.43 mmol) with chloramine-T (2.87 g, 10.19 mmol) and iodine (0.168 g, 0.66 mmol) in CH_3CN (10 mL) in 14 h as per the general procedure. Flash chromatography of the crude reaction mixture was performed with Hexane: Ethyl acetate (2:1). Yield 60%; white solid; mp 203 ºC (recrystallized from hot benzene); $[\alpha]_D^{28}$ +24.7 (*c* 1.13 in $CHCl_3$); Anal. Calcd. for $C_{22}H_{30}N_2O_7S_2$: Found: C, 52.91; H, 6.02; N, 5.96. requires C, 52.99; H, 6.06; N, 5.62; IR (KBr): ν 3265.4, 2988.2, 2964.7, 2927.8, 2882.1, 2833.8, 1598.2, 1459.3, 1328.1, 1158.9, 1086.0, 1042.1, 891.8, 815.4, 678.9 cm^{-1} ; ^1H NMR (300 MHz, $CDCl_3$): *δ* 7.81 (2 H, d, *J* = 8.3 Hz), 7.75 (2 H, d, *J* = 8.3 Hz), 7.31-7.26 (4 H, m), 6.01 (1 H, d, *J* = 7.8 Hz, N*H*, exchangeable with D_2O), 5.14 (1 H, d, *J* = 8.3 Hz, N*H*, exchangeable with D_2O), 4.59 (1 H, t, *J* = 8.4 Hz), 3.42 (3 H, s), 3.22 (1 H, dq, *J* = 9.3, 6.3 Hz), 3.13 (3 H, s), 3.09 (1 H, dd, *J* = 9.9, 8.7 Hz), 2.99 (1 H, dd, *J* = 9.6, 8.4 Hz), 2.64 (1 H, t, *J* = 8.8 Hz), 2.42 (3 H, s), 2.41 (3 H, s), 1.08 (3 H, d, *J* = 6.3 Hz); ^{13}C NMR (75 MHz, $CDCl_3$): *δ* 143.36 (s), 138.47 (s), 137.68 (s), 129.41 (d), 129.28 (d), 127.26 (2 x d), 85.86 (d), 84.40 (d), 83.27 (d), 72.56 (d), 60.51 (q), 60.29 (q), 58.12 (q), 21.52 (q), 17.40 (q).

3,4,6-Tri-*O*-acetyl-1,2-dideoxy-1,2-di-(*p*-toluenesulfonamido)-β-D-galactopyranose 17.

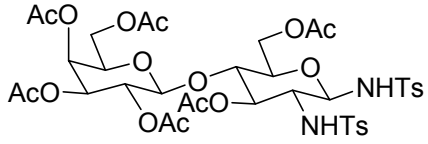


17

0.472 g of **17** was obtained from the reaction of tri-*O*-acetyl-D-galactal **16** (1.00 g, 3.68 mmol) with chloramine-T (2.38 g, 8.44 mmol) and iodine (0.140 g, 0.55 mmol) in CH_3CN (10 mL) in 72 h as per the general procedure. Flash chromatography of the crude

reaction mixture was performed with Hexane: Ethyl acetate (2:1). 0.445 g (44.5%) of starting material was also recovered during the column chromatography. Isolated yield: 21% (38% based on recovered starting material); white solid; mp 112 ºC (recrystallized from hot benzene); $[\alpha]_D^{28}$ +27.3 (*c* 1.50 in $CHCl_3$); Anal. Calcd. for $C_{26}H_{32}N_2O_{11}S_2$: Found: C, 50.58; H, 5.25; N, 4.67. requires C, 50.97; H, 5.26; N, 4.57; IR (KBr): ν 3430.9, 2923.8, 1749.9, 1455.3, 1371.73, 1337.2, 1237.6, 1160.9, 1082.2, 1043.8, 667.45 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 7.83 (2 H, d, *J* = 8.0 Hz), 7.73 (2 H, d, *J* = 8.0 Hz), 7.29-7.26 (4 H, m), 6.31 (1 H, d, *J* = 7.4 Hz, N*H*, exchangeable with D_2O), 5.56 (1 H, t, *J* = 8.0 Hz, N*H*, exchangeable with D_2O), 5.28 (1 H, brs), 4.94-4.90 (1 H, m), 4.81 (1 H, brt), 3.99-3.92 (3 H, m), 3.61 (1 H, dq, *J* = 9.6, 8.8 Hz), 2.41 (6 H, s), 2.06 (3 H, s), 1.99 (3 H, s), 1.49 (3 H, s); ^{13}C NMR (75 MHz, $CDCl_3$): δ 170.36 (s), 169.88 (s), 143.67 (s), 138.29 (s), 137.74 (s), 129.55 (d), 129.37 (d), 127.18 (2 x d), 83.78 (d), 71.68 (d), 70.79 (d), 66.82 (d), 61.14 (t), 53.01 (d), 21.45 (q), 20.47 (2 x q), 20.02 (q); HRMS (ESI): $[M+Na]^+$ Found: 635.1349, $C_{26}H_{32}N_2O_{11}S_2Na$ requires 635.1345.

4-*O*-[2,3,4,6-Tetra-*O*-acetyl-(β-D-galactopyranosyl)]-3,6-di-*O*-acetyl-1,2-dideoxy-1,2-di-(*p*-toluenesulfonamido)-β-D-glucopyranose 19.

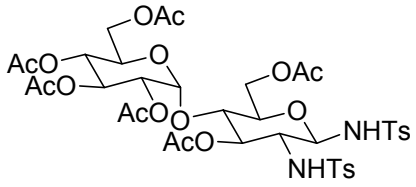


19

In this case 3 equiv. of chloramine-T and 20 mol% of iodine were used. 6.23 g of **19** was obtained from the reaction of hexa-*O*-acetyl lactal **18** (5.46 g, 9.75 mmol) with chloramine-T (8.24 g, 29.25 mmol) and iodine (0.495 g, 1.95 mmol) in CH_3CN (50 mL) in 96 h as per the general procedure. Flash chromatography of crude the reaction mixture was performed with Hexane: Ethyl acetate (1:1). Yield 71%; white solid; mp 107 ºC (recrystallized from hot benzene); $[\alpha]_D^{28}$ +17.7 (*c* 1.21 in $CHCl_3$); IR (KBr): ν 3478.9, 3279.1, 2927.4, 1751.1, 1456.4, 1371.2, 1338.3, 1226.8, 1161.6, 1048.0, 899.3, 815.7, 668.7 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 7.80 (2 H, d, *J* = 8.1), 7.71 (2 H, t, *J* = 8.1 Hz), 7.34 (2 H, t, *J* = 7.2 Hz), 7.30 (2 H, t, *J* = 8.5 Hz), 6.36 (1 H, d, *J* = 6.9 Hz, N*H*, exchangeable with D_2O), 5.32 (1 H, brs), 5.06-4.81 (4 H, m), 4.54 (1 H, dd, *J* = 8.4, 7.9

Hz), 4.37 (1 H, d, *J* = 7.8 Hz), 4.33 (1 H, m), 4.11-3.92 (3 H, m), 3.82-3.80 (1 H, m), 3.65-3.56 (2 H, m), 3.35 (1 H, q, *J* = 9.4 Hz), 2.44 (6 H, s), 2.11 (3 H, s), 2.10 (3 H, s), 2.04 (3 H, s), 2.03 (3 H, s), 1.95 (3 H, s), 1.57 (3 H, s); ^{13}C NMR (75 MHz, $CDCl_3$): δ 171.32 (s), 170.03 (2 x s), 169.05 (s), 144.07 (s), 143.57 (s), 137.34 (s), 129.87 (d), 129.44 (d), 127.24 (2 x d), 100.75 (d), 83.74 (d), 75.66 (d), 73.94 (d), 72.92 (d), 70.81 (d), 70.63 (d), 69.00 (d), 66.55 (d), 61.74 (t), 60.74 (t), 60.71 (t), 56.85 (d), 21.48 (q), 20.53 (q), 20.23 (q).

4-*O*-[2,3,4,6-Tetra-*O*-acetyl-(α-D-glucopyranosyl)]-3,6-di-*O*-acetyl-1,2-dideoxy-1,2-di-(*p*-toluenesulfonamido)-β-D-glucopyranose 21.

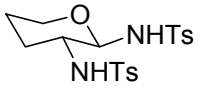


21

In this case 3 equiv. of chloramine-T and 20 mol% of iodine were used. 1.00 g of **21** was obtained from the reaction of hexa-*O*-acetyl-D-maltal **20** (1.00 g, 1.79 mmol) with chloramine-T (1.50 g, 5.34 mmol) and iodine (0.090 g, 0.36 mmol) in CH_3CN (6 mL) in 96 h as per the general. Flash chromatography of the crude reaction mixture was performed with Hexane: Ethyl acetate (1:1). Yield 65%; white solid; mp 168 ºC (recrystallized from hot benzene); $[\alpha]_D^{28}$ +68.1 (*c* 0.52 in $CHCl_3$); IR (KBr): ν 3297.1, 2925.8, 1749.5, 1456.8, 1372.1, 1332.9, 1234.4, 1162.7, 1088.1, 1046.5, 896.5, 675.2 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.80 (2 H, d, *J* = 7.8), 7.72 (2 H, t, *J* = 7.8 Hz), 7.30 (4 H, m), 6.19 (1 H, d, *J* = 7.5 Hz, N*H*, exchangeable with D_2O), 5.30-5.25 (2 H, m), 5.05-4.94 (3 H, m), 4.82 (1 H, dd, *J* = 10.2, 3.6 Hz), 4.63 (1 H, t, *J* = 8.5 Hz), 4.26-4.00 (4 H, m), 3.89-3.81 (2 H, m), 3.60 (1H, d, *J* = 9.6 Hz), 3.38 (1 H, q, *J* = 9.0 Hz), 2.43 (6 H, s), 2.10 (3 H, s), 2.08 (3 H, s), 2.01 (3 H, s), 1.97 (6 H, s), 1.59 (3 H, s); ^{13}C NMR (75 MHz, $CDCl_3$): δ 171.46 (s), 170.44 (s), 170.33 (s), 170.16 (s), 169.77 (s), 169.32 (s), 143.94 (s) 143.50 (s), 138.12 (s), 137.41 (s), 129.83 (d) 129.32 (d), 127.11 (2 x d), 95.28 (d), 83.35 (d), 75.07 (d), 73.30 (d), 72.66 (d), 69.76 (d), 69.20 (d), 68.34 (d), 67.78 (d), 62.45 (t),

61.30 (t), 56.97 (d), 21.37 (q), 20.59 (q), 20.51 (q), 20.40 (q), 20.34 (q); HRMS (ESI): MH^+ Found: 901.2374, $C_{38}H_{49}N_2O_{19}S_2$ requires 901.2371.

1,2-Di-(*p*-toluenesulfonamido)-tetrahydropyran 23.



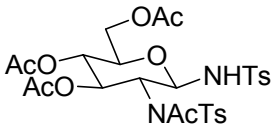
23

3.08 g of **23** was obtained from the reaction dihydropyran **22** (1.00 g, 11.90 mmol), using chloramine-T (7.71 g, 27.37 mmol) and iodine (0.452 g, 1.78 mmol) in CH_3CN (30 mL) in 14 h as per general procedure. Flash chromatography of the crude reaction mixture was performed with Hexane: Ethyl acetate (3:1). Yield 61%; white solid; mp 120 ºC (4:1 diastereomeric mixture) (recrystallized from hot benzene); IR (KBr): ν 3391.1, 3288.9, 2957.0, 2874.6, 1597.8, 1495.9, 1428.3, 1328.5, 1162.7, 1089.6, 1050.3, 1009.3, 955.5, 894.0, 812.5, 710.4, 659.9 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, for major diastereomer): δ 7.79-7.74 (4 H, m), 7.33-7.27 (4 H, m), 5.72 (1 H, d, J = 8.2 Hz, N*H* exchangeable with D_2O), 4.83 (1 H, d, J=7.3 Hz, N*H* exchangeable with D_2O), 4.40 (1 H, t, J = 8.4 Hz), 3.77-3.73 (1 H, m), 3.43-3.30 (1 H, m), 2.95-2.90 (1 H, m), 2.43 (3 H, s), 2.41 (3 H, s), 1.93-1.90 (1 H, m), 1.52-1.39 (3 H, m); ^{13}C NMR (75 MHz, $CDCl_3$ for major diastereomer): δ 143.65 (s), 143.31 (s), 138.23 (s), 137.26 (s), 129.78 (d), 129.37 (d), 126.99 (2 x d), 84.98 (d), 66.96 (t), 52.86 (d), 29.94 (t), 24.36, (t), 21.44 (2 x q); HRMS (ESI): $[M+Na]^+$ Found: 447.1024, $C_{19}H_{24}N_2O_5S_2Na$ requires 447.1024.

General procedure for chemoselective acetylation of disulfonamides at *C2*-nitrogen of 7 and 19:

To an ice cooled solution of a disulfonamide (1 equiv.) in pyridine were added acetic anhydride (2 equiv.) and DMAP (1 equiv.) and the reaction mixture was allowed to come to room temperature and stirred for 24 h. The colour of the reaction mixture changed from colourless to dark brown. It was then quenched with 10% HCl solution and extracted with ethyl acetate and washed with water. The organic layer was dried over sodium sulfate and concentrated. Product was purified by flash chromatography.

2-*N*-Acetyl-3,4,6-tri-*O*-acetyl-1,2-dideoxy-1,2-di-(*p*-toluenesulfonamido)-β-D-glucopyranose 24.

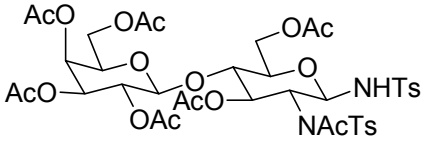


24

4.20 g of **24** was obtained by the acetylation of **7** (4.68 g, 7.64 mmol) using Ac_2O (1.44 mL, 15.28 mmol) and DMAP (0.932 g, 7.64 mmol) and in pyridine (8 mL) as per the general procedure. Flash chromatography of the crude reaction mixture was performed with Hexane: Ethyl acetate (2:1). Yield: 84%; white crystalline solid; mp 121 ºC (recrystallized from benzene/hexane); $[\alpha]_D^{28}$ -23.2 (*c* 1.20 in $CHCl_3$); Anal. Calcd. for $C_{28}H_{34}N_2O_{12}S_2$: Found: C, 51.02; H, 5.22; N, 3.80. requires C, 51.37; H, 5.23; N, 4.28; IR (KBr): ν 3226.0, 2985.9, 2927.9, 1754.0, 1707.2, 1596.9, 1461.6, 1343.6, 1233.6, 1166.7, 1087.9, 1057.6, 928.7, 893.7, 815.4, 683.7, 663.1 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 7.93 (2 H, d, *J* = 8.2 Hz), 7.77 (2 H, d, *J* = 8.2 Hz), 7.40 (2 H, d, *J* = 8.2 Hz), 7.28 (2 H, d, *J* = 8.1 Hz), 5.80-5.71 (3 H, m), 4.99 (1 H, t, *J* = 9.5 Hz), 4.29 (1 H, t, *J* = 9.0 Hz), 4.09 (1 H, dd, *J* = 12.3, 4.3 Hz), 3.83 (1 H, dd, *J* = 12.3, 2.00 Hz), 3.73-3.68 (1 H, m), 2.45 (3 H, m), 2.42 (3 H, s), 2.07 (3 H, s), 2.04 (3 H, s), 1.98 (3 H, s), 1.79 (3 H, s); ^{13}C NMR (75 MHz, $CDCl_3$): δ 170.62 (s), 170.46 (s), 170.01 (s), 169.41 (s), 145.69 (s), 143.66 (s), 137.81 (s), 135.76 (s), 130.23 (d), 129.38 (s), 128.29 (s), 127.33 (s), 80.95 (d), 73.09 (d), 69.69 (d), 69.03 (d), 61.74 (t), 60.87 (d), 25.69 (q), 21.55 (q), 21.46 (q), 20.60 (q), 20.49 (q), 20.37 (q).

.

4-*O*-[2,3,4,6-Tetra-*O*-acetyl-(β-D-galactopyranosyl)]-2-*N*-acetyl-3,6-di-*O*-acetyl-1,2-dideoxy-1,2-di-(*p*-toluenesulfonamido)-β-D-glucopyranose 25.

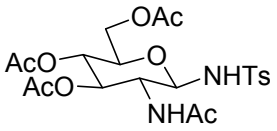


25

2.82 g of **25** was obtained by the acetylation of **19** (3.00 g, 3.33 mmol) using Ac_2O (0.628 mL, 6.66 mmol) and DMAP (0.406 g, 3.33 mmol) in pyridine (6 mL) as per

the general procedure. Flash chromatography of the crude reaction mixture was performed with Hexane: Ethyl acetate (1:1). Yield 90%; white solid; mp 90 ºC (recrystallized from benzene/hexane); $[\alpha]_D^{28}$ -12.3 (*c* 0.73 in $CHCl_3$); IR (KBr): ν 3629.0, 3257.9, 2981.5, 1761.7, 1597.6, 1495.8, 1434.8, 1370.6, 1239.1, 1167.1, 1058.6, 924.4, 816.1, 705.6, 662.3 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 7.94 (2 H, d, *J* = 7.8 Hz), 7.75 (2 H, d, *J* = 7.8 Hz), 7.40 (2 H, d, *J* = 7.8), 7.27 (2 H, d, *J* = 7.8 Hz), 5.76-5.69 (2 H, m), 5.51 (1 H, d, *J* = 10.3, N*H*, exchangeable with D_2O), 5.33 (1 H, s), 5.06 (1 H, dd, *J* = 9.5, 8.4 Hz), 4.92 (1 H, d, *J* = 10.3 Hz), 4.44 (1 H, d, *J* = 7.5 Hz), 4.20-3.94 (5 H, m), 3.86-3.67 (3 H, m), 2.46 (3 H, s), 2.43 (3 H, s), 2.13 (3 H, s), 2.07 (6 H, s), 2.05 (3 H, s), 2.02 (3 H, s), 1.95 (3 H, s), 1.90 (3 H, s); ^{13}C NMR (75 MHz, $CDCl_3$): δ 170.42 (s), 170.27 (s), 170.04 (2 x s), 169.74 (s), 168.82 (s), 145.59 (s), 143.58 (s), 137.74 (s), 135.75 (s), 130.14 (d), 129.28 (d), 128.22 (d), 127.20 (d), 100.45 (d), 80.81 (d), 76.80 (d), 73.77 (d), 70.85 (d), 70.46 (d), 69.47 (d), 68.90 (d), 66.50 (d), 61.81 (t), 60.96 (d), 60.69 (t), 25.53 (q), 21.45 (q), 21.37 (q), 20.60 (q), 20.46 (q); HRMS (ESI): MH^+, Found 943.2465. $C_{40}H_{51}N_2O_{20}S_2$, requires 943.2477.

2-Acetamido-3,4,6-tri-*O*-acetyl-1,2-dideoxy-1-(*p*-toluenesulfonamido)-β-D-glucopyranose 26.



26

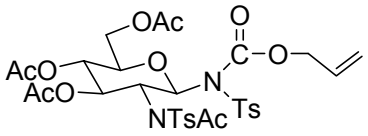
In a flame dried 100 mL three-necked round-bottomed flask, was taken powdered samarium metal (0.344 g, 2.29 mmol, 10 equiv.) and further flame dried under argon atmosphere. After cooling under argon atmosphere, dry THF (24 mL) and CH_2I_2 (0.157 mL, 1.95 mmol, 8.5 equiv.) were added to it and reaction mixture was subjected to sonication at room temperature. Deep blue colour was obtained in 5 minutes. After 30 minutes, reaction flask was taken out and compound **24** (0.150 g, 0.23 mmol) was added. After stirring for 5 minutes at room temperature, degassed water (0.206 g, 11.45 mmol, 50 equiv.) was added drop-wise under argon atmosphere. The colour of reaction mixture turned grey black initially and finally to yellow brown. After 25 min, the reaction mixture

was quenched with saturated NH_4Cl solution (10 mL) and extracted with $CHCl_3$ (4 x 25 mL). The combined organic layer was washed by water (2 x 25 mL) and dried over sodium sulfate and concentrated. Flash chromatography (1:1 hexane/ethyl acetate) of the resulting residue provided **26** (0.102 g) as a white solid. Yield 89%; white solid; mp 160-162 ºC decompose (recrystallized from CH_2Cl_2/hexane); $[\alpha]_D^{28}$ +31.0 (*c* 0.59 in THF); IR (KBr): ν 3294.1, 1746.0, 1658.4, 1543.3, 1459.4, 1377.2, 1332.6, 1238.4, 1157.5, 1086.5, 1049.4, 678.8 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 7.76 (2 H, d, *J* = 7.8), 7.27 (2H, d, *J* = 7.8), 6.54 (1 H, d, *J* = 7.8, N*H*, exchangeable with D_2O), 6.00 (1 H, d, *J* = 7.8, N*H*, exchangeable with D_2O), 5.02 (2 H, d, *J* = 8.8), 4.72 (1H, t, *J* = 8.7), 4.14-3.97 (3 H, m), 3.65 (1 H, brs), 2.41 (3 H, s), 2.05 (6 H, s), 2.03 (3 H, s), 1.91 (3 H, s); ^{13}C NMR (75 MHz, $CDCl_3$): δ 172.40 (s), 171.54 (s), 170.49 (s), 169.27 (s), 143.44 (s), 138.62 (s), 129.32 (d), 126.99 (d), 84.41 (d), 73.02 (d), 72.62 (d), 68.05 (d), 61.88 (t), 53.24 (d), 22.92 (q), 21.45 (q), 20.61 (q); HRMS (ESI): $[M+Na]^+$, Found 523.1358. $C_{21}H_{28}N_2O_{10}SNa$ requires 523.1362.

General Procedure for 1-*N*-Alloc Protection of 24 and 25:

In a flame dried 50 mL three-necked round bottomed flask, was taken **24** or **25** (1 equiv.) and dissolved in dry CH_2Cl_2 under a N_2 atmosphere. To this, DMAP (20 mol%) and Et_3N (2 equiv.) were added and the reaction mixture was cooled to ice-salt temperature. Alloc chloride (4 equiv.) was injected into the reaction mixture drop-wise. After complete addition of alloc chloride, reaction mixture was warmed to 30 °C and stirred till the reaction was over (as indicated by TLC). The reaction mixture was quenched with saturated NH_4Cl solution and extracted with $CHCl_3$. The combined organic layer was washed with water and dried over sodium sulfate and concentrated. Flash chromatography of the resulting residue provided **28** or **29** respectively as a white solid.

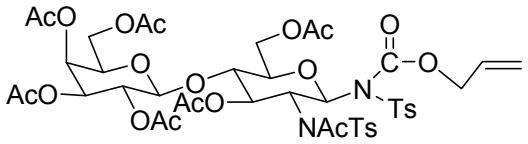
2-*N*-Acetyl-3,4,6-tri-*O*-acetyl-1-*N*-allyloxycarbonyl-1,2-dideoxy-1,2-di-(*p*-toluenesulfonamido)-β-D-glucopyranose 28.



28

2.58 g of **28** was obtained in 9 h from **24** (2.89 g, 4.42 mmol), using DMAP (0.108 g, 0.88 mmol), Et_3N (1.23 mL, 8.84 mmol) and alloc chloride (1.89 ml, 17.68 mmol) in 10 mL of dry CH_2Cl_2 as per the general procedure. Flash chromatography of the crude reaction mixture was performed with Hexane: Ethyl acetate (3:1). Yield 79%; white solid; mp 52 ºC; $[\alpha]_D^{28}$ -33.4 (*c* 0.64 in $CHCl_3$); IR (KBr): ν 3029.4, 2957.5, 1749.1, 1708.0, 1448.6, 1369.6, 1234.8, 1168.2, 1086.4, 1053.7, 924.2, 664.8 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 7.87 (2 H, d, *J* = 8.1 Hz), 7.76 (2 H, d, *J* = 8.1 Hz), 7.33 (2 H, d, *J* = 8.7 Hz), 7.29 (2 H, d, *J* = 8.4 Hz), 6.76 (1 H, d, *J* = 9.3 Hz), 5.99 (1 H, t, *J* = 9.6 Hz), 5.90-5.77 (1 H, m), 5.57 (1 H, t, *J* = 9.6 Hz), 5.33 (1 H, d, *J* = 17.1 Hz), 5.23 (1 H, d, *J* = 10.5 Hz), 5.11 (1 H, t, *J* = 9.6 Hz), 4.68 (1 H, dd, *J* = 13.2, 5.4 Hz), 4.52 (1 H, dd, *J* = 13.2, 5.4 Hz), 4.13 (2 H, s), 3.99-3.95 (1 H, m), 2.43 (6 H, s), 2.15 (3 H, s), 2.07 (3 H, s), 2.04 (3 H, s), 2.02 (3 H, s); ^{13}C NMR (75 MHz, $CDCl_3$): δ 171.51 (s), 170.43 (2 x s), 169.42 (s), 150.90 (s), 145.26 (s), 144.86 (s), 136.14 (s), 135.84 (s), 130.16 (d), 129.32 (d), 128.57 (d), 127.98 (d), 119.21 (t), 82.49 (d), 73.90 (d), 70.16 (d), 69.04 (d), 68.06 (t), 62.16 (t), 58.21 (d), 26.07 (q), 21.58 (q), 21.53 (q), 20.67 (q), 20.56 (q); HRMS (ESI): MH^+, found: 739.1847. $C_{32}H_{39}N_2O_{14}S_2$ requires 739.1843.

4-*O*-[2,3,4,6-Tetra-*O*-acetyl-(β-D-galactopyranosyl)]-2-*N*-acetyl-1-*N*-allyloxycarbonyl-3,6-di-*O*-acetyl-1,2-dideoxy-1,2-di-(*p*-toluenesulfonamido)-β-D-glucopyranose 29.

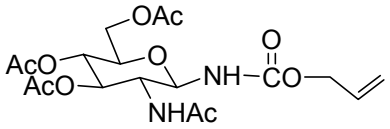


29

1.63 g of **29** was obtained in 12 h from **25** (2.00 g, 2.12 mmol), DMAP (0.052 g, 0.424 mmol), Et_3N (0.589 mL, 4.24 mmol) and alloc chloride (0.904 g, 8.48 mmol) in dry CH_2Cl_2 (10 mL) as per the general procedure. Flash chromatography of the crude

reaction mixture was performed with 2:1 (Hexane: Ethyl acetate). Yield 75%; white solid; mp 79 ºC; $[\alpha]_D^{28}$ -27.7 (*c* 0.49 in $CHCl_3$); IR (KBr): ν 3481.9, 2983.2, 1747.3, 1597.2, 1432.7, 1370.2, 1228.2, 1170.4, 1062.9, 918.6, 816.0, 665.7 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 7.85 (2 H, d, *J* = 7.5 Hz), 7.77 (2 H, d, *J* = 7.5 Hz), 7.31 (4 H, m), 6.74 (1 H, d, *J* = 9.3 Hz), 5.97 (1 H, t, *J* = 9.0), 5.80-5.71 (1 H, m), 5.52 (1 H, t, *J* = 9.3 Hz), 5.36-5.10 (4 H, m), 4.97 (1 H, d, *J* = 10.2 Hz), 4.64-4.44 (4 H, m), 4.18-4.03 (4 H, m), 3.93-3.79 (2 H, m), 2.44 (6 H, s), 2.16 (3 H, s), 2.13 (3 H, s), 2.09 (9 H, s), 2.06 (3 H, s), 1.97 (3 H, s); ^{13}C NMR (75 MHz, $CDCl_3$): δ 171.22 (s), 170.22 (s), 170.05 (s), 169.97 (s), 169.83 (s), 169.77 (s), 150.62 (s), 145.04 (s), 144.63 (s), 136.10 (s), 135.86 (s), 129.99 (d), 129.22 (d), 128.26 (d), 127.78 (d), 118.72 (t), 100.35 (d), 82.32 (d), 76.71 (d), 74.60 (d), 70.88 (d), 70.56 (d), 69.64 (d), 68.99 (d), 67.69 (t), 66.60 (d), 61.90 (t), 60.78 (t), 58.19 (d), 25.84 (q), 21.38 (q), 20.78 (d), 20.58 (q), 20.39 (q), 20.27 (q); HRMS (ESI): MH^+, found: 1027.2697. $C_{44}H_{55}N_2O_{22}S_2$ requires 1027.2688.

2-Acetamido-3,4,6-tri-*O*-acetyl-1-*N*-allyloxycarbonyl-1,2-dideoxy-β-D-glucopyranose 30.

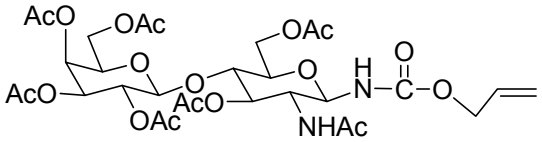


30

In a flame dried 100 mL three-necked round-bottomed flask, was taken powdered samarium metal (0.610 g, 4.07 mmol, 15 equiv.) and further flame dried under argon atmosphere. After cooling under argon atmosphere, dry THF (42 mL) and CH_2I_2 (0.284 mL, 3.53 mmol, 13 equiv.) were added to it and reaction mixture was subjected to sonication at room temperature. Deep blue colour was obtained in 5 minutes. After 1 h, reaction was taken out and compound **28** (0.200 g, 0.27 mmol) was added. After stirring for 5 minutes at room temperature, degassed water (0.366 mL, 20.32 mmol, 75 equiv.) was added drop-wise under argon atmosphere and the colour of reaction mixture turned grey black initially and finally to yellow brown. After 1 h, the reaction mixture was quenched with saturated NH_4Cl solution (10 mL) and extracted with $CHCl_3$ (4 x 25 mL). The combined organic layer was washed with water (2 x 25 mL) and dried over sodium

sulfate and concentrated. Flash chromatography (1:1 hexane/ethyl acetate) of the resulting residue provided **30** (0.105 g, 90%) as a white solid. Mp 164 ºC (decomp.) (recrystallized from CH_2Cl_2/hexane); $[\alpha]_D^{28}$ -9.3 (*c* 0.59 in $CHCl_3$); IR (KBr) ν 3324.8, 3268.3, 2925.2, 1748.6, 1708.6, 1654.6, 1541.6, 1223.7, 1045.7, 771.8 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): *δ* 6.23 (1 H, d, *J* = 8.7 Hz, N*H*, exchangeable with D_2O), 5.99 (1 H, brd, N*H*, exchangeable with D_2O), 5.94-5.81 (1 H, m), 5.27 (1 H, d, *J* = 17.1 Hz), 5.20 (1 H, d, *J* = 10.5 Hz), 5.16-5.02 (2 H, m), 4.86 (1 H, t, *J* = 9.3 Hz), 4.56 (2 H, d, *J* = 5.4 Hz), 4.30 (1 H, dd, *J* = 12.3, 3.9 Hz), 4.19-4.07 (2 H, m), 3.74 (1 H, brd), 2.09 (3 H, s), 2.07 (3 H, s), 2.04 (3 H, s), 1.96 (3 H, s); ^{13}C NMR (75 MHz, $CDCl_3$): *δ* 171.47 (2 x s), 170.65 (s), 169.27 (s), 155.81 (s), 132.21 (d), 117.74 (t), 82.21, 73.06 (2 x d), 68.04 (d), 65.94 (t), 61.81 (t), 52.71 (d), 22.96 (q), 20.62 (q), 20.51 (s); HRMS (ESI): MH^+, found: 431.1667. $C_{18}H_{27}N_2O_{10}$ requires 431.1666.

4-*O*-[2,3,4,6-Tetra-*O*-acetyl-(β-D-galactopyranosyl)]-2-acetamido-3,6-di-*O*-acetyl-1-*N*-allyloxycarbonyl-1,2-dideoxy-β-D-glucopyranose 31.

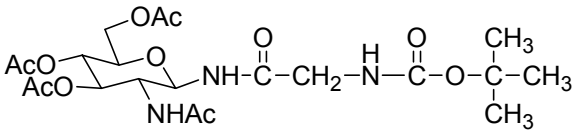


31

Following the similar procedure as for compound **30**, compound **31** was obtained in 88% yield (0.123 g) as a white solid from the reaction of **29** (0.200 g, 0.195 mmol) with SmI_2 (3.31 mmol, 17 equiv.) in dry THF (35 mL) and degassed water (0.351 mL, 19.50 mmol, 100 equiv.). Flash chromatography of the crude reaction mixture was performed with 1:2 (Hexane: Ethyl acetate). Mp 94 ºC; $[\alpha]_D^{28}$ +5.4 (*c* 0.58 in $CHCl_3$); IR (KBr) ν 3369.4, 2926.6, 1746.5, 1539.9, 1370.2, 1226.8, 1047.6 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 6.17 (1 H, d, *J* = 7.4 Hz, N*H*, exchangeable with D_2O), 6.02 (1 H, d, *J* = 7.8 Hz, N*H*, exchangeable with D_2O), 5.92-5.81 (1 H, m), 5.33 (2 H, d, *J* = 17.0 Hz), 5.22 (1 H, dd, *J* = 10.4, 8.7 Hz), 5.14-4.95 (3 H, m), 4.79 (1 H, t, *J* = 8.0 Hz), 4.56-4.41 (4 H, m), 4.15-3.96 (4 H, m), 3.89-3.67 (3 H, m), 2.15 (3 H, s), 2.12 (3 H, s), 2.10 (3 H, s), 2.06 (6 H, s), 1.97 (3 H, s), 1.96 (3 H, s); ^{13}C NMR (75 MHz, $CDCl_3$): δ 171.76 (s),

171.40 (s), 170.32 (s), 170.24 (s), 169.98 (2 x s), 169.11 (s), 155.63 (s), 132.21 (d), 117.72 (t), 100.96 (d), 82.29 (d), 75.74 (d), 73.95 (d), 73.08 (d), 70.82 (d), 70.63 (t), 68.97 (d), 66.60 (d), 65.90 (t), 62.03 (t), 60.81 (t), 53.07 (d), 22.94 (q), 20.71 (q), 20.49 (s).

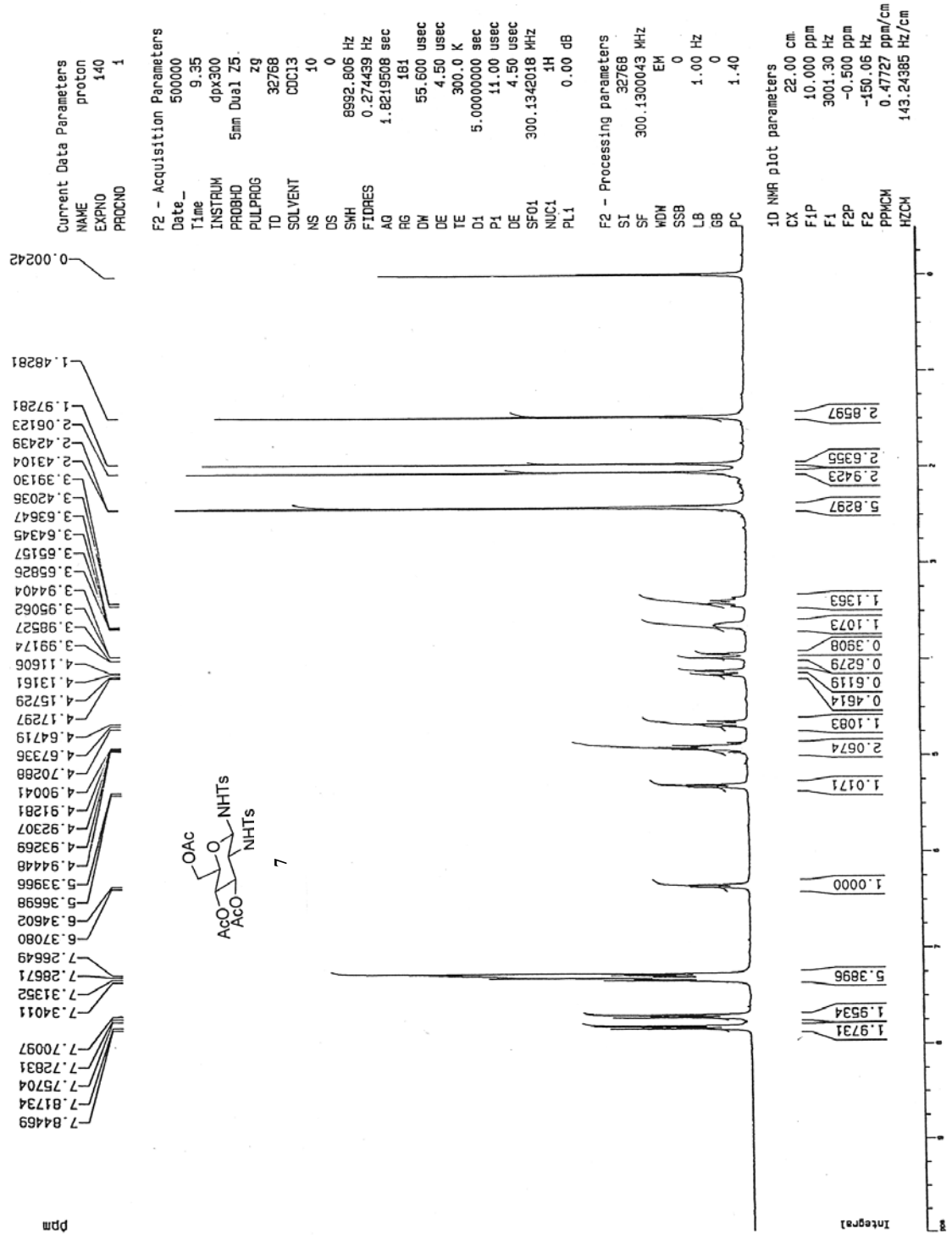
α-*N*-*tert*-Butoxylcarbonyl-(*N*-2-Acetamido-3,4,6-tri-*O*-acetyl-1,2-dideoxy-β-D-glucopyranosyl)glycine 32.

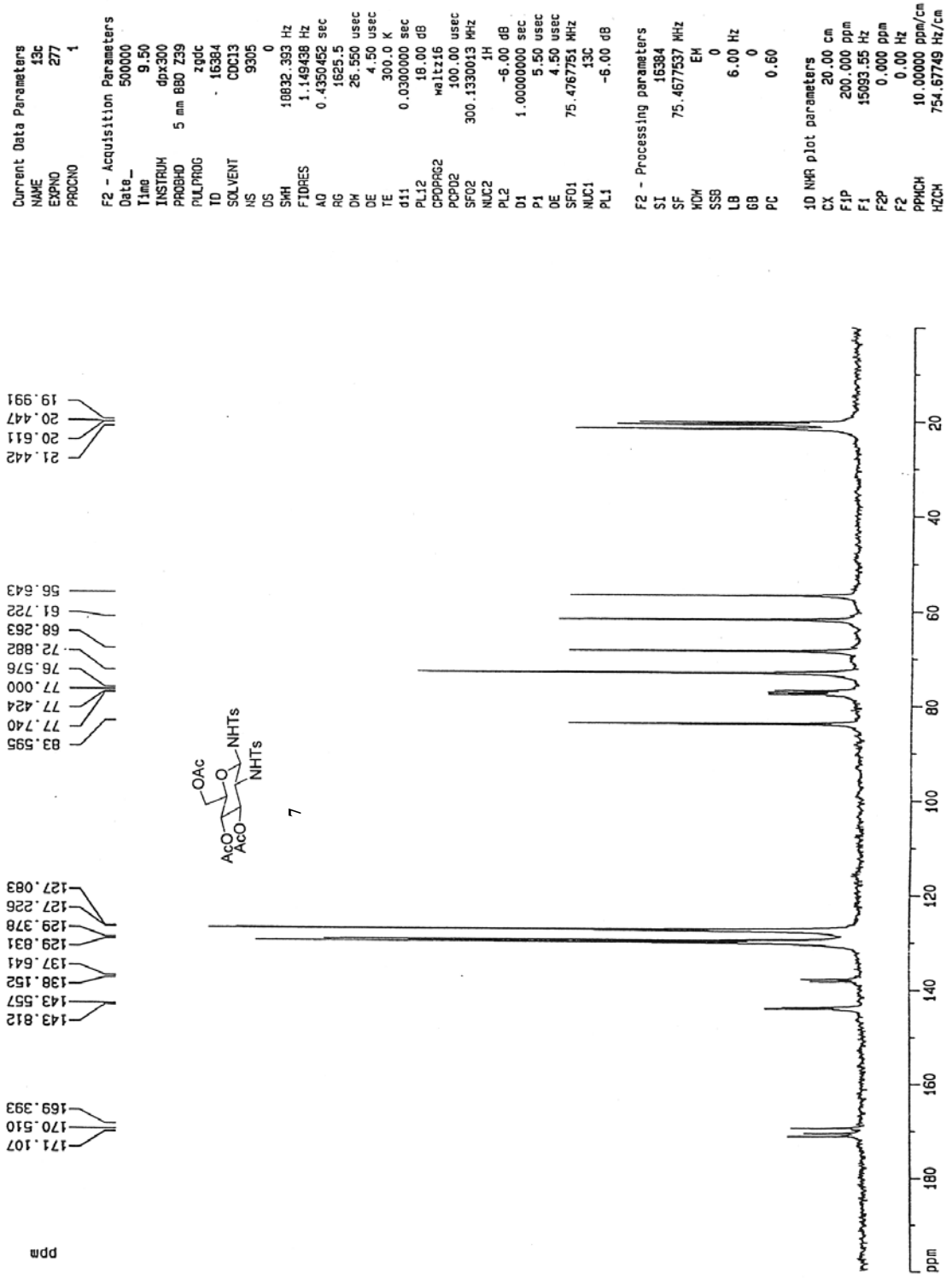


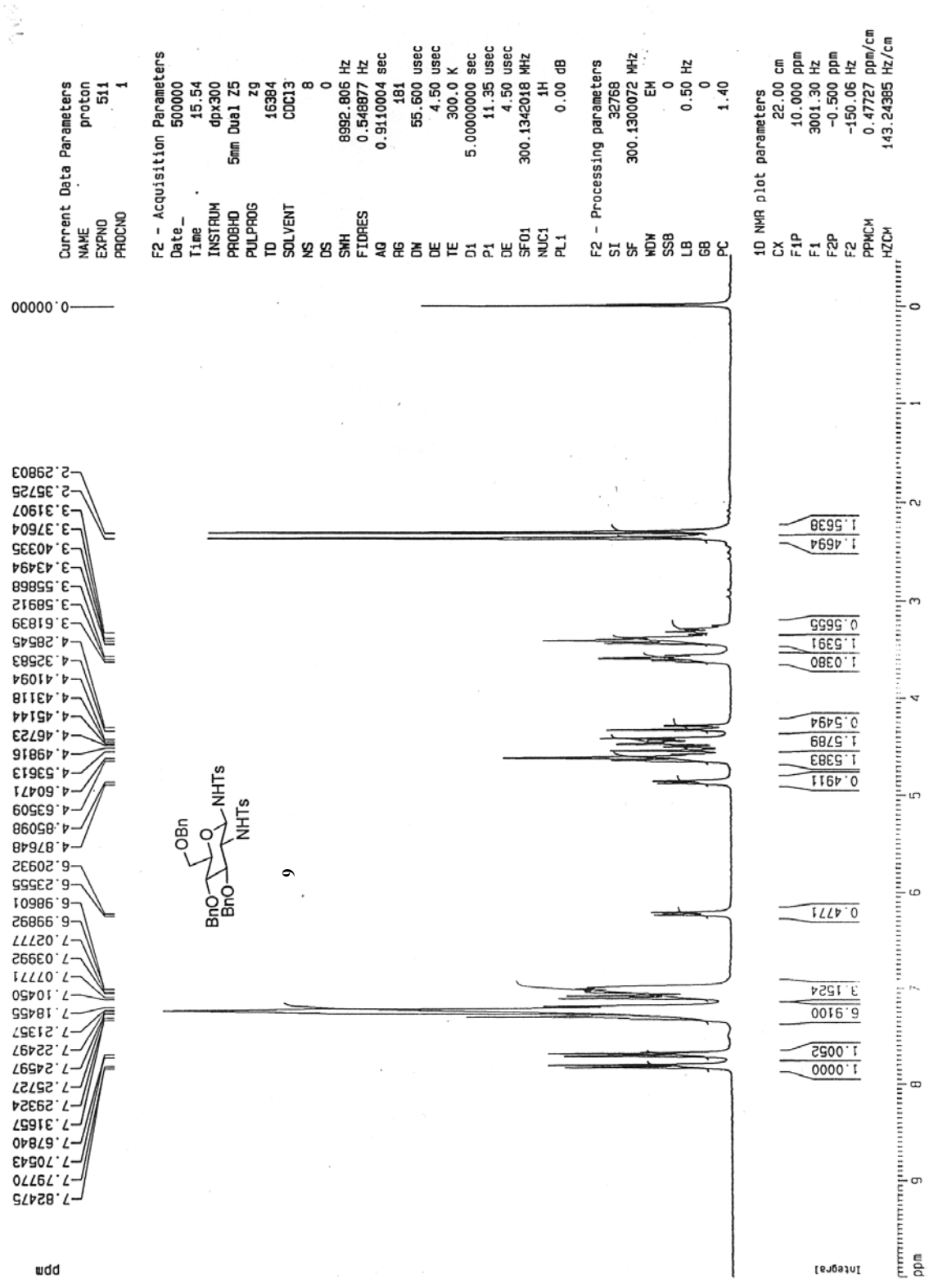
32

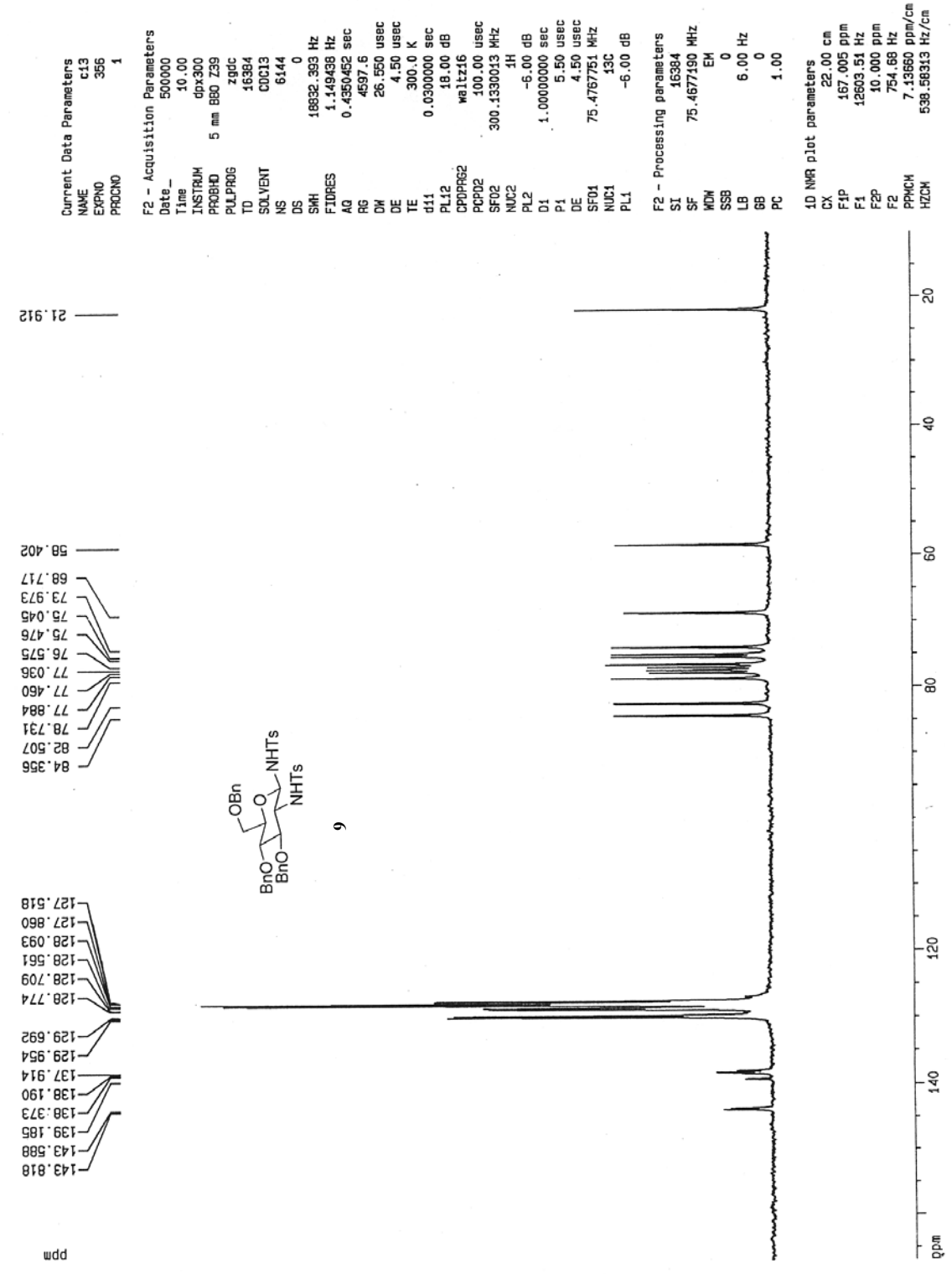
In a flame dried 50 mL three-necked round bottomed flask, was taken **30** (0.198 g, 0.46 mmol) and dissolved in dry THF (2 mL) under a N_2 atmosphere. To this, $(Ph_3P)_4Pd$ (0.058g, 0.05 mmol, 10 mol%) was added followed by drop-wise addition of Et_2NH (0.477 mL, 4.60 mmol, 10 equiv.). The reaction mixture was allowed to stir at room temperature (30 ºC). After completion of reaction (20 minutes, as indicated by TLC), THF was evaporated completely. This was then dissolved in dry CH_2Cl_2 (5 mL) and transferred drop-wise to a suspension of *N*-boc glycine (0.184 g, 0.69 mmol, 1.5 equiv.), DCC (0.171 g, 0.83 mmol, 1.8 equiv.) and DMAP (0.084 g, 0.69 mmol, 1.5 equiv.) in dry CH_2Cl_2 (6 mL) that was pre-stirred for 2 h. Reaction mixture was then allowed to stir at 30 ºC for 12 h., after which it was filtered and the residue was washed with more amount of CH_2Cl_2 (100 mL). Organic layer was washed with 5% $NaHCO_3$ (2 x 20 mL), saturated NH_4Cl (3 x 20 mL), dried over sodium sulfate and concentrated. Flash chromatography (ethyl acetate) of the resulting residue provided **32** (0.167 g, 72%) as a white solid. Mp 85 ºC (recrystallized from ethyl acetate: hexane); $[\alpha]_D^{28}$ +3.4 (*c* 0.24 in $CHCl_3$); IR (KBr): ν 3319.3, 2975.7, 2358.9, 1747.5, 1665.0, 1533.1, 1376.4, 1241.1, 1168.6, 1046.9 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 7.46 (1 H, d, J = 7.6 Hz, N*H*, exchangeable with D_2O), 6.26 (1 H, d, J = 8.3 Hz, N*H*, exchangeable with D_2O), 5.16-5.09 (4 H, m), 4.28 (1 H, dd, J = 12.3, 4.2 Hz), 4.21-4.13 (1H, m), 4.08 (1 H, dd, J = 12.3, 1.8 Hz), 3.81-3.80 (3 H, m), 2.09 (3 H, s), 2.06 (3 H, s), 2.04 (3 H, s), 1.94 (3 H, s),

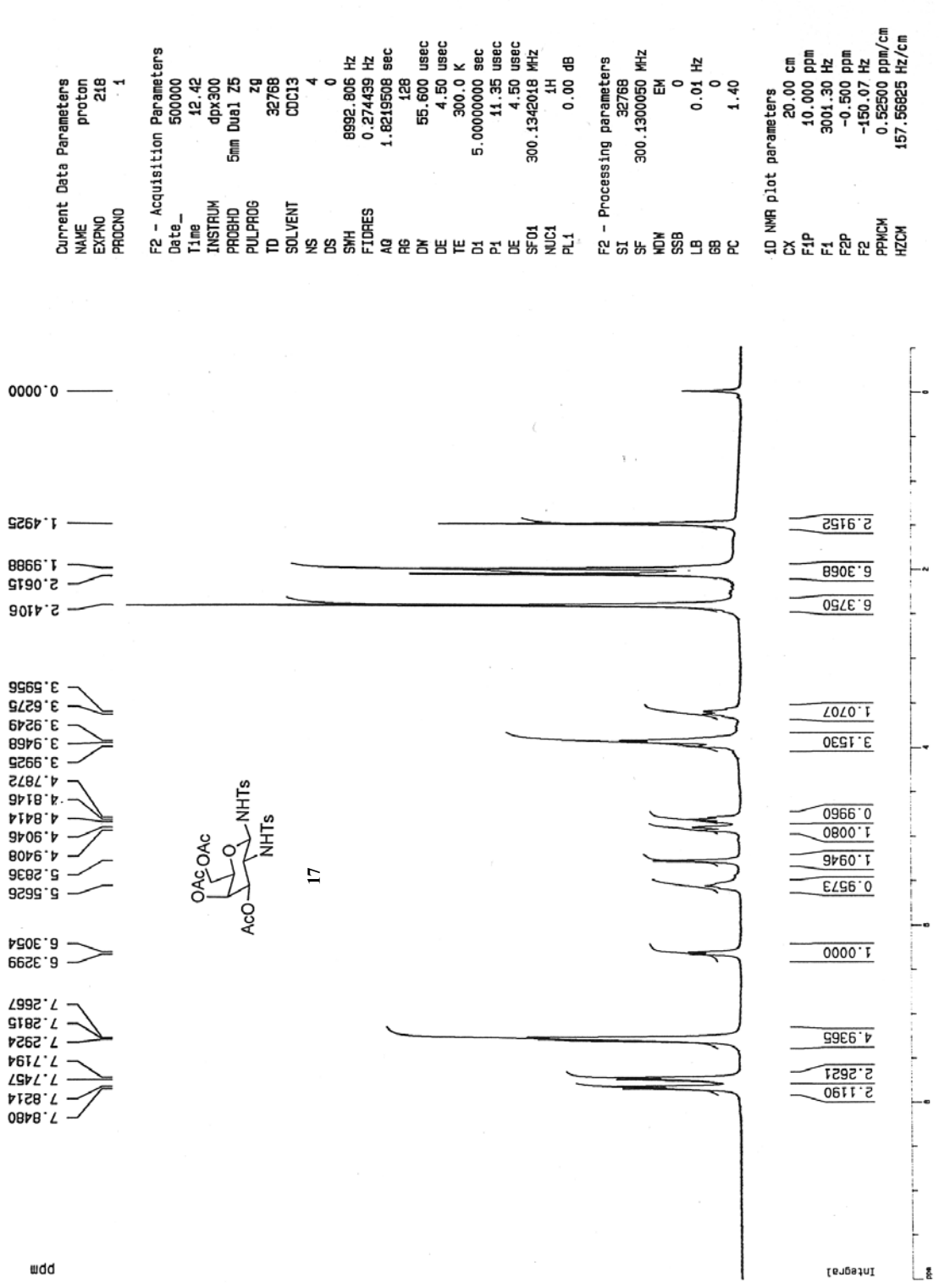
1.46 (9 H, s); ^{13}C NMR (75 MHz, $CDCl_3$): δ 172.00 (s), 171.58 (s), 170.70 (s), 170.65 (s), 169.28 (s), 155.73 (s), 79.96 (d), 73.51 (d), 72.84 (d), 67.90 (s), 61.78 (t), 53.14 (d), 44.11 (t), 28.27 (3 x q), 22.94 (3 x q), 20.68 (q), 20.63 (q), 20.54 (q); HRMS (ESI): MH^+, Found 504.2204. $C_{21}H_{34}N_3O_{11}$ requires 504.2193.

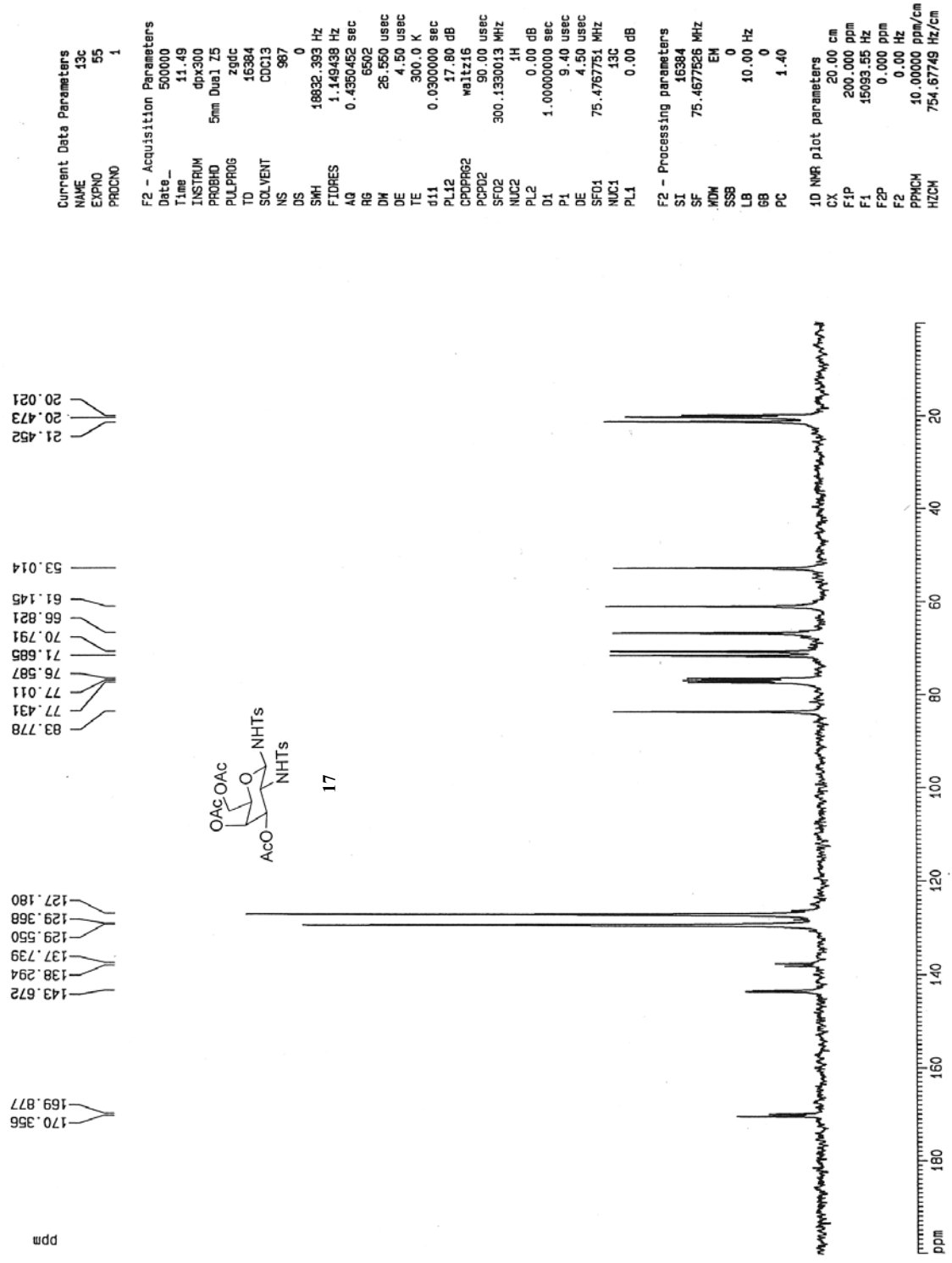

Current Data Parameters
NAME proton
EXPNO 140
PROCNO 1
F2 - Acquisition Parameters
Date_ 500000
Time 9.35
INSTRUM dpx300
PROBHD 5mm Dual Z5.
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 10
DS 0
SWH 8992.806 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 181
DW 55.600 usec
DE 4.50 usec
TE 300.0 K
D1 5.00000000 sec
P1 11.00 usec
DE 4.50 usec
SFO1 300.1342018 MHz
NUC1 1H
PL1 0.00 dB
F2 - Processing parameters
SI 32768
SF 300.1300043 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 22.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.47727 ppm/cm
HZCM 143.24385 Hz/cm
0.00242
1.48281
1.97281
2.06123
2.42439
2.43104
3.39130
3.42036
3.63647
3.64345
3.65157
3.65826
3.94404
3.95062
3.98527
3.99174
4.11606
4.13161
4.15729
4.17297
4.64719
4.67336
4.70288
4.90041
4.91281
4.92307
4.93269
4.94448
5.33966
5.36698
6.34602
6.37080
7.26649
7.28671
7.31352
7.34011
7.70097
7.72831
7.75704
7.81734
7.84469
2.8597
2.6355
2.9423
5.8297
1.1353
1.1073
0.3908
0.6279
0.6119
0.4614
1.1083
2.0674
1.0171
1.0000
5.3896
1.9534
1.9731
OAc
NHTs
NHTs
AcO
AcO
7
ppm
Integral

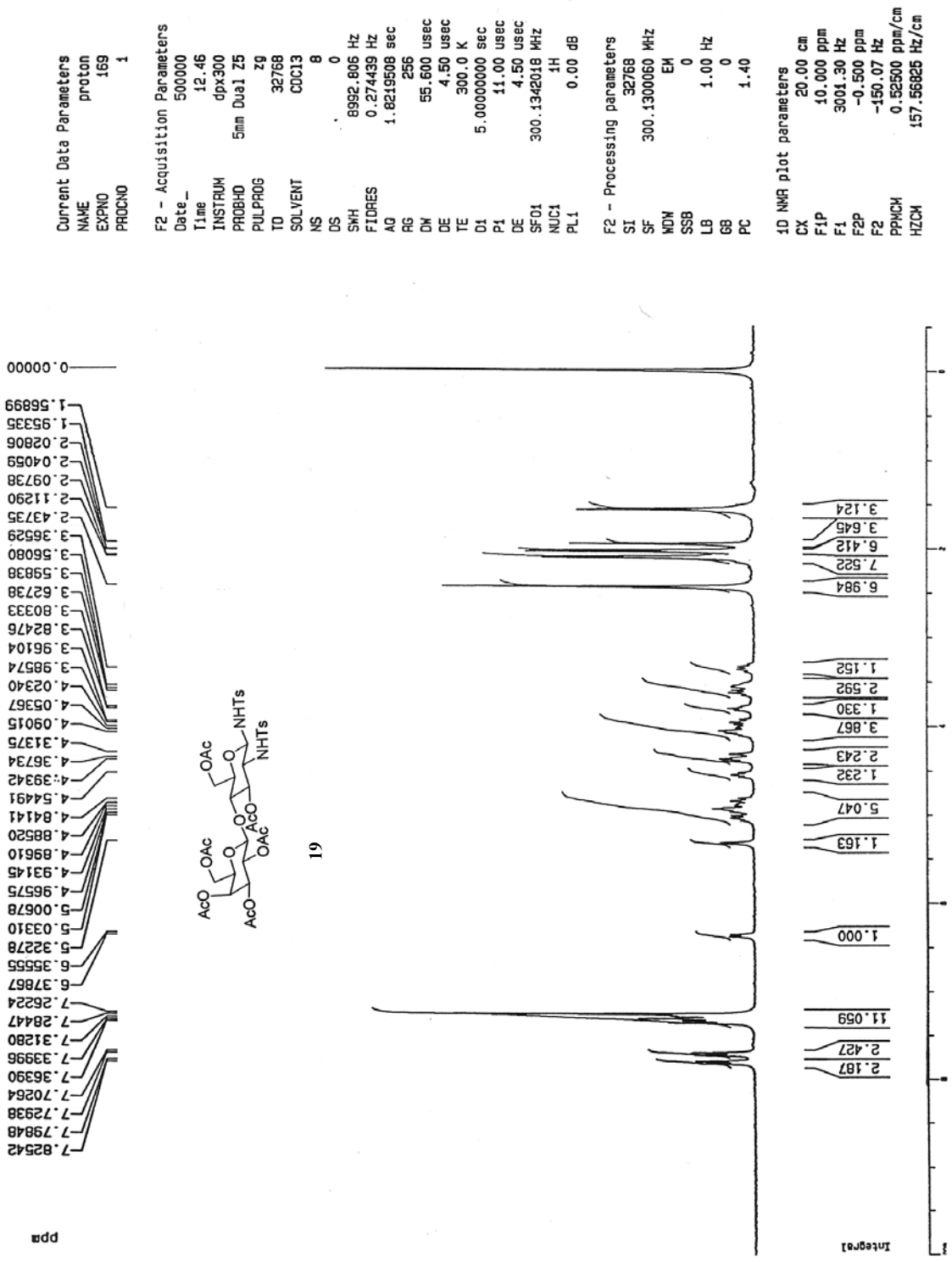

Current Data Parameters
NAME 13c
EXPNO 277
PROCNO 1
F2 - Acquisition Parameters
Date_ 500000
Time 9.50
INSTRUM dpx300
PROBHD 5 mm BBO Z39
PULPROG zgdc
TD 16384
SOLVENT CDCl3
NS 9305
DS 0
SWH 10832.393 Hz
FIDRES 1.149438 Hz
AQ 0.4350452 sec
RG 1625.5
DW 26.550 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
PL12 18.00 dB
CPDPRG2 waltz16
PCPD2 100.00 usec
SFO2 300.1330013 MHz
NUC2 1H
PL2 -6.00 dB
D1 1.00000000 sec
P1 5.50 usec
DE 4.50 usec
SFO1 75.4767751 MHz
NUC1 13C
PL1 -6.00 dB
F2 - Processing parameters
SI 16384
SF 75.4677537 MHz
WDW EM
SSB 0
LB 6.00 Hz
GB 0
PC 0.60
1D NMR plot parameters
CX 20.00 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 10.00000 ppm/cm
HZCM 754.67749 Hz/cm
19.991
20.447
20.611
21.442
56.643
61.722
68.263
72.882
76.576
77.000
77.424
77.740
83.595
127.083
127.226
129.378
129.831
137.641
138.152
143.557
143.812
169.393
170.510
171.107
OAc
AcO
AcO
O
NHTs
NHTs
7
ppm
20
40
60
80
100
120
140
160
180

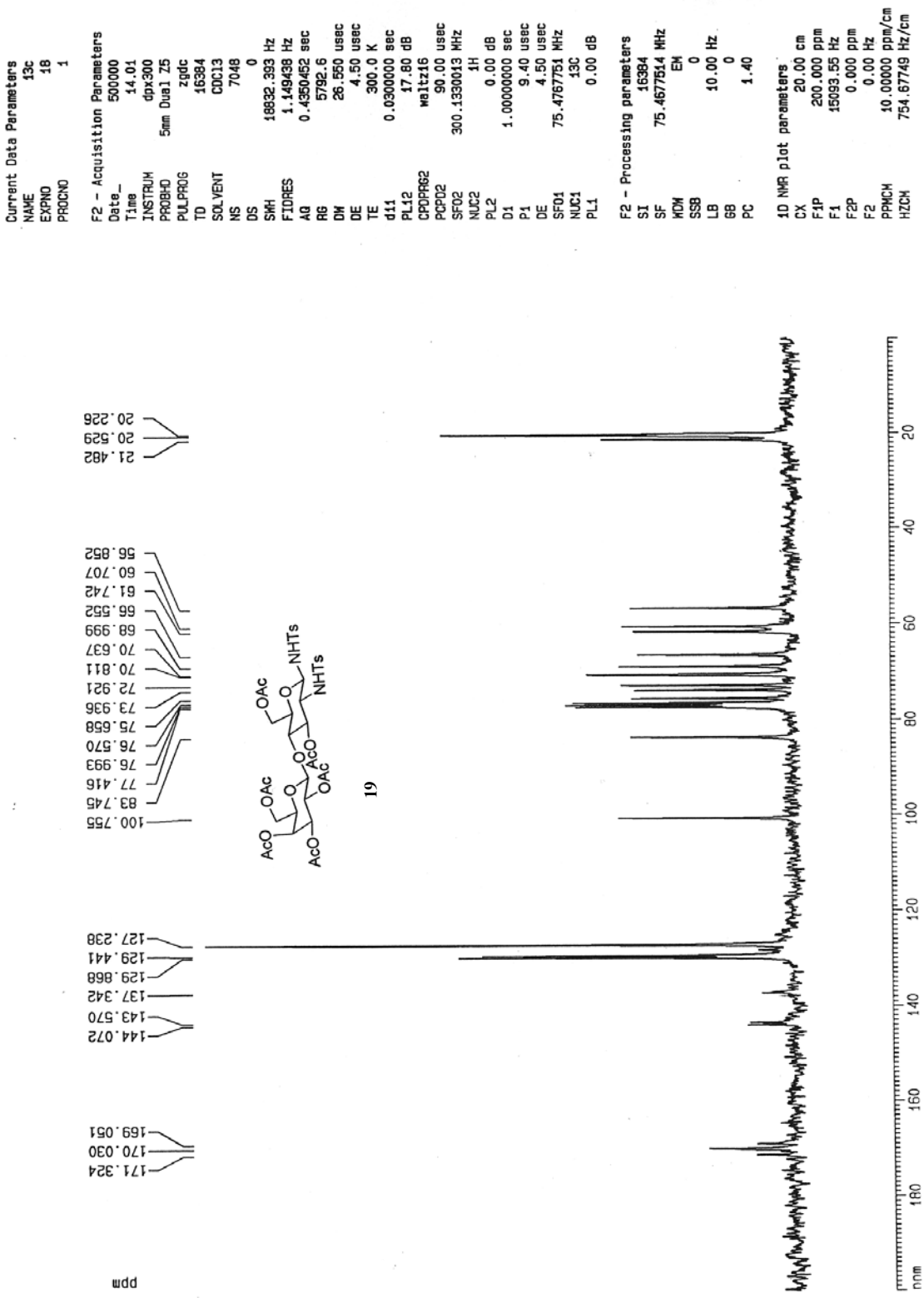

Current Data Parameters
NAME proton
EXPNO 511
PROCNO 1
F2 - Acquisition Parameters
Date_ 500000
Time 15.54
INSTRUM dpx300
PROBHD 5mm Dual Z5
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 8
DS 0
SWH 8992.806 Hz
FIDRES 0.548877 Hz
AQ 0.9110004 sec
RG 181
DW 55.600 usec
DE 4.50 usec
TE 300.0 K
D1 5.00000000 sec
P1 11.35 usec
DE 4.50 usec
SFO1 300.1342018 MHz
NUC1 1H
PL1 0.00 dB
F2 - Processing parameters
SI 32768
SF 300.1300072 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 22.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.06 Hz
PPMCM 0.47727 ppm/cm
HZCM 143.24385 Hz/cm
OBn
NHTs
NHTs
BnO
BnO
9
ppm
Integral
ppm

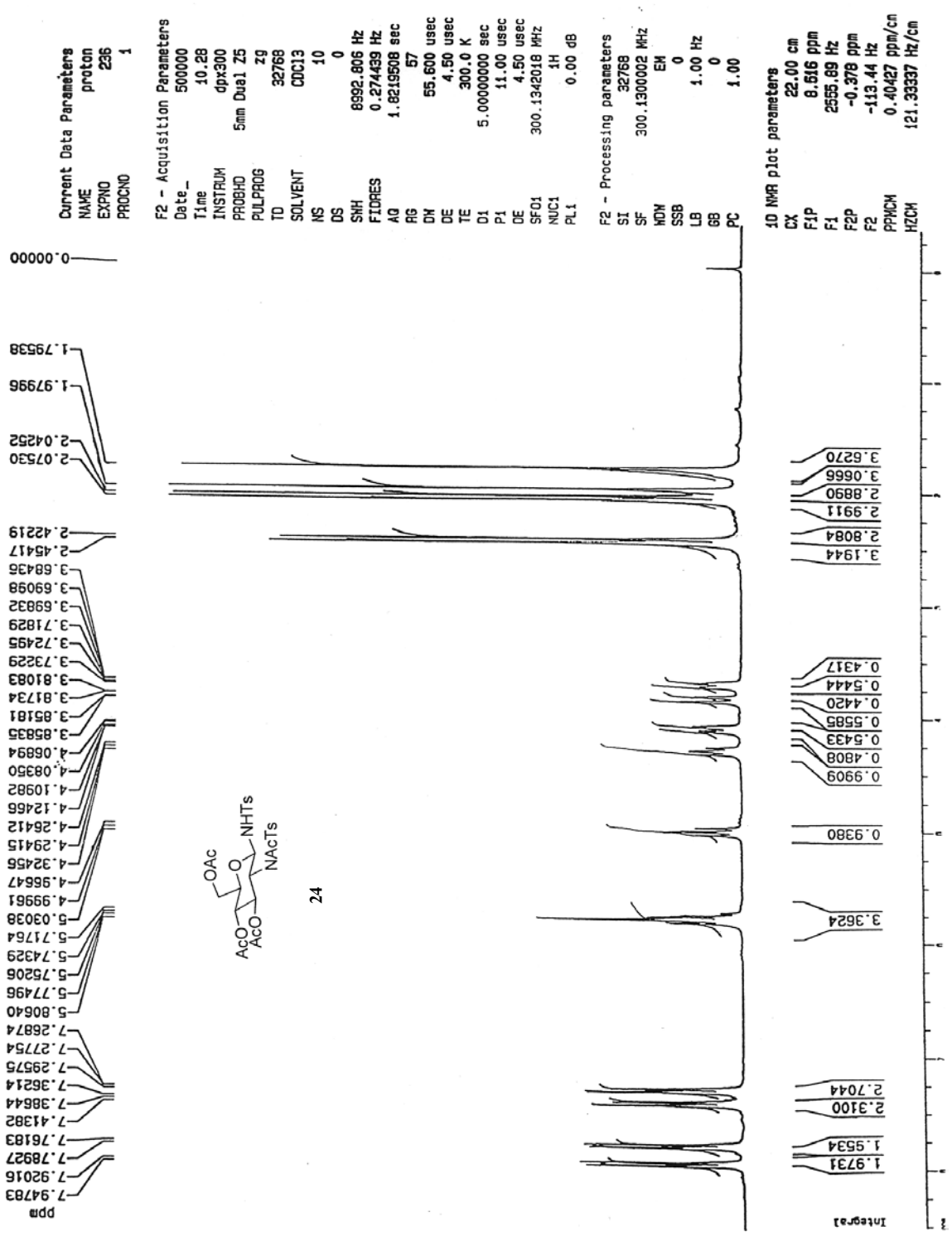

Current Data Parameters
NAME c13
EXPNO 356
PROCNO 1
F2 - Acquisition Parameters
Date_ 500000
Time 10.00
INSTRUM dpx300
PROBHD 5 mm BBO Z39
PULPROG zgdc
TD 16384
SOLVENT CDCl3
NS 6144
DS 0
SWH 18832.393 Hz
FIDRES 1.149438 Hz
AQ 0.4350452 sec
RG 4597.6
DW 26.550 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
PL12 18.00 dB
CPDPRG2 waltz16
PCPD2 100.00 usec
SFO2 300.1330013 MHz
NUC2 1H
PL2 -6.00 dB
D1 1.00000000 sec
P1 5.50 usec
DE 4.50 usec
SFO1 75.4767751 MHz
NUC1 13C
PL1 -6.00 dB
F2 - Processing parameters
SI 16384
SF 75.4677190 MHz
WDW EM
SSB 0
LB 6.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.00 cm
F1P 167.005 ppm
F1 12603.51 Hz
F2P 10.000 ppm
F2 754.68 Hz
PPMCM 7.13660 ppm/cm
HZCM 538.58313 Hz/cm
21.912
58.402
68.717
73.973
75.045
75.476
76.575
77.036
77.460
77.884
78.731
82.507
84.356
127.518
127.860
128.093
128.561
128.709
128.774
129.692
129.954
137.914
138.190
138.373
139.185
143.588
143.818
OBn
O
NHTs
NHTs
BnO
BnO
9
ppm
20
40
60
80
120
140
ppm

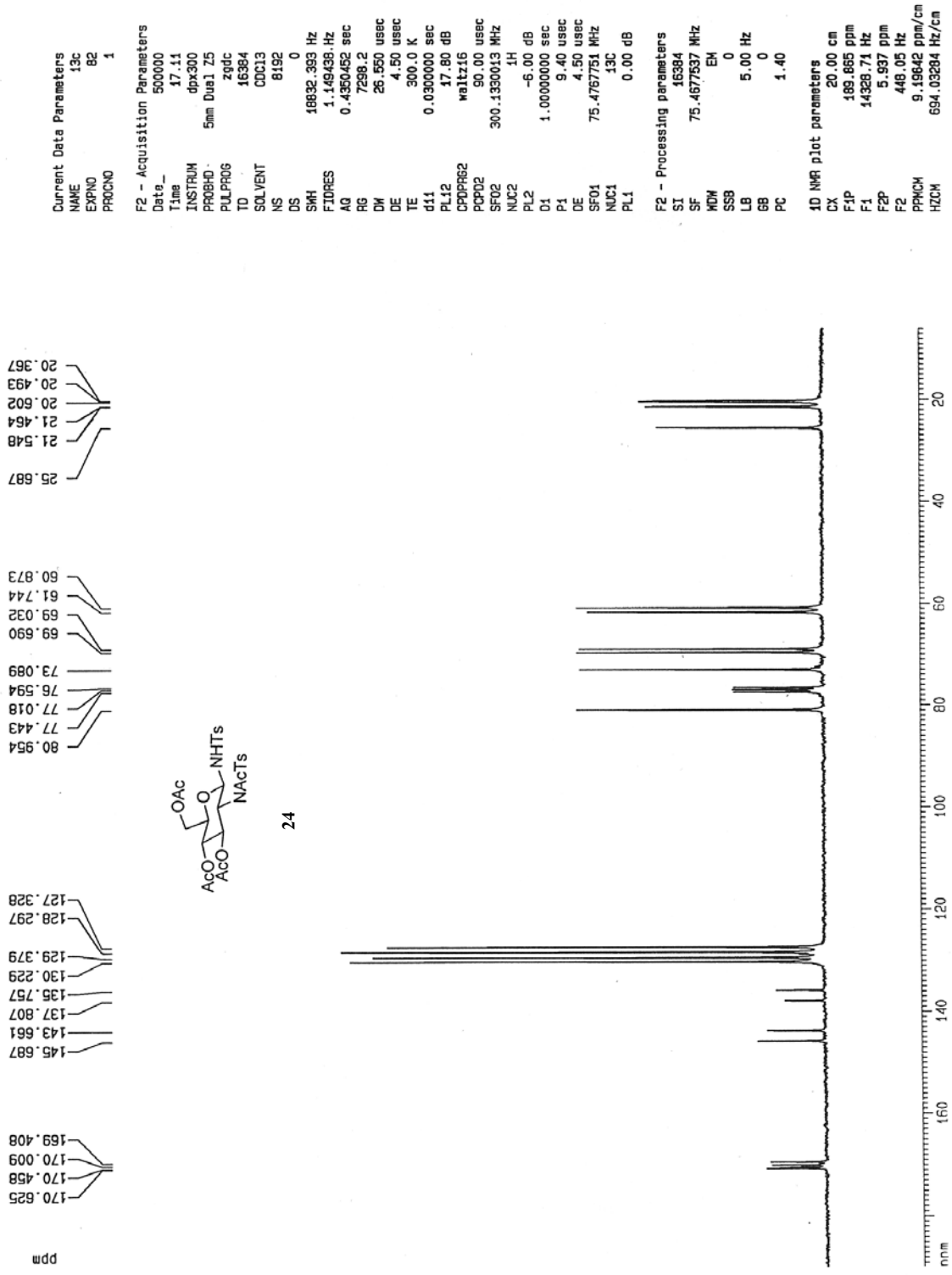

Current Data Parameters
NAME proton
EXPNO 218
PROCNO 1
F2 - Acquisition Parameters
Date_ 500000
Time 12.42
INSTRUM dpx300
PROBHD 5mm Dual 25
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 4
DS 0
SWH 8992.806 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 128
DW 55.600 usec
DE 4.50 usec
TE 300.0 K
D1 5.00000000 sec
P1 11.35 usec
DE 4.50 usec
SFO1 300.1342018 MHz
NUC1 1H
PL1 0.00 dB
F2 - Processing parameters
SI 32768
SF 300.1300050 MHz
WDW EM
SSB 0
LB 0.01 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm
0.0000
1.4925
1.9988
2.0615
2.4106
3.5956
3.6275
3.9249
3.9468
3.9925
4.7872
4.8146
4.8414
4.9046
4.9408
5.2836
5.5626
6.3054
6.3299
7.2667
7.2815
7.2924
7.7194
7.7457
7.8214
7.8480
2.9152
6.3068
6.3750
1.0707
3.1530
0.9960
1.0080
1.0946
0.9573
1.0000
4.9365
2.2621
2.1190
OAc
OAc
O
NHTs
NHTs
AcO
17
ppm
Integral

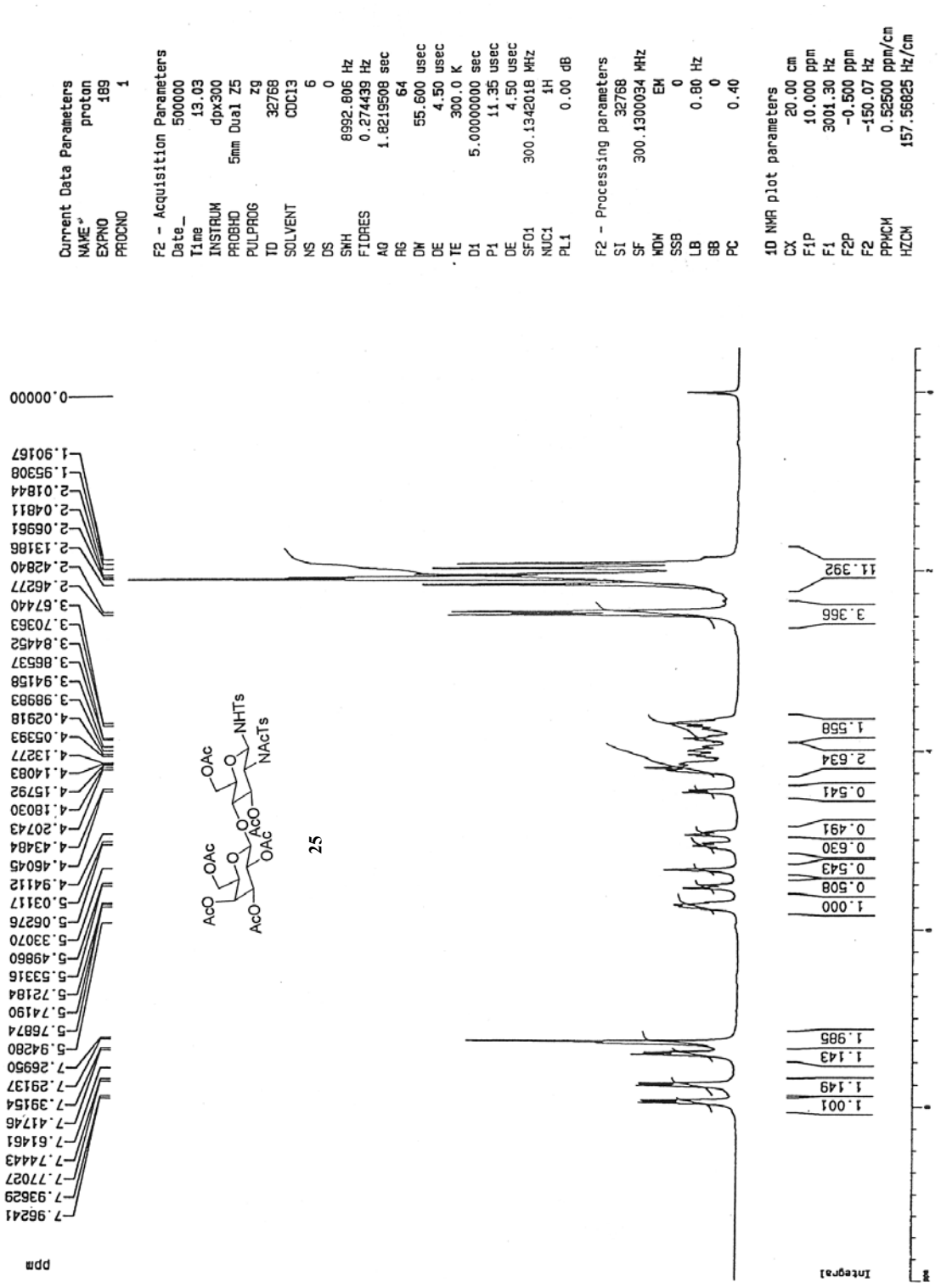

Current Data Parameters
NAME 13c
EXPNO 55
PROCNO 1
F2 - Acquisition Parameters
Date_ 500000
Time 11.49
INSTRUM dpx300
PROBHD 5mm Dual Z5
PULPROG zgdc
TD 16384
SOLVENT CDCl3
NS 987
DS 0
SWH 18832.393 Hz
FIDRES 1.149438 Hz
AQ 0.4350452 sec
RG 6502
DW 26.550 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
PL12 17.80 dB
CPDPRG2 waltz16
PCPD2 90.00 usec
SFO2 300.1330013 MHz
NUC2 1H
PL2 0.00 dB
D1 1.00000000 sec
P1 9.40 usec
DE 4.50 usec
SFO1 75.4767751 MHz
NUC1 13C
PL1 0.00 dB
F2 - Processing parameters
SI 16384
SF 75.4677526 MHz
WDW EM
SSB 0
LB 10.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 10.00000 ppm/cm
HZCM 754.67749 Hz/cm
170.356
169.877
143.672
138.294
137.739
129.550
129.368
127.180
83.778
77.431
77.011
76.587
71.685
70.791
66.821
61.145
53.014
21.452
20.473
20.021
OAc
OAc
O
NHTs
NHTs
AcO
17
ppm
20
40
60
80
100
120
140
160
180
ppm

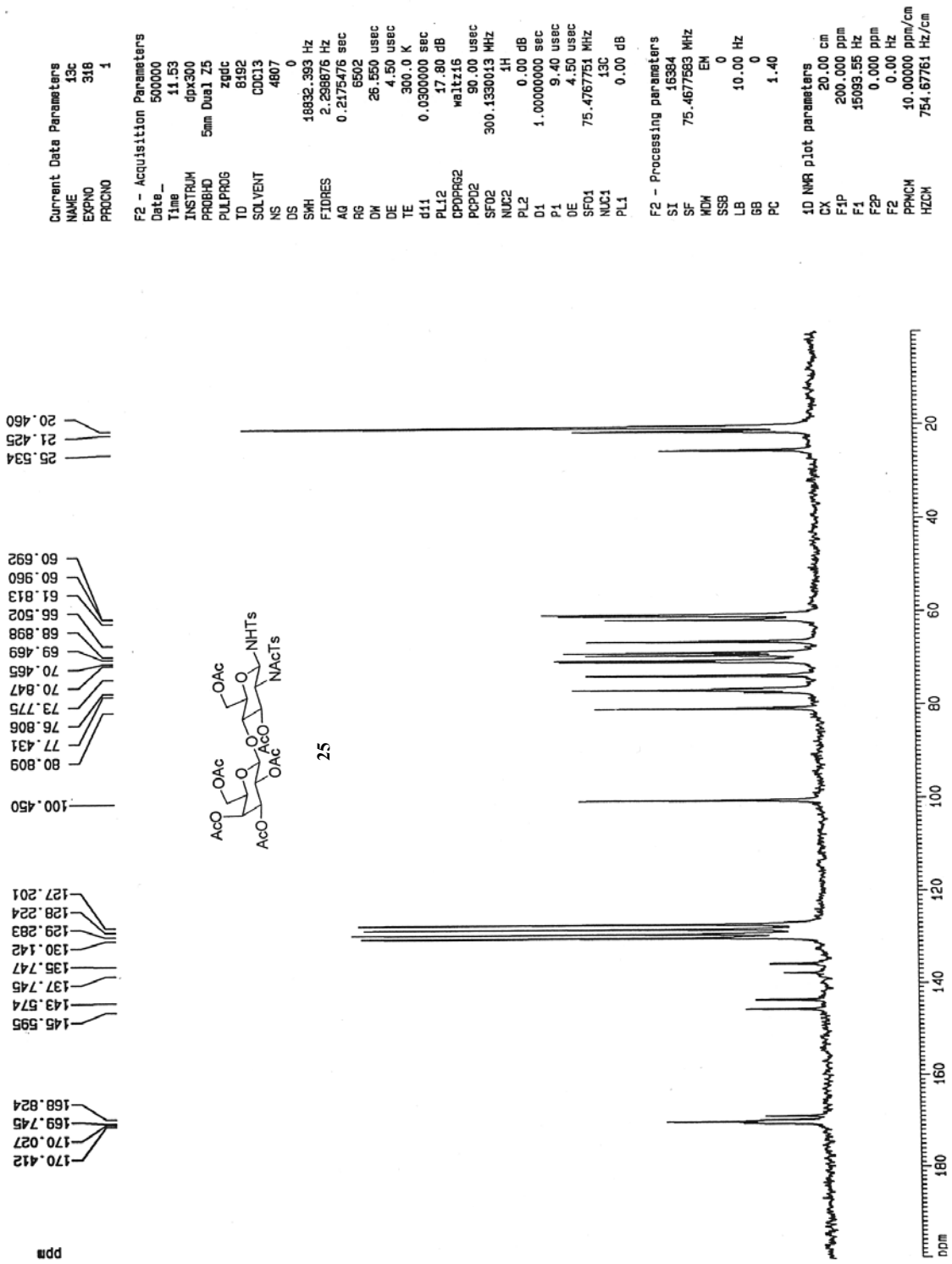

Current Data Parameters
NAME proton
EXPNO 169
PROCNO 1
F2 - Acquisition Parameters
Date_ 500000
Time 12.46
INSTRUM dpx300
PROBHD 5mm Dual Z5
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 8992.806 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 256
DW 55.600 usec
DE 4.50 usec
TE 300.0 K
D1 5.00000000 sec
P1 11.00 usec
DE 4.50 usec
SFO1 300.1342018 MHz
NUC1 1H
PL1 0.00 dB
F2 - Processing parameters
SI 32768
SF 300.1300060 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm
0.00000
1.56899
1.95335
2.02806
2.04059
2.09738
2.11290
2.43735
3.36529
3.56080
3.59838
3.62738
3.80333
3.82476
3.96104
3.98574
4.02340
4.05367
4.09015
4.31375
4.36734
4.39342
4.54491
4.84141
4.88520
4.89610
4.93145
4.96575
5.00578
5.03310
5.32278
6.35555
6.37867
7.26224
7.28447
7.31280
7.33996
7.36390
7.70264
7.72938
7.79848
7.82542
ppm
3.124
3.645
6.412
7.522
6.984
1.152
2.592
1.330
3.867
2.243
1.232
5.047
1.163
1.000
11.059
2.427
2.187
Integral
AcO
OAc
OAc
O
AcO
O
AcO
OAc
NHTs
NHTs

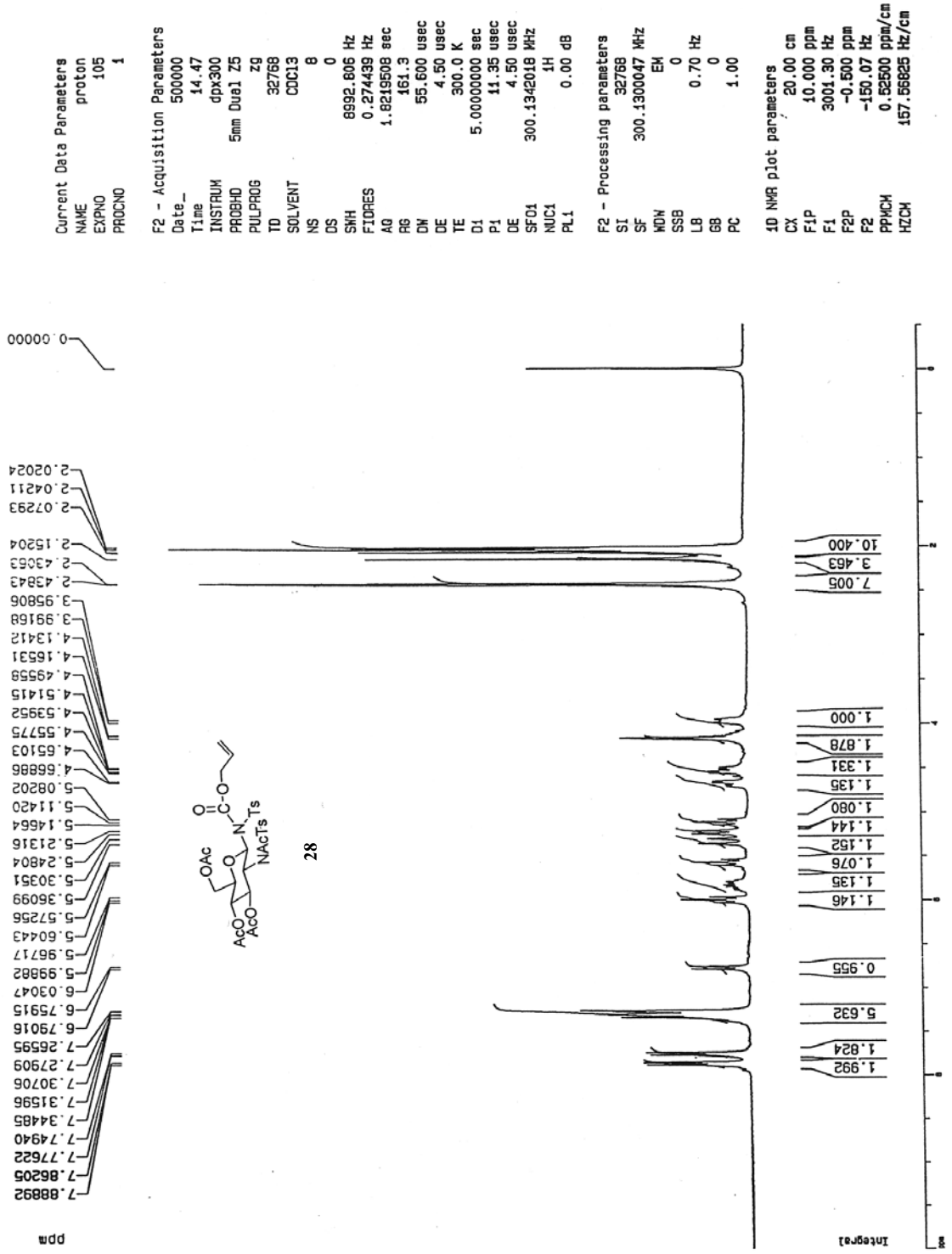

Current Data Parameters
NAME 13c
EXPNO 18
PROCNO 1
F2 - Acquisition Parameters
Date_ 500000
Time 14.01
INSTRUM dpx300
PROBHD 5mm Dual Z5
PULPROG zgdc
TD 16384
SOLVENT CDCl3
NS 7048
DS 0
SWH 18832.393 Hz
FIDRES 1.149438 Hz
AQ 0.4350452 sec
RG 5792.6
DW 26.550 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
PL12 17.80 dB
CPDPRG2 waltz16
PCPD2 90.00 usec
SFO2 300.1330013 MHz
NUC2 1H
PL2 0.00 dB
D1 1.00000000 sec
P1 9.40 usec
DE 4.50 usec
SFO1 75.4767751 MHz
NUC1 13C
PL1 0.00 dB
F2 - Processing parameters
SI 16384
SF 75.4677514 MHz
WDW EM
SSB 0
LB 10.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 10.00000 ppm/cm
HZCM 754.67749 Hz/cm
20.226
20.529
21.482
56.852
60.707
61.742
66.552
68.999
70.637
70.811
72.921
73.936
75.658
76.570
76.993
77.416
83.745
100.755
127.238
129.441
129.868
137.342
143.570
144.072
169.051
170.030
171.324
AcO
OAc
O
NHTs
19
ppm
nnm
20
40
60
80
100
120
140
160
180

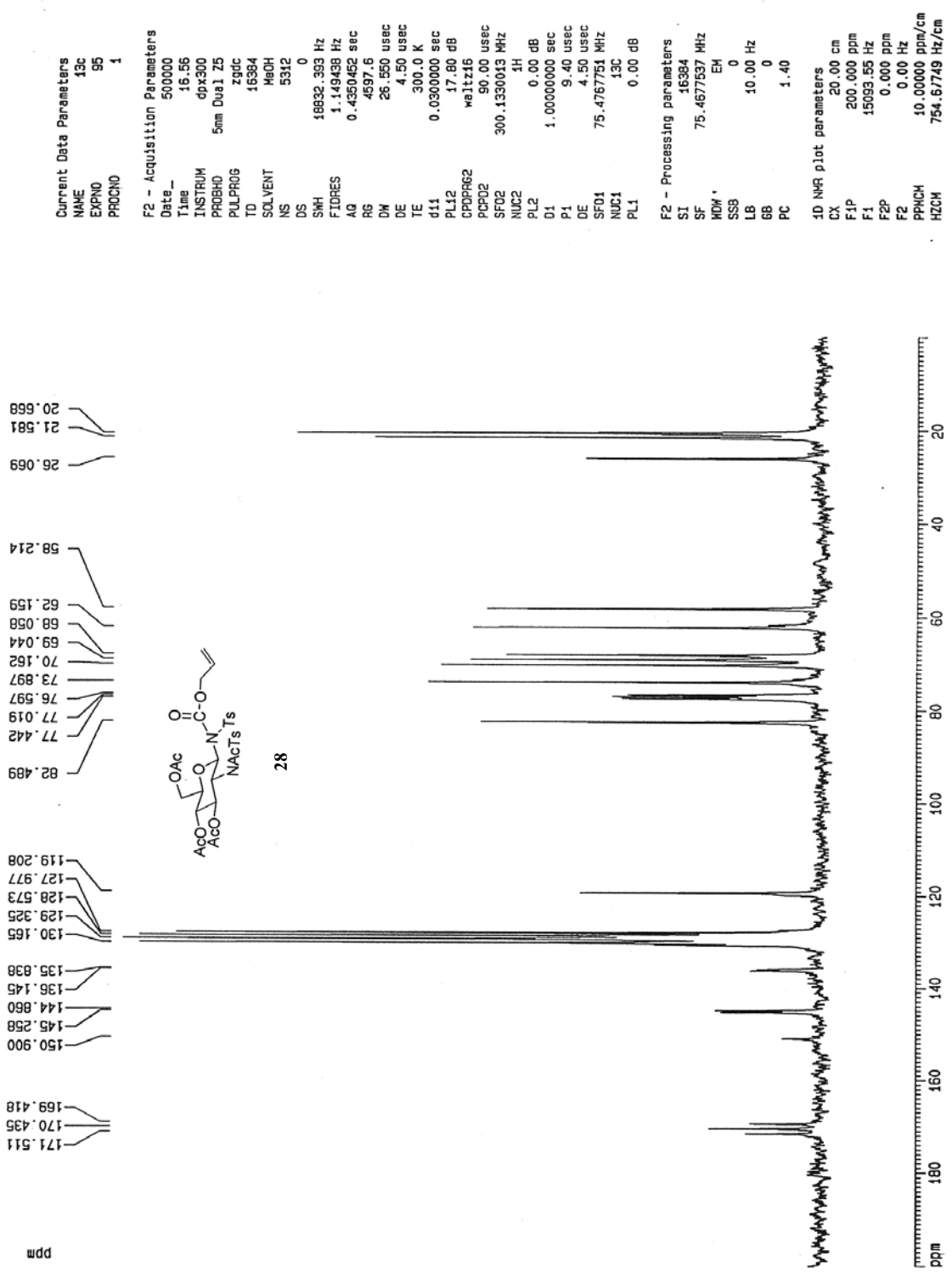

Current Data Parameters
NAME proton
EXPNO 236
PROCNO 1
F2 - Acquisition Parameters
Date_ 500000
Time 10.28
INSTRUM dpx300
PROBHD 5mm Dual Z5
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 10
DS 0
SWH 8992.806 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 57
DW 55.600 usec
DE 4.50 usec
TE 300.0 K
D1 5.00000000 sec
P1 11.00 usec
DE 4.50 usec
SFO1 300.1342018 MHz
NUC1 1H
PL1 0.00 dB
F2 - Processing parameters
SI 32768
SF 300.1300002 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.00 cm
F1P 8.616 ppm
F1 2555.89 Hz
F2P -0.378 ppm
F2 -113.44 Hz
PPMCM 0.40427 ppm/cm
HZCM 121.33337 Hz/cm
OAc
O
NHTs
NACTs
AcO
AcO
24
ppm
Integral

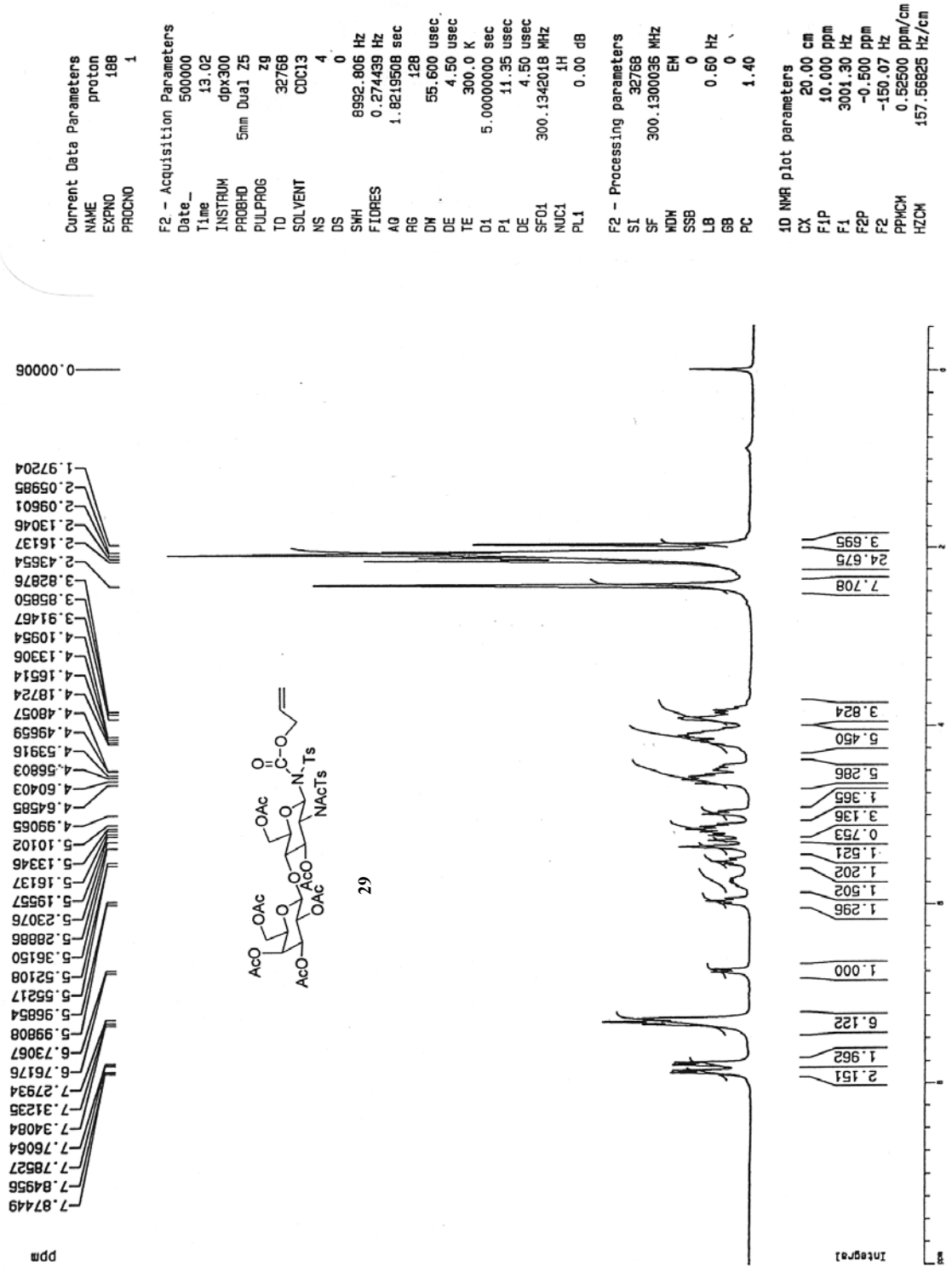

Current Data Parameters
NAME 13c
EXPNO 82
PROCNO 1
F2 - Acquisition Parameters
Date_ 500000
Time 17.11
INSTRUM dpx300
PROBHD 5mm Dual 25
PULPROG zgdc
TD 16384
SOLVENT CDCl3
NS 8192
DS 0
SWH 18832.393 Hz
FIDRES 1.149438 Hz
AQ 0.4350452 sec
RG 7298.2
DW 26.550 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
PL12 17.80 dB
CPDPRG2 waltz16
PCPD2 90.00 usec
SFO2 300.1330013 MHz
NUC2 1H
PL2 -6.00 dB
D1 1.00000000 sec
P1 9.40 usec
DE 4.50 usec
SFO1 75.4767751 MHz
NUC1 13C
PL1 0.00 dB
F2 - Processing parameters
SI 16384
SF 75.4677537 MHz
WDW EM
SSB 0
LB 5.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 189.865 ppm
F1 14328.71 Hz
F2P 5.937 ppm
F2 448.05 Hz
PPMCM 9.19842 ppm/cm
HZCM 694.03284 Hz/cm
20.367
20.493
20.602
21.464
21.548
25.687
60.873
61.744
69.032
69.690
73.089
76.594
77.018
77.443
80.954
127.328
128.297
129.379
130.229
135.757
137.807
143.661
145.687
169.408
170.009
170.458
170.625
OAc
AcO
AcO
O
NHTs
NACTs
24
ppm

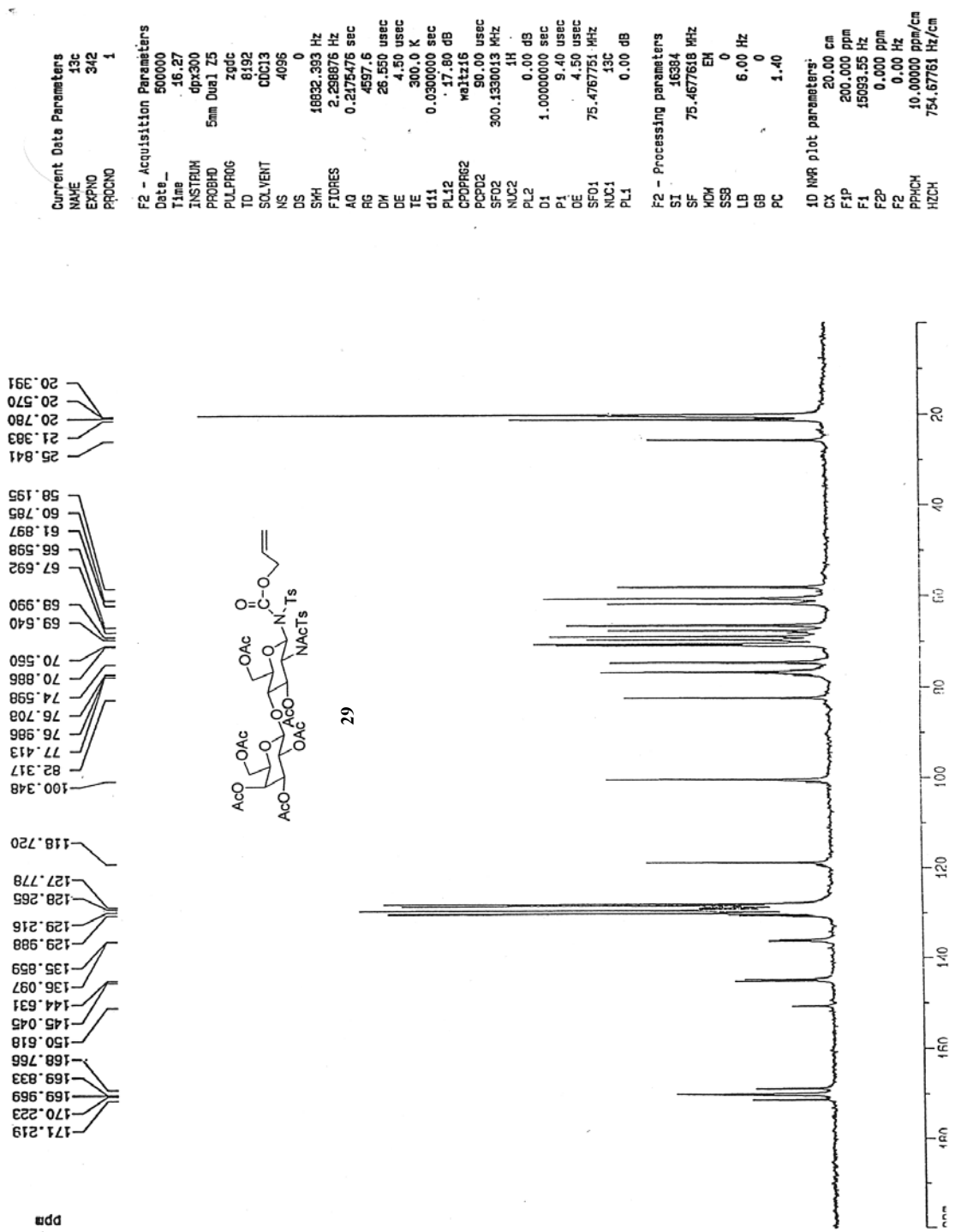

Current Data Parameters
NAME proton
EXPNO 189
PROCNO 1
F2 - Acquisition Parameters
Date_ 500000
Time 13.03
INSTRUM dpx300
PROBHD 5mm Dual Z5
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 6
DS 0
SWH 8992.806 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 64
DW 55.600 usec
DE 4.50 usec
TE 300.0 K
D1 5.00000000 sec
P1 11.35 usec
DE 4.50 usec
SFO1 300.1342018 MHz
NUC1 1H
PL1 0.00 dB
F2 - Processing parameters
SI 32768
SF 300.1300034 MHz
WDW EM
SSB 0
LB 0.80 Hz
GB 0
PC 0.40
1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm
11.392
3.366
1.558
2.634
0.541
0.491
0.630
0.543
0.508
1.000
1.985
1.143
1.149
1.001
Integral
ppm
AcO
OAc
AcO
OAc
AcO
OAc
NHTs
NAcTs
25


Current Data Parameters
NAME 13c
EXPNO 318
PROCNO 1
F2 - Acquisition Parameters
Date_ 500000
Time 11.53
INSTRUM dpx300
PROBHD 5mm Dual Z5
PULPROG zgdc
TD 8192
SOLVENT CDCl3
NS 4807
DS 0
SWH 18832.393 Hz
FIDRES 2.298876 Hz
AQ 0.2175476 sec
RG 6502
DW 26.550 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
PL12 17.80 dB
CPDPRG2 waltz16
PCPD2 90.00 usec
SFO2 300.1330013 MHz
NUC2 1H
PL2 0.00 dB
D1 1.00000000 sec
P1 9.40 usec
DE 4.50 usec
SFO1 75.4767751 MHz
NUC1 13C
PL1 0.00 dB
F2 - Processing parameters
SI 16384
SF 75.4677583 MHz
WDW EM
SSB 0
LB 10.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 10.00000 ppm/cm
HZCM 754.67761 Hz/cm
20.460
21.425
25.534
60.692
60.960
61.813
66.502
68.898
69.469
70.465
70.847
73.775
76.806
77.431
80.809
100.450
127.201
128.224
129.283
130.142
135.747
137.745
143.574
145.595
168.824
169.745
170.027
170.412
AcO
OAc
AcO
OAc
AcO
OAc
NHTs
NAcTs
25
ppm
20
40
60
80
100
120
140
160
180

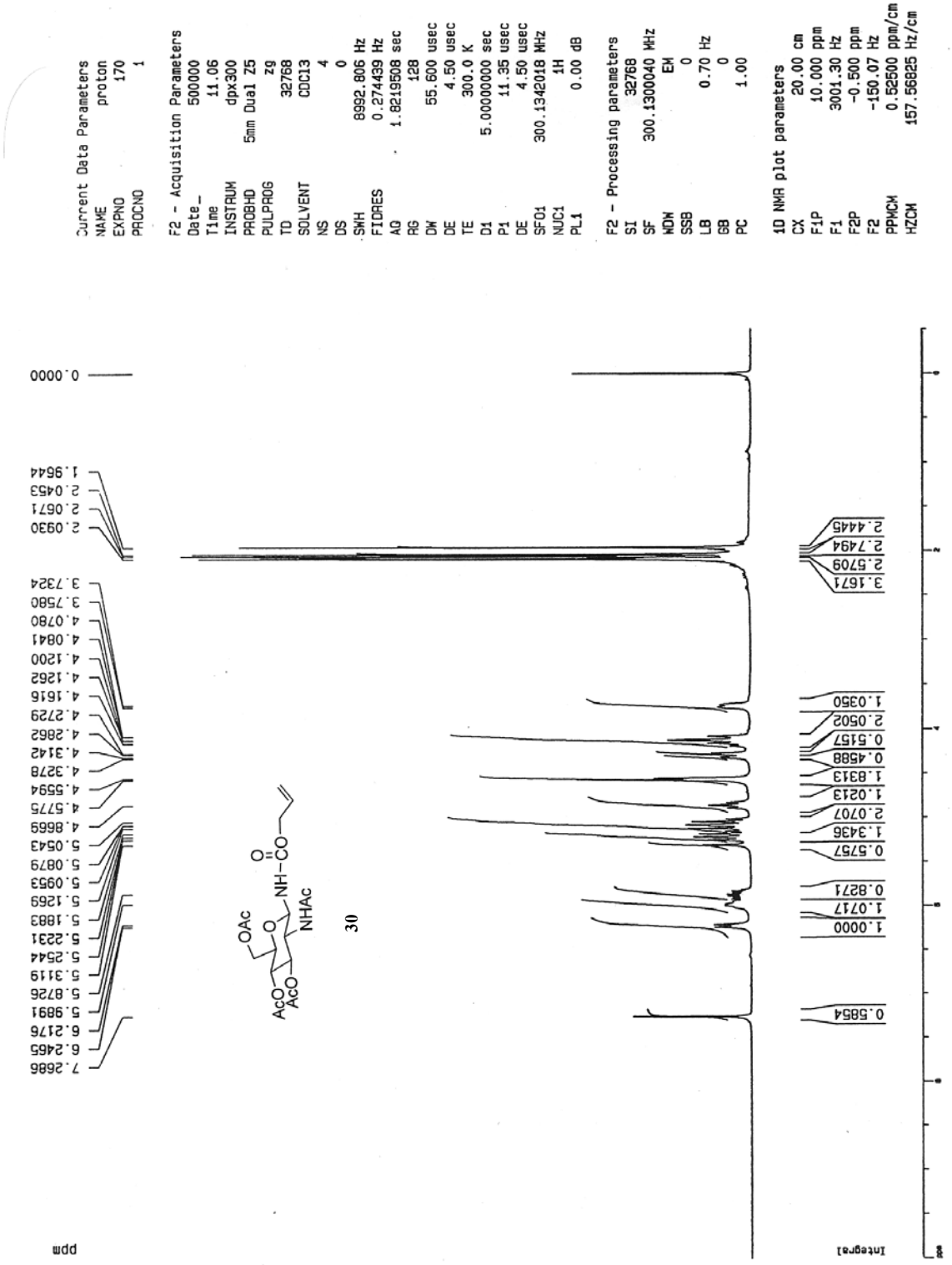

Current Data Parameters
NAME proton
EXPNO 105
PROCNO 1
F2 - Acquisition Parameters
Date_ 500000
Time 14.47
INSTRUM dpx300
PROBHD 5mm Dual Z5
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 8992.806 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 161.3
DW 55.600 usec
DE 4.50 usec
TE 300.0 K
D1 5.00000000 sec
P1 11.35 usec
DE 4.50 usec
SFO1 300.1342018 MHz
NUC1 1H
PL1 0.00 dB
F2 - Processing parameters
SI 32768
SF 300.1300047 MHz
WDW EM
SSB 0
LB 0.70 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm
ppm
Integral
28

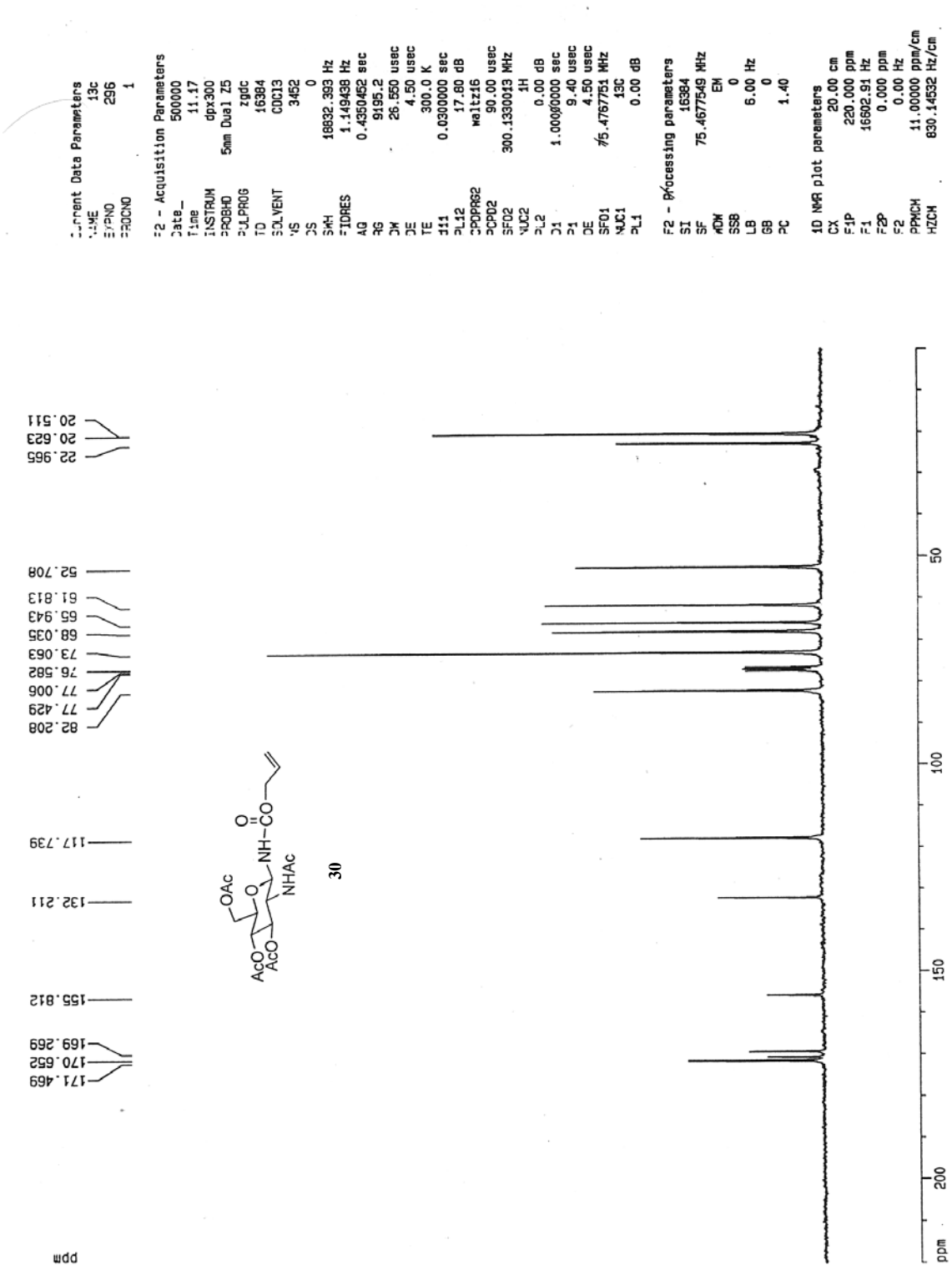

Current Data Parameters
NAME 13c
EXPNO 95
PROCNO 1
F2 - Acquisition Parameters
Date_ 500000
Time 16.56
INSTRUM dpx300
PROBHD 5mm Dual Z5
PULPROG zgdc
TD 16384
SOLVENT MeOH
NS 5312
DS 0
SWH 18832.393 Hz
FIDRES 1.149438 Hz
AQ 0.4350452 sec
RG 4597.6
DW 26.550 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
PL12 17.80 dB
CPDPRG2 waltz16
PCPD2 90.00 usec
SFO2 300.1330013 MHz
NUC2 1H
PL2 0.00 dB
D1 1.00000000 sec
P1 9.40 usec
DE 4.50 usec
SFO1 75.4767751 MHz
NUC1 13C
PL1 0.00 dB
F2 - Processing parameters
SI 16384
SF 75.4677537 MHz
WDW EM
SSB 0
LB 10.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 10.00000 ppm/cm
HZCM 754.6749 Hz/cm
20.668
21.581
26.069
58.214
62.159
68.058
69.044
70.162
73.897
76.597
77.019
77.442
82.489
119.208
127.977
128.573
129.325
130.165
135.838
136.145
144.860
145.258
150.900
169.418
170.435
171.511
OAc
AcO
AcO
NACTs
Ts
28
ppm

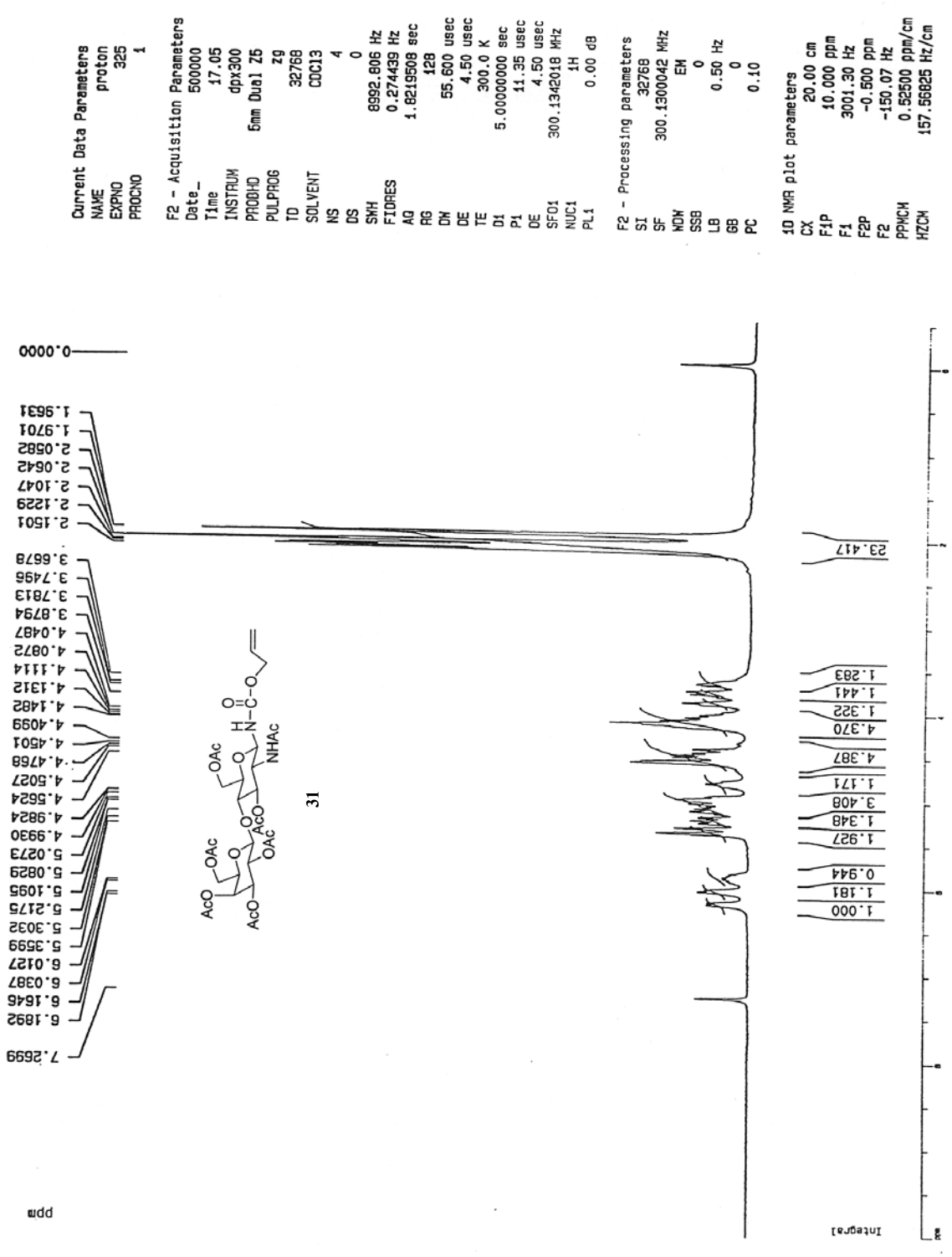

Current Data Parameters
NAME proton
EXPNO 188
PROCNO 1
F2 - Acquisition Parameters
Date_ 500000
Time 13.02
INSTRUM dpx300
PROBHD 5mm Dual Z5
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 4
DS 0
SWH 8992.806 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 128
DW 55.600 usec
DE 4.50 usec
TE 300.0 K
D1 5.00000000 sec
P1 11.35 usec
DE 4.50 usec
SFO1 300.1342018 MHz
NUC1 1H
PL1 0.00 dB
F2 - Processing parameters
SI 32768
SF 300.1300036 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm
0.00006
1.97204
2.05985
2.09601
2.13046
2.16137
2.43654
3.82876
3.85850
3.91467
4.10954
4.13306
4.16514
4.18724
4.48057
4.49659
4.53916
4.56803
4.60403
4.64585
4.99065
5.10102
5.13346
5.16137
5.19557
5.23076
5.28886
5.36150
5.52108
5.55217
5.96854
5.99808
6.73067
6.76176
7.27934
7.31235
7.34084
7.76064
7.78527
7.84956
7.87449
ppm
AcO
OAc
OAc
AcO
OAc
O
NAcTs
N
Ts
O
C
O
3.695
24.675
7.708
3.824
5.450
5.286
1.365
3.136
0.753
1.521
1.202
1.502
1.296
1.000
6.122
1.962
2.151
Integral

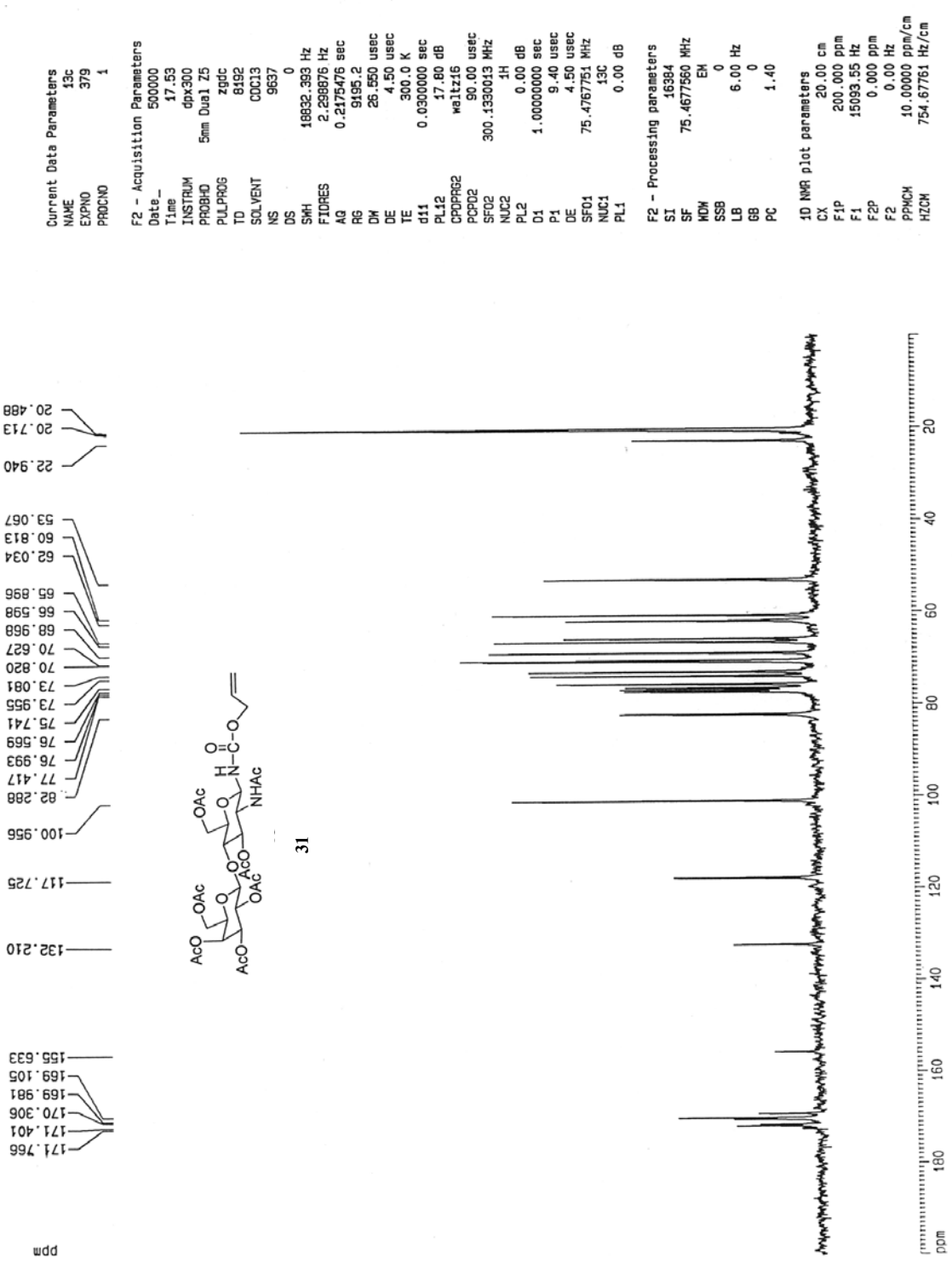

Current Data Parameters
NAME 13c
EXPNO 342
PROCNO 1
F2 - Acquisition Parameters
Date_ 500000
Time 16.27
INSTRUM dpx300
PROBHD 5mm Dual Z5
PULPROG zgdc
TD 8192
SOLVENT CDCl3
NS 4096
DS 0
SWH 18832.393 Hz
FIDRES 2.298876 Hz
AQ 0.2175476 sec
RG 4597.6
DW 26.550 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
PL12 17.80 dB
CPDPRG2 waltz16
PCPD2 90.00 usec
SFO2 300.1330013 MHz
NUC2 1H
PL2 0.00 dB
D1 1.00000000 sec
P1 9.40 usec
DE 4.50 usec
SFO1 75.4767751 MHz
NUC1 13C
PL1 0.00 dB
F2 - Processing parameters
SI 16384
SF 75.4677618 MHz
WDW EM
SSB 0
LB 6.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 10.00000 ppm/cm
HZCM 754.67761 Hz/cm
20.391
20.570
20.780
21.383
25.841
58.195
60.785
61.897
66.598
67.692
69.640
69.990
70.560
70.886
74.598
76.708
76.986
77.413
82.317
100.348
118.720
127.778
128.265
129.216
129.988
135.859
136.097
144.631
145.045
150.618
168.766
169.833
169.969
170.223
171.219
ppm
20
40
60
80
100
120
140
160
180
AcO
OAc
OAc
O
AcO
OAc
O
AcO
NAcTs
N
Ts
O
C
O
O

29

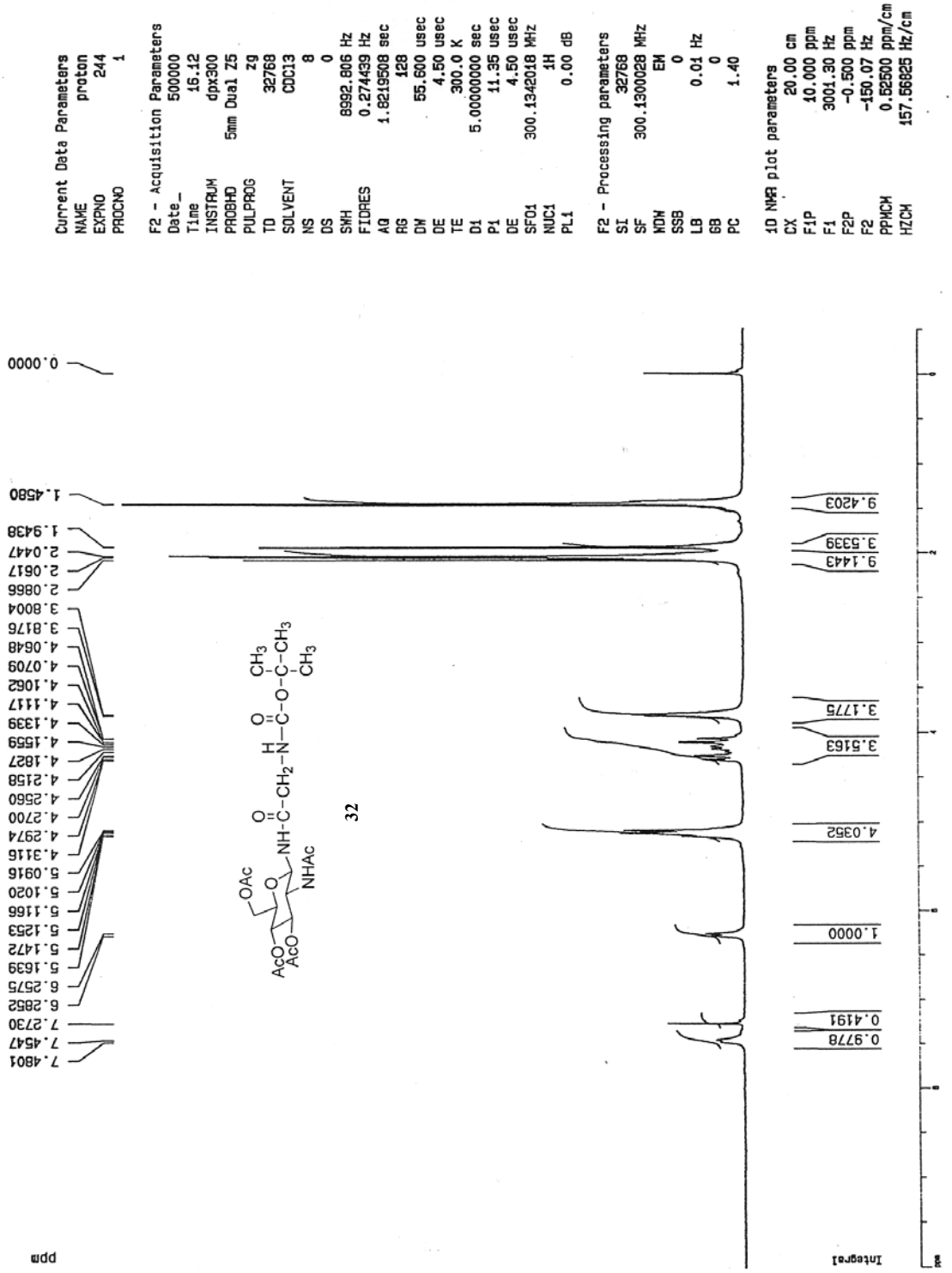

Current Data Parameters
NAME proton
EXPNO 170
PROCNO 1
F2 - Acquisition Parameters
Date_ 500000
Time 11.06
INSTRUM dpx300
PROBHD 5mm Dual Z5
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 4
DS 0
SWH 8992.806 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 128
DW 55.600 usec
DE 4.50 usec
TE 300.0 K
D1 5.00000000 sec
P1 11.35 usec
DE 4.50 usec
SFO1 300.1342018 MHz
NUC1 1H
PL1 0.00 dB
F2 - Processing parameters
SI 32768
SF 300.1300040 MHz
WDW EM
SSB 0
LB 0.70 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm
0.0000
1.9644
2.0453
2.0671
2.0930
3.7324
3.7580
4.0780
4.0841
4.1200
4.1262
4.1616
4.2729
4.2862
4.3142
4.3278
4.5594
4.5775
4.8669
5.0543
5.0879
5.0953
5.1269
5.1883
5.2231
5.2544
5.3119
5.8726
5.9891
6.2176
6.2465
7.2686
0.5854
1.0000
1.0717
0.8271
0.5757
1.3436
2.0707
1.0213
1.8313
0.4588
0.5157
2.0502
1.0350
3.1671
2.5709
2.7494
2.4445
OAc
AcO
AcO
NHAc
NH
C=O
O
30
ppm
Integral

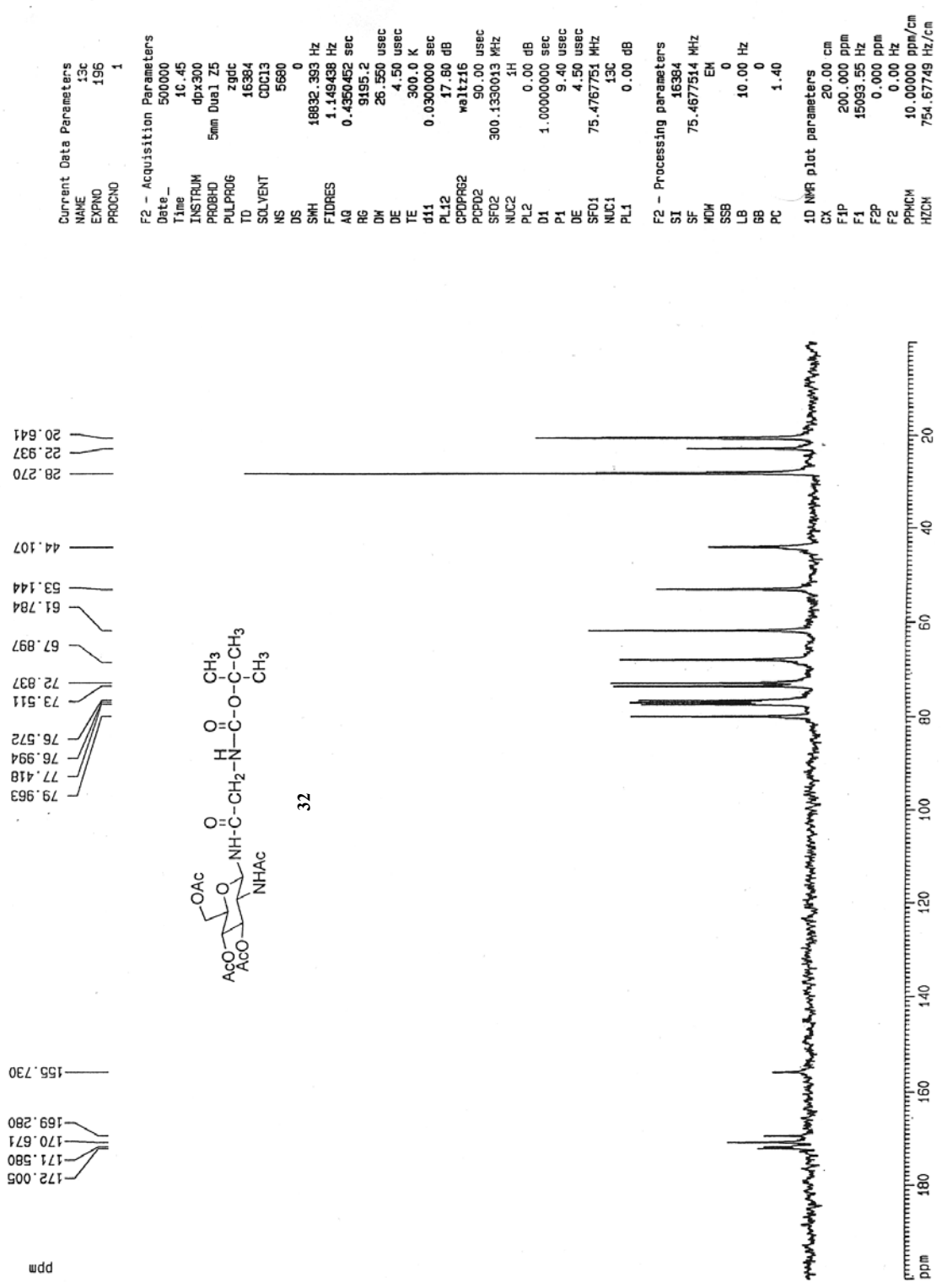

Current Data Parameters
NAME 13c
EXPNO 296
PROCNO 1
F2 - Acquisition Parameters
Data_ 500000
Time 11.17
INSTRUM dpx300
PROBHD 5mm Dual Z5
PULPROG zgdc
TD 16384
SOLVENT CDCl3
NS 3462
DS 0
SWH 18832.393 Hz
FIDRES 1.149438 Hz
AQ 0.4350462 sec
RG 9195.2
DW 26.550 usec
DE 4.50 usec
TE 300.0 K
D11 0.0300000 sec
PL12 17.80 dB
CPDPRG2 waltz16
PCPD2 90.00 usec
SFO2 300.1330013 MHz
NUC2 1H
PL2 0.00 dB
D1 1.0000000 sec
P1 9.40 usec
DE 4.50 usec
SFO1 75.4767751 MHz
NUC1 13C
PL1 0.00 dB
F2 - Processing parameters
SI 16384
SF 75.4677549 MHz
WDW EM
SSB 0
LB 6.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 220.000 ppm
F1 16602.91 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 11.00000 ppm/cm
HZCM 830.14532 Hz/cm
20.511
20.623
22.965
52.708
61.813
65.943
68.035
73.063
76.582
77.006
77.429
82.208
117.739
132.211
155.812
169.269
170.652
171.469
30
NHAc
NH
OAc
AcO
AcO
ppm
50
100
150
200


Current Data Parameters
NAME proton
EXPNO 325
PROCNO 1
F2 - Acquisition Parameters
Date_ 500000
Time 17.05
INSTRUM dpx300
PROBHD 5mm Dual Z5
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 4
DS 0
SWH 8992.806 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 128
DW 55.600 usec
DE 4.50 usec
TE 300.0 K
D1 5.00000000 sec
P1 11.35 usec
DE 4.50 usec
SFO1 300.1342018 MHz
NUC1 1H
PL1 0.00 dB
F2 - Processing parameters
SI 32768
SF 300.1300042 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 0.10
1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.52500 ppm/cm
HZCM 157.56825 Hz/cm
0.0000
1.9631
1.9701
2.0582
2.0642
2.1047
2.1229
2.1501
3.6678
3.7496
3.7813
3.8794
4.0487
4.0872
4.1114
4.1312
4.1482
4.4099
4.4501
4.4768
4.5027
4.5624
4.9824
4.9930
5.0273
5.0829
5.1095
5.2175
5.3032
5.3599
6.0127
6.0387
6.1646
6.1892
7.2699
23.417
1.283
1.441
1.322
4.370
4.387
1.171
3.408
1.348
1.927
0.944
1.181
1.000
AcO
OAc
AcO
OAc
AcO
OAc
AcO
NHAc
31
ppm
Integral


Current Data Parameters
NAME 13c
EXPNO 379
PROCNO 1
F2 - Acquisition Parameters
Date_ 500000
Time 17.53
INSTRUM dpx300
PROBHD 5mm Dual Z5
PULPROG zgdc
TD 8192
SOLVENT CDCl3
NS 9637
DS 0
SWH 18832.393 Hz
FIDRES 2.298876 Hz
AQ 0.2175476 sec
RG 9195.2
DW 26.550 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
PL12 17.80 dB
CPDPRG2 waltz16
PCPD2 90.00 usec
SFO2 300.1330013 MHz
NUC2 1H
PL2 0.00 dB
D1 1.00000000 sec
P1 9.40 usec
DE 4.50 usec
SFO1 75.4767751 MHz
NUC1 13C
PL1 0.00 dB
F2 - Processing parameters
SI 16384
SF 75.4677560 MHz
WDW EM
SSB 0
LB 6.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 10.00000 ppm/cm
HZCM 754.67761 Hz/cm
20.488
20.713
22.940
53.067
60.813
62.034
65.896
66.598
68.968
70.627
70.820
73.081
73.955
75.741
76.569
76.993
77.417
82.288
100.956
117.725
132.210
155.633
169.105
169.981
170.306
171.401
171.766
ppm
20
40
60
80
100
120
140
160
180
ppm
AcO
OAc
AcO
OAc
AcO
OAc
NHAc
O
H
N
C
O
O

31


Current Data Parameters
NAME proton
EXPNO 244
PROCNO 1
F2 - Acquisition Parameters
Date_ 500000
Time 15.12
INSTRUM dpx300
PROBHD 5mm Dual Z5
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 8992.806 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 128
DW 55.600 usec
DE 4.50 usec
TE 300.0 K
D1 5.00000000 sec
P1 11.35 usec
DE 4.50 usec
SFO1 300.1342018 MHz
NUC1 1H
PL1 0.00 dB
F2 - Processing parameters
SI 32768
SF 300.1300028 MHz
WDW EM
SSB 0
LB 0.01 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.62500 ppm/cm
HZCM 157.66825 Hz/cm
0.0000
1.4580
1.9438
2.0447
2.0617
2.0856
3.8004
3.8176
4.0648
4.0709
4.1062
4.1117
4.1339
4.1559
4.1827
4.2158
4.2560
4.2700
4.2974
4.3116
5.0916
5.1020
5.1166
5.1253
5.1472
5.1639
6.2575
6.2852
7.2730
7.4547
7.4801
9.4203
3.5339
9.1443
3.1775
3.5163
4.0352
1.0000
0.4191
0.9778
32
ppm
Integral


Current Data Parameters
NAME 13c
EXPNO 196
PROCNO 1
F2 - Acquisition Parameters
Date_ 500000
Time 10.45
INSTRUM dpx300
PROBHD 5mm Dual Z5
PULPROG zgdc
TD 16384
SOLVENT CDCl3
NS 5680
DS 0
SWH 18832.393 Hz
FIDRES 1.149438 Hz
AQ 0.4350452 sec
RG 9195.2
DW 26.550 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
PL12 17.80 dB
CPDPRG2 waltz16
PCPD2 90.00 usec
SFO2 300.1330013 MHz
NUC2 1H
PL2 0.00 dB
D1 1.00000000 sec
P1 9.40 usec
DE 4.50 usec
SFO1 75.4767751 MHz
NUC1 13C
PL1 0.00 dB
F2 - Processing parameters
SI 16384
SF 75.4677514 MHz
WDW EM
SSB 0
LB 10.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 10.00000 ppm/cm
HZCM 754.67749 Hz/cm
20.641
22.937
28.270
44.107
53.144
61.784
67.897
72.837
73.511
76.572
76.994
77.418
79.963
155.730
169.280
170.671
171.580
172.005
32
ppm
ppm