A Four-Component One-Pot Synthesis of α-Gal Pentasaccharide

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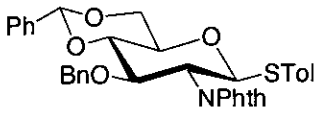
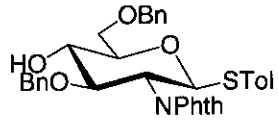
Supporting Information

Contents:



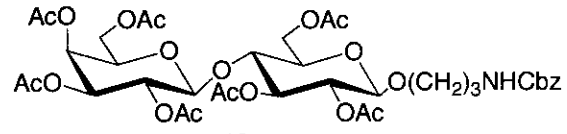
8

Compound 8. *p*-Methylphenyl 4,6-*O*-Benzylidene-2-deoxy-2-phthalimido-1-thio-β-D-glucopyranoside (70.0 mg. 0.14 mmol) and sodium hydride (95%, 7.0 mg, 0.28 mmol) were stirred in DMF (5 mL) at 0 °C for 10 min and benzyl bromide (25 μL, 0.21 mmol) was added. The mixture was stirred at room temperature for 4 h and then poured into ice water (10 mL), which was extracted with EtOAc (3 × 10 mL), then dried (Na_2SO_4). The organic phase was concentrated for column chromatography on silica gel (petroleum ether/EtOAc = 3:1). Saccharide **8** (74.5 mg, 92%) was obtained as a colorless foam. ^{1}H NMR (300 MHz, CD_3COCD_3-d_6) δ: 7.89 (m, 3H, aromatic), 7.72 (m, 1H, aromatic), 7.56 (dd, *J* = 2.4, 8.1 Hz, 2H, aromatic), 7.35-7.44 (m, 3H, aromatic), 7.25 (d, *J* = 8.1 Hz, 2H, aromatic), 7.09 (d, *J* = 8.1 Hz, 2H,aromatic), 6.87-6.99 (m, 5H, aromatic), 5.79 (s, 1H, benzylidene-C*H*), 5.63 (dd, *J* = 0.9, 10.5 Hz, 1H), 4.76 (d, *J* = 12.6 Hz, 1H), 4.50 (d, *J* = 12.3 Hz, 1H), 4.41 (q, *J* = 9.3 Hz, 1H), 4.36 (d, *J* = 10.2 Hz, 1H), 4.23 (t, *J* = 9.9 Hz, 1H), 3.86-3.93 (m, 2H), 3.77 (m, 1H), 2.27 (s, 3H, SPhC*H*$_3$); HRMS (M + Na) calcd for $C_{35}H_{31}O_6NSNa$ 616.1770, found 616.1789.



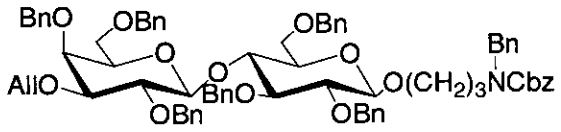
6

Compound 6. To a mixture of compound **8** (74.0 mg, 0.18 mmol), $NaCNBH_3$ (0.11 g, 1.625 mmol), and 3Å molecular sieves (2.5 g) in dry THF (10 mL), was added dropwise a solution of HCl-Et_2O (1M solution in ether, 2.5 mL, 2.5 mmol) under N_2 at room temperature. The mixture was stirred for 10 min, then filtered off through Celite. The filtrate was washed sequentially with saturated aqueous sodium bicarbonate and brine. The organic layer was dried (Na_2SO_4) and concentrated. The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 3:1). The product (66.7 mg, 90%) was obtained as a foam. ^{1}H NMR (300 MHz, CD_3COCD_3-d_6) δ: 7.69-7.84 (m, 4H, aromatic), 7.27-7.40 (m, 7H, aromatic), 6.88-7.01 (m, 7H, aromatic), 5.56 (d, *J* = 10.2 Hz, 1H), 4.83 (d, *J* = 12.0 Hz, 1H), 4.51-4.64 (m, 3H), 4.28 (dd, *J* = 7.8, 10.2 Hz, 1H), 4.16 (t, *J* = 10.2 Hz, 1H), 3.95 (d, *J* = 9.3 Hz, 1H), 3.74-3.82 (m, 2H), 3.32 (br.s, 2H), 2.22 (s, 3H, SPhC*H*$_3$); HRMS (M + Na) calcd for $C_{35}H_{33}O_6NSNa$ 618.1926, found 618.1952.



10

Compound 10. Compound **9** (200 mg, 0.3 mmol) and benzyl *N*-(3-hydroxypropyl)-carbamate (63.0 mg, 0.35 mmol) were stirred in dry CH_2Cl_2 (15 mL) and $BF_3{\cdot}Et_2O$ (1.01 mL, 0.90 mmol) was added. The reaction mixture was stirred at room temperature for 8 h and then diluted with CH_2Cl_2 (50 mL), washed sequentially with sat. aq. sodium bicarbonate and brine. The organic layer was dried (Na_2SO_4), filtered, and concentrated. The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 1:1). The product (98.4 mg, 43%) was obtained as a yellow syrup. ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.24-7.32 (m, 5H, aromatic), 5.31 (d, J = 2.7 Hz, 1H), 5.16 (t, J = 9.6 Hz, 1H), 5.05-5.11 (m, 3H), 4.93 (dd, J = 3.6, 10.5 Hz, 1H), 4.85 (dd, J = 7.8, 9.6 Hz, 1H), 4.42-4.51 (m, 3H), 3.97-4.13 (m, 4H), 3.79-3.86 (m, 2H), 3.75 (t, J = 9.6 Hz, 1H), 3.52-3.59 (m, 2H), 3.14-3.27 (m, 2H), 2.11 (s, 3H, $COCH_3$), 2.05 (s, 3H, $COCH_3$), 2.02 (s, 3H, $COCH_3$), 2.01 (s, 3H, $COCH_3$), 1.98 (s, 3H, $COCH_3$), 1.93 (s, 3H, $COCH_3$), 1.89 (s, 3H, $COCH_3$), 1.75 (m, 2H, $OCH_2CH_2CH_2NH$); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 170.8, 170.7, 170.5, 170.4, 170.1, 169.4, 156.8, 137.0, 128.9, 128.5, 128.4, 101.4, 100.8, 76.6, 73.1, 73.1, 72.0, 71.3, 71.1, 69.5, 67.9, 67.0, 62.2, 61.2, 60.8, 38.6, 29.80, 21.2, 21.0, 20.9; FAB-MS $(M + H)^+$ 828. Anal. Calcd for $C_{37}H_{49}O_{20}N$: C, 53.69; H, 5.97; N, 1.69. Found: C, 53.59; H, 5.96; N, 1.66.

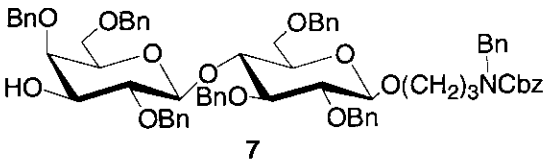


12

Compound 12. To a stirred solution of **10** (107.5 mg, 0.13 mmol) in methanol (20 mL), NaOMe (30% in MeOH, 10 μL) was added. The reaaction mixture was stirred at room temperature for 5 h and then neutralized with cation exchange resin (H^+). The resin was filtered off and the filtrate was concentrated. The residue in dry methanol (20 mL) was refluxed under N_2 for 16 h in the presence of Bu_2SnO (58.0 mg, 0.023 mmol) and the solution was concentrated under reduced pressure. The residue was dissolved in dry benzene (20 mL) under N_2, followed by the addition of tetrabutylammoniun iodide (2.2 mg, 0.006 mmol), and 4Å MS (2.0 g), then allyl bromide (18 μL, 25.2 mg, 0.208 mmol) was added to the mixture. The mixture was refluxed for 5 h and then concentrated. Dry methanol (20 mL) and Bu_2SnO (58.0 mg, 0.023 mmol) were added to the residue and the mixture was refluxed for 16 h and concentrated. Then dry benzene (20 mL), tetrabutylammoniun iodide (2.2 mg, 0.006 mmol), 4Å MS (2.0 g) and allyl bromide (18 μL, 25.2 mg, 0.208 mmol) were added, and the mixture was again refluxed for 5 h. The volatiles were evaporated and the resulting residue was purified by column chromatography on silica gel (MeOH/EtOAc = 1:15) to get a yellow syrupy crude product.

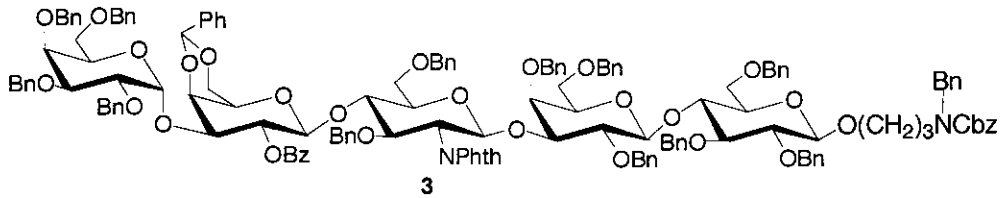
The crude product and sodium hydride (95%, 39.0 mg, 1.56 mmol) were stirred in DMF (4 mL) at 0 °C for 10 min and benzyl bromide (0.14 mL, 200 mg, 1.17 mmol) was added. The mixture was stirred at room temperature for 5 h and then poured into ice water (20 mL), which was extracted with EtOAc (3 × 10 mL), then dried (Na_2SO_4). The organic phase was concentrated for column chromatography on silica gel (petroleum ether/EtOAc = 3:1). Compound **12** (0.111 g, 70% over three steps) was obtained as a yellow syrup. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.12-7.34 (m, 40H, aromatic), 5.92 (m, 1H, CH_2=$CHCH_2O$), 5.32 (dd, J = 1.5, 17.0 Hz, 1H,

H^a-CH_2=$CHCH_2O$), 5.15-5.18 (m, 3H), 4.99 (d, J = 11.0 Hz, 1H), 4.95 (d, J = 11.5 Hz, 1H), 4.66-4.81 (m, 5H), 4.14-4.55 (m, 11H), 3.91 (t, J = 10.0 Hz, 2H), 3.86 (d, J = 2.5 Hz, 1H), 3.76 (m, 1H), 3.68 (dd, J = 7.5, 8.6 Hz, 2H), 3.53 (t, J = 10 Hz 2H), 3.29-3.35 (m, 8H), 1.85 (m, 2H, $OCH_2CH_2CH_2N$); ^{13}C NMR (75 MHz , $CDCl_3$) δ: 162.6, 139.1, 139.1, 138.9 , 138.6, 138.3, 138.1, 137.9, 135.0, 128.5, 128.4, 128.3, 128.2, 128.2, 128.1, 128.0, 127.9, 127.9, 127.8, 127.7, 127.6, 127.5, 127.5, 127.4, 127.4, 127.3, 127.0, 116.4, 103.5, 102.7, 82.9, 82.3, 81.7, 79.9, 77.2, 76.6, 75.3, 75.2, 75.1, 74.9, 74.6, 73.5, 73.4, 73.0, 72.9, 71.5, 68.3, 68.1, 67.2, 50.8, 36.6, 31.6; MALDI-TOF-MS $(M + Na + H)^+$ 1227, $(M + K + H)^+$ 1243. Anal. Calcd for $C_{75}H_{81}O_{13}N$: C, 74.79; H, 6.78; N, 1.16. Found: C, 74.72; H, 6.68; N, 1.08.



7

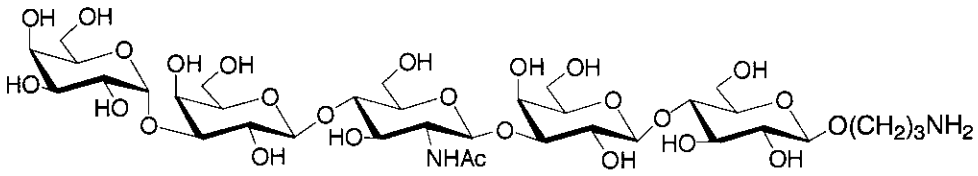
Compound 7. Saccharide **12** (21.0 mg, 0.017 mmol) and $PdCl_2$ (1.1 mg, 0.0068 mmol) were stirred in dry methanol (1.5 mL) under N_2 for 4 h. The reaction mixture was diluted with methanol (5 mL) and filtered. After removal of the solvent, the residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 1:2). The product (19.8 mg, 98%) was obtained as a yellow syrup. ^{1}H NMR (500 MHz, $CDCl_3$) δ:7.14-7.34 (m, 40H, aromatic), 5.15 (br.s, 2H), 5.00 (d, J = 10.5 Hz, 1H), 4.25-4.81 (m, 16H), 3.95 (t, J = 9.5 Hz, 1H), 3.86-3.92 (m, 1H), 3.83 (br.s, 1H), 3.66-3.79 (m, 2H), 3.26-3.56 (m, 10H), 2.18(d, J = 3.5 Hz, 1H), 1.85 (m, 2H, $OCH_2CH_2CH_2N$); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 157.9 , 139.0, 138.6, 138.5, 138.4, 138.1, 137.9, 137.8, 136.6, 128.9, 128.4, 128.4, 128.3, 128.2, 128.0, 127.9 , 127.8, 127.8, 127.7, 127.6, 127.5, 127.5, 127.1, 103.4, 102.6, 82.7, 81.6 ,80.5, 75.8, 75.3, 75.0, 74.9, 74.0, 73.3, 73.1, 73.0, 68.1, 67.9, 67.1, 50.7, 44.7, 43.6, 28.6; MALDI-TOF-MS $(M + Na + H)^+$ 1187, $(M + K + H)^+$ 1203. Anal. Calcd for $C_{72}H_{77}O_{13}N$: C, 74.27; H, 6.66; N, 1.20. Found: C, 74.10; H, 6.61; N, 1.12.



3

Compound 3. Building block **4** (100 mg, 0.155 mmol), building block **5** (74 mg, 0.155 mmol), BSP (16.2 mg, 0.077 mmol), and 4Å MS (500 mg) were stirred in dry CH_2Cl_2 (5 mL) at room temperature for 0.5 h under N_2. The mixture was cooled to –70 °C, followed by the addition of Tf_2O (15.7 μL, 26.2 mg, 0.093 mmol) and the temperature was increased gradually to room temperature. After 2 h, the donor was consumed and the reaction temperature was cooled to –70 °C again, followed by the addition of the solution of building block **6** (92 mg, 0.155 mmol) and BSP (16.2 mg,, 0.077 mmol) in dry CH_2Cl_2 (5 mL). Subsequently Tf_2O (15.7 μL, 26.2 mg, 0.093 mmol) was added to the reaction mixture. The temperature was then increased gradually to room

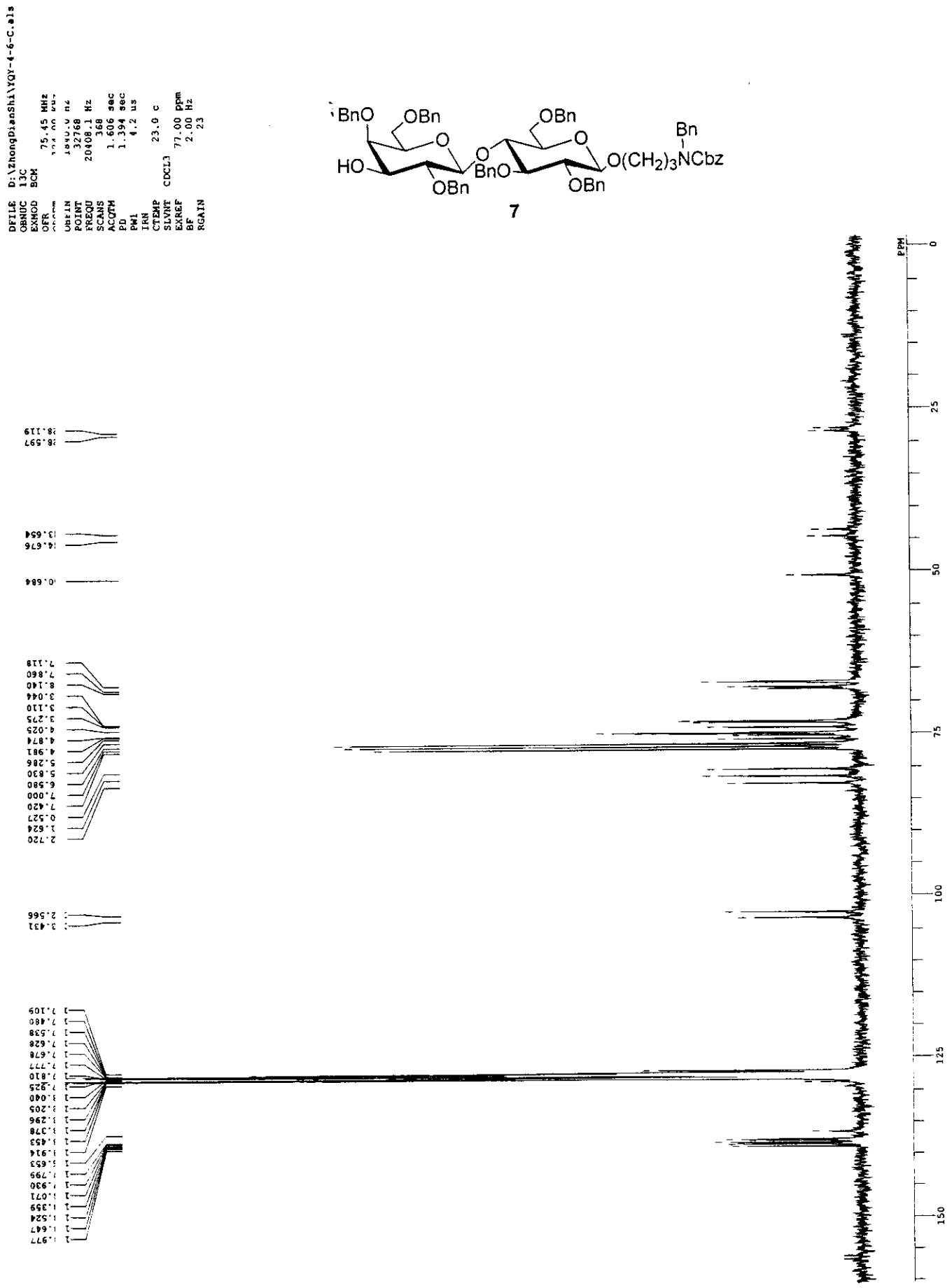
temperature. After 2 h, TLC detection showed the product last step produced had disappeared, and the reaction temperature was cooled to –70 °C before the solution of building block **7** (180 mg, 0.155 mmol) and BSP (26 mg,, 0.124 mmol) in dry CH_2Cl_2 (5 mL) was added to the reaction mixture. Then Tf_2O (23.5 μL, 39.3 mg, 0.139 mmol) was added to the stirring mixture. The temperature was increased to room temperature in 2 h. After stirred for another 2 h, the reaction was quenched with triethylamine (5 mL) and diluted with CH_2Cl_2 (20 mL). The reaction mixture was filtered and washed sequentially with sat. aq. sodium bicarbonate and brine, dried (Na_2SO_4), filtered, and concentrated. The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 3:1). The product (153 mg, 39%) was obtained as a yellow syrup. ^{1}H NMR (500 MHz, DMSO-d_6) δ: 7.97 (d, *J* = 7.5 Hz, 2H, aromatic), 7.63-7.73 (m, 6H, aromatic), 7.44 (t, *J* = 7.5 Hz, 2H, aromatic), 7.37-7.02 (m, 67H, aromatic), 6.86 (d, *J* = 7.5 Hz, 3H, aromatic), 6.74 (t, *J* = 7.5 Hz, 3H, aromatic), 5.55 (s, 1H, benzylidene-C*H*), 5.40 (dd, *J* = 8.0, 10.0 Hz, 1H, H-2'''), 5.32 (d, *J* = 3.0 Hz, 1H, H-1''''), 5.28 (d, *J* = 8.0 Hz, 1H, anomeric), 5.05 (s, 2H), 4.90 (d, *J* = 11.5 Hz, 1H), 4.87 (d, *J* = 8.0 Hz, 1H), 4.75 (d, *J* = 11.0 Hz, 1H), 3.92-4.65 (m, 36H), 33.76-3.80 (m, 3H), 3.18-3.76 (m, 16H), 2.95-3.14 (m, 2H), 1.66-1.75 (m, 2H, $OCH_2CH_2CH_2N$); ^{13}C NMR (125 MHz, DMSO-d_6) δ: 167.4, 164.5, 155.8, 139.1, 138.9, 138.6, 138.3, 138.2, 138.0, 137.9, 137.8, 136.9, 134.4, 133.6, 131.5, 130.5, 129.4, 128.9, 128.8, 128.6, 128.4, 128.3, 128.2, 128.1, 128.0, 127.9, 127.8, 127.7, 127.6, 127.5, 127.4, 127.3, 127.2, 127.1, 127.0, 126.9, 126.7, 126.3, 126.2, 123.1, 102.2, 101.2, 100.0, 99.9, 99.0, 93.3, 81.8, 81.0, 78.1, 77.7, 77.4, 76.8, 76.4, 75.1, 74.4, 74.2, 74.1, 74.0, 73.7, 73.6, 73.4, 73.3, 72.5, 72.4, 72.3, 71.9, 71.2, 70.8, 69.0, 68.4, 68.2, 68.0, 67.9, 67.4, 66.3, 66.1, 65.0, 55.8, 50.0, 48.6, 44.5, 43.6, 30.0; MALDI-TOF-MS $(M + Na + H)^+$ 2534, $(M + K + H)^+$ 2550.



2

Compound 2. Compound **3** (54.0 mg, 0.02 mmol) was dissolved in EtOH (3 mL) and treated with $NH_2NH_2 \cdot H_2O$ (1 mL). After the mixture had been heated under reflux for 20 h, it was concentrated under reduced pressure and the residue was co-evaporated with toluene (3 × 5 mL). The crude product was dissolved in pyridine (3 mL) and acetic anhydride (1 mL). After stirring for 8 h, the solvent was removed in a vacuum. The residue was dissolved in methanol (3 mL), and NaOMe (30% in MeOH, 10 μL) was added. The mixture was stirred for 6 h and then neutralized with cation exchange resin (H^+). The resin was filtered off and the filtrate was evaporated in a vacuum. The residue was purified by silica gel column chromatography (petroleum ether/EtOAc = 2:1). A mixture of the purified residue, 10% Pd-C (15.0 mg) in HOAc (0.25 mL), THF (2 mL), H_2O (1 mL) was stirred under H_2 atmosphere for 22 h. The catalyst was then removed by filtration and the filtrate was concentrated. The residue was subjected to a C-18 reverse phase column chromatography (H_2O) to give **2** (9.0 mg, 43%) as a white solid after lyophilization. ^{1}H NMR (500 MHz, D_2O) δ: 5.14 (d, *J* = 4.0 Hz, 1H, H-1''''), 4.70 (d, *J* = 8.5 Hz, 1H, H-1'''), 4.54 (d, *J* = 8.0 Hz, 1H, H-1'), 4.50 (d, *J* = 8.0 Hz, 1H, H-1), 4.42 (d, *J* = 8.0 Hz, 1H, H-1''), 4.20-4.16 (m, 2H),

4.15 (d, J = 3.0 Hz, 1H), 4.07-3.93 (m, 5H), 3.87-3.69 (m, 14H), 3.67-3.56 (m, 6H), 3.36-3.30 (m, 2H, H-6), 3.15 (t, J = 7.0 Hz, 2H, $OCH_2CH_2CH_2N$), 2.03 (s, 3H, $NHCOCH_3$) 2.00 (m, 2H, $OCH_2CH_2CH_2N$), 1.90 (s, 7H, CH_3COOH); ^{13}C NMR (125 MHz, D_2O) δ: 175.6 ($NHCOCH_3$), 103.7 (C-1''), 103.5 (C-1'), 103.4 (C-1'''), 102.8 (C-1), 96.2 (C-1''''), 82.8, 79.1 (C-6), 79.0, 77.9, 75.8, 75.6, 75.5, 75.3, 75.1, 73.4 (C-6''), 73.0, 71.6, 70.7, 70.3, 70.0, 69.8, 69.0, 68.9, 68.6 ($OCH_2CH_2CH_2N$), 65.5, 61.7, 61.6, 61.5, 60.7, 60.6, 55.9, 38.3 ($OCH_2CH_2CH_2N$), 27.5 ($OCH_2CH_2CH_2N$), 24.0, 22.9 ($NHCOCH_3$); HRMS (M + H) calcd for $C_{35}H_{63}O_{26}N_2$ 927.3664, found 927.3672.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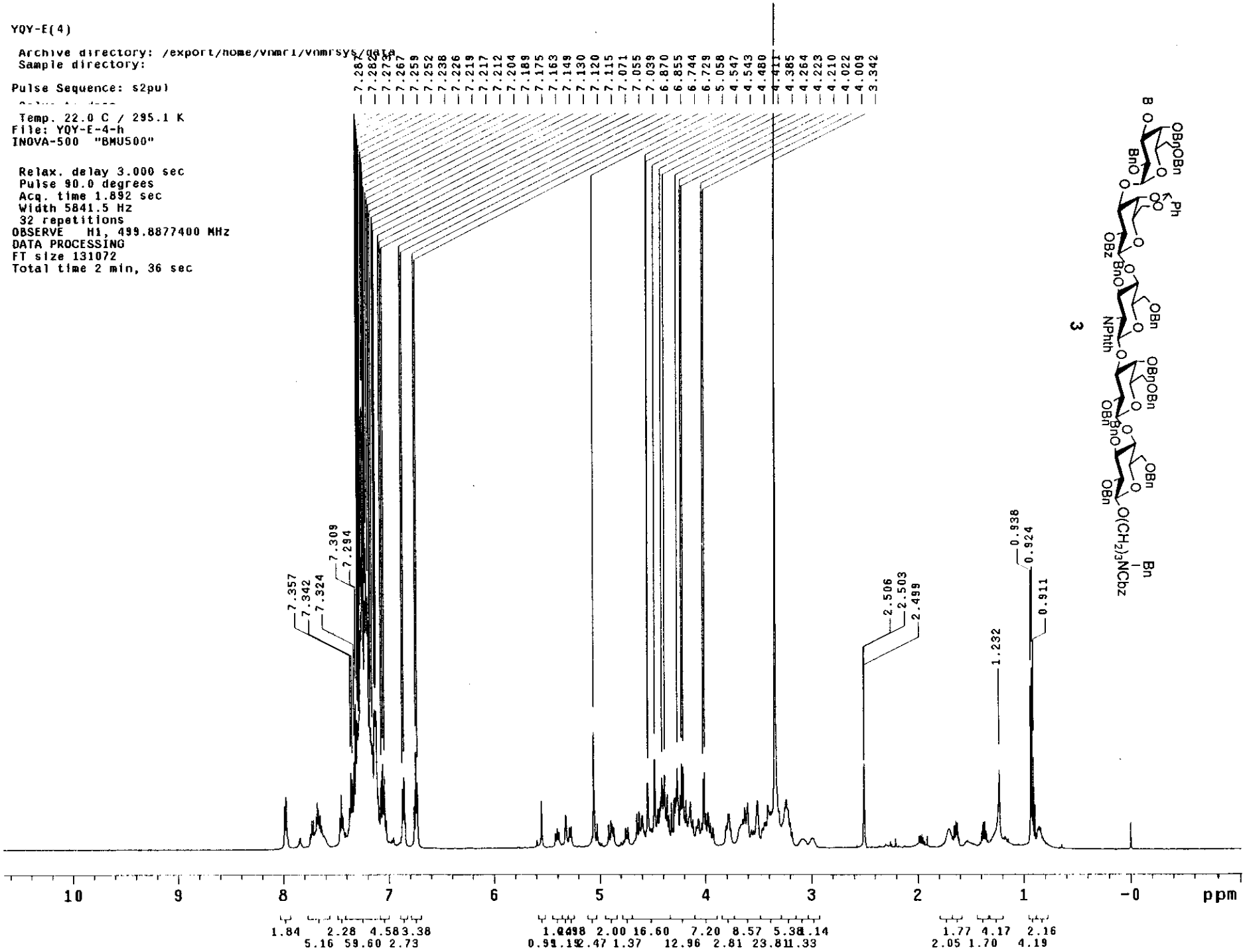
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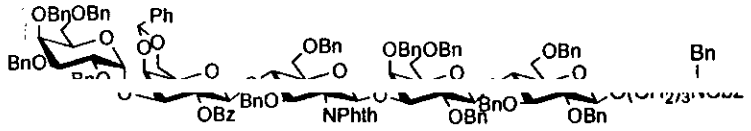

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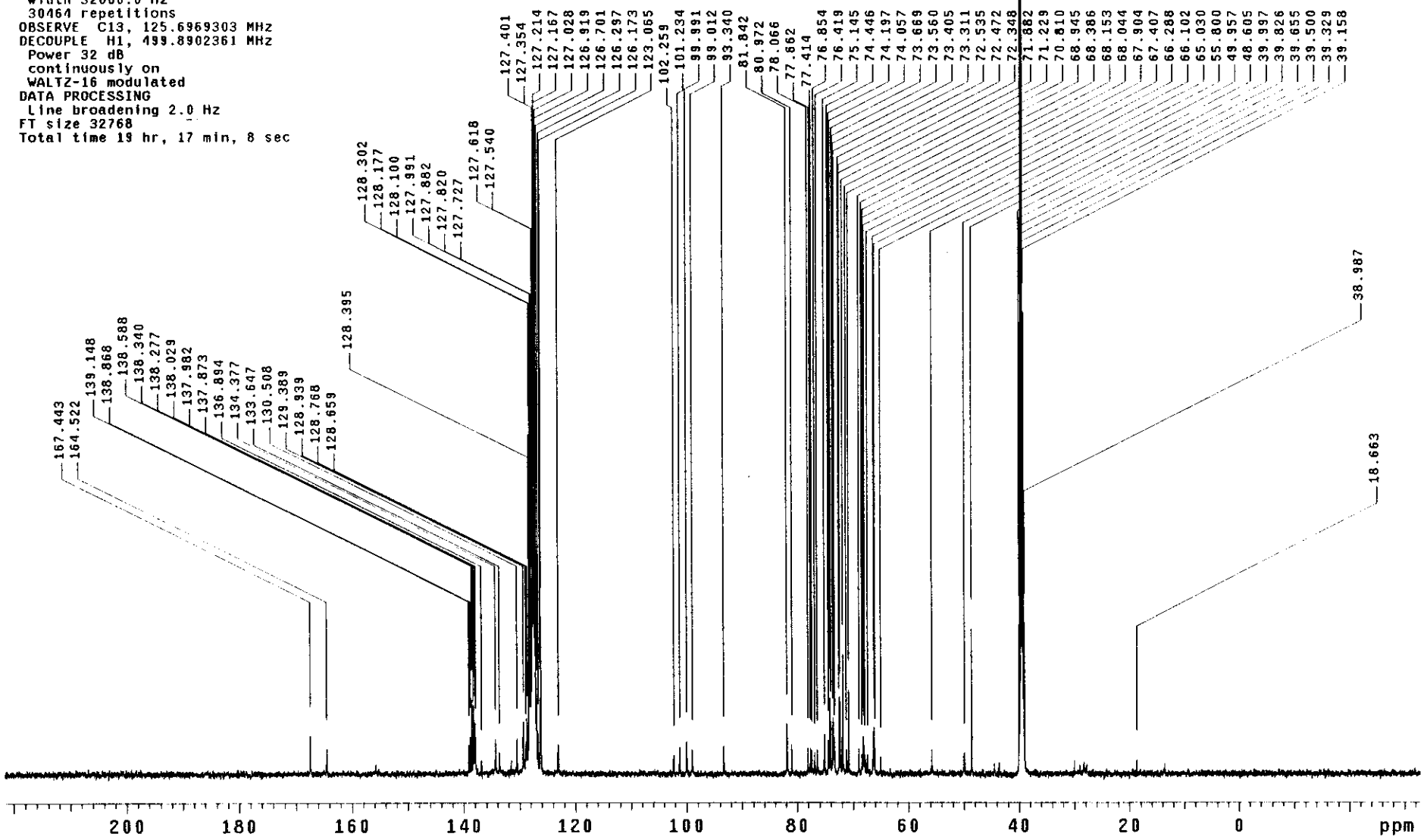

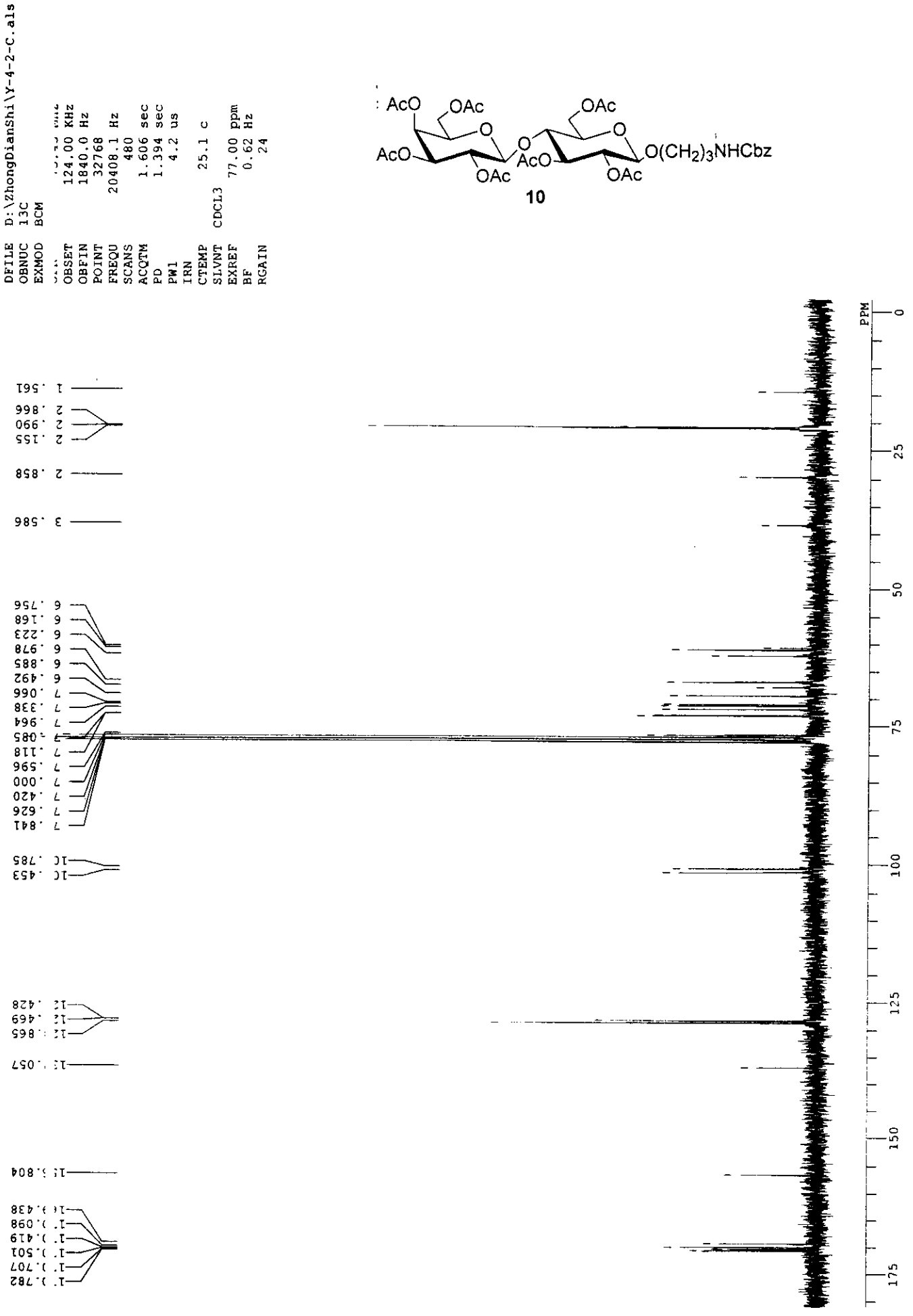
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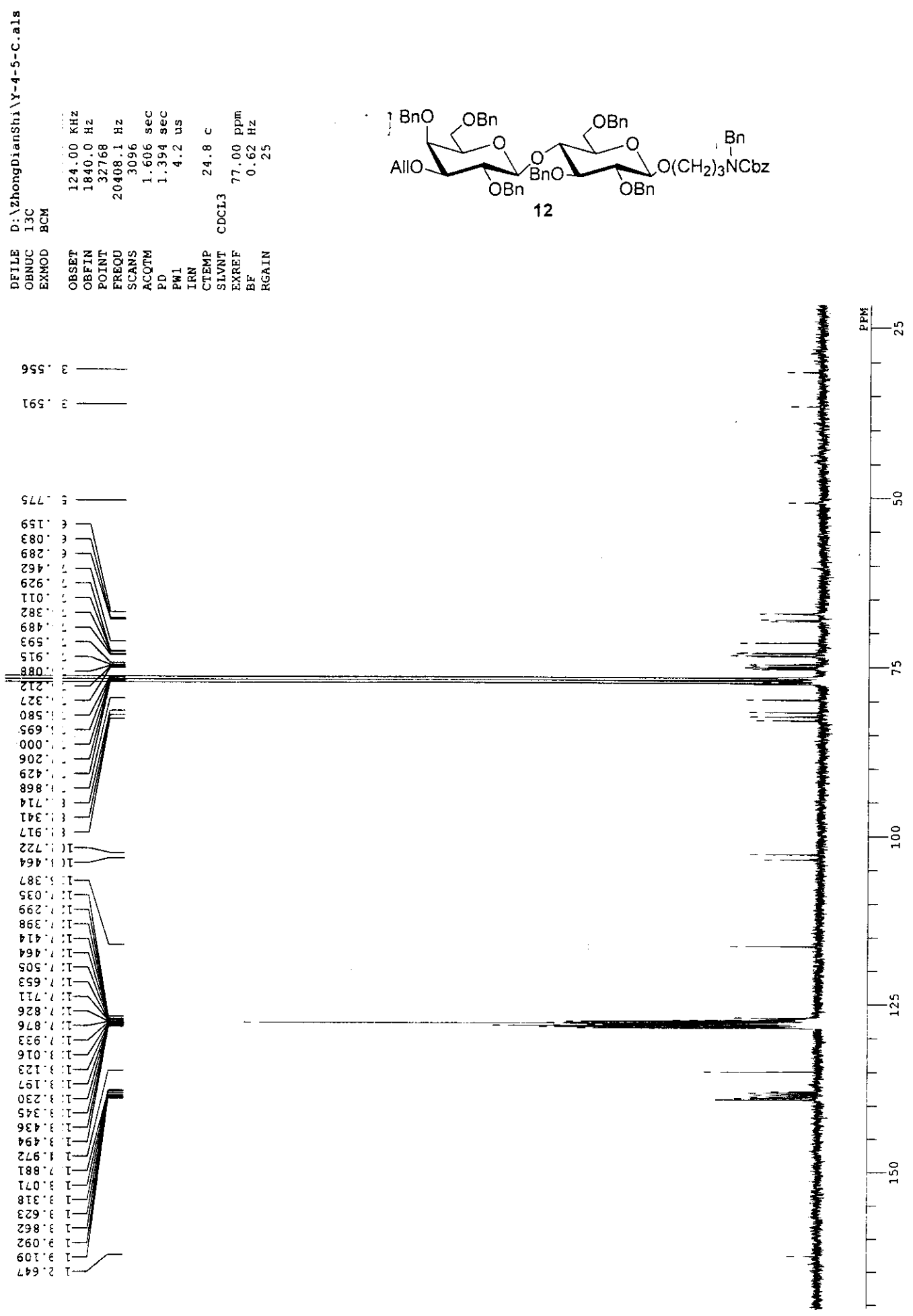
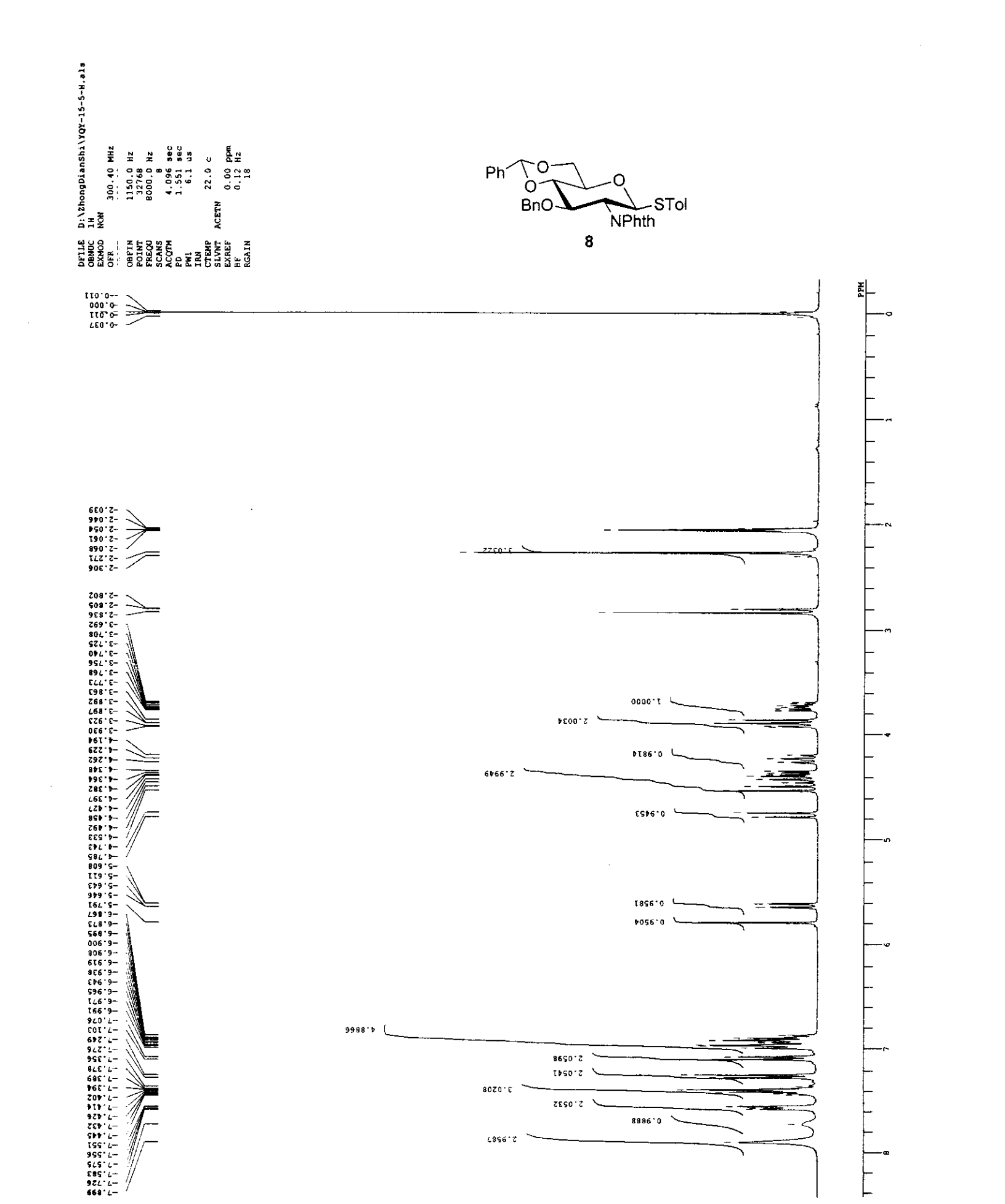
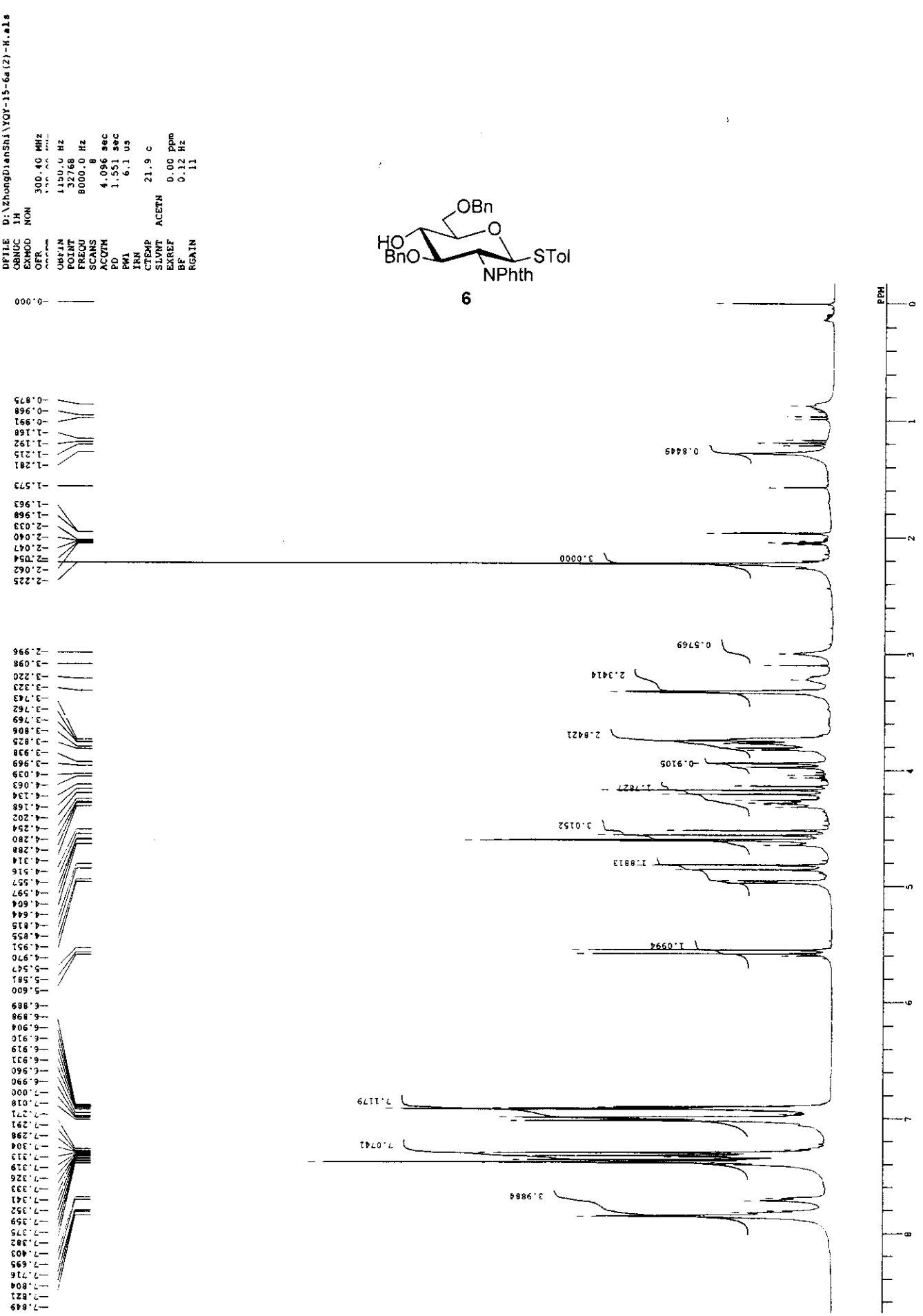
Total time 0 min, 48 sec

12


D:\ZhongDianShi\Y-4-5-C.als
DFILE
OBNUC 13C
EXMOD BCM
OBSET 124.00 KHz
OBFIN 1840.0 Hz
POINT 32768
FREQU 20408.1 Hz
SCANS 3096
ACQTM 1.606 sec
PD 1.394 sec
PW1 4.2 us
IRN
CTEMP 24.8 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.62 Hz
RGAIN 25
BnO
OBn
AllO
OBn
BnO
OBn
OBn
O
O
O
Bn
O(CH2)3NCbz
12
PPM
25
50
75
100
125
150

YQY-4-6
Pulse Sequence: presat
Solvent: cdcl3
Temp. 22.0 C / 295.1 K
INOVA-500 "bmu500"
Relax. delay 1.000 sec
Pulse 70.4 degrees
Acq. time 1.200 sec
Width 10000.0 Hz
128 repetitions
OBSERVE H1, 499.8853759 MHz
DATA PROCESSING
FT size 32768
Total time 4 min, 47 sec
7.347
7.332
7.318
7.313
7.306
7.303
7.299
7.292
7.285
7.281
7.276
7.267
7.256
7.243
7.238
7.225
7.193
7.179
7.165
7.151
7.136
7.40 7.30 7.20 7.10 ppm
0.894
0.889
0.880
0.874
0.868
0.855
0.843
0.830
0.95 0.80 ppm
2.189
2.182
2.045
1.597
1.274
1.254
ppm
7
OBn
BnO
O(CH2)3NCbz
Bn
33.34
1.00 1.33 1.94 2.05 1.88 5.82 2.82 2.93 2.38
0.69 2.18 10.14 0.38 4.58 3.10


D:\ZhongDianShi\YQY-15-5-H.als
1H
NON
300.40 MHz
1150.0 Hz
32768
8000.0 Hz
8
4.096 sec
1.551 sec
6.1 us
22.0 c
ACETN
0.00 ppm
0.12 Hz
18
Ph
O
O
O
BnO
STol
NPhth
8
PPM
4.8866
3.0322
2.9587
2.9949
2.0034
2.0598
2.0541
2.0532
3.0208
1.0000
0.9888
0.9814
0.9504
0.9581
0.9453


DFILE D:\ZhongDianShi\YQY-15-6a(2)-H.als
OBNUC 1H
EXMOD NON
OFR 300.40 MHz
OBSWIN 4150.6 Hz
POINT 32768
FREQU 8000.0 Hz
SCANS 8
ACQTM 4.096 sec
PD 1.551 sec
PW1 6.1 us
IRN
CTEMP 21.9 c
SLVNT ACETN
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 11
OBn
HO
BnO
O
STol
NPhth
6
PPM
0.8449
3.0000
0.5769
2.3414
2.8421
0.9105
1.7827
3.0152
1.8813
1.0994
7.1179
7.0741
3.9884

DFILE D:\ZhongDianShi\Y-4-2A-H.a
OBNUC 1H
EXMOD NON
OBSET 130.00 KHz
OBFIN 1150.0 Hz
POINT 32768
FREQU 6013.2 Hz
SCANS 4
ACQTM 5.449 sec
PD 1.551 sec
PW1 6.1 us
IRN
CTEMP 21.8 c
SLVNT CDCL3
EXREF 7.24 ppm
BF 0.09 Hz
RGAIN 17

10

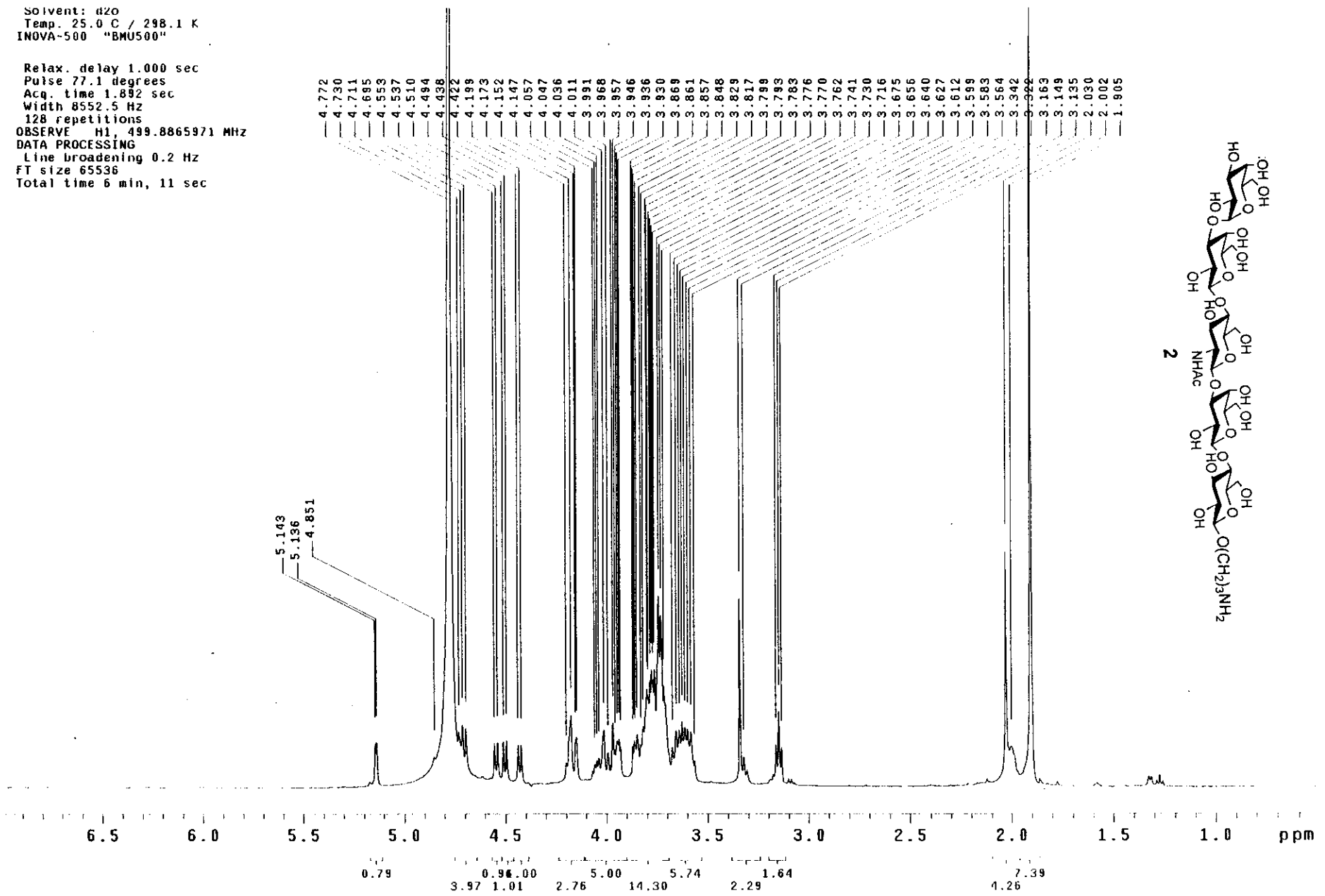
WYH040315

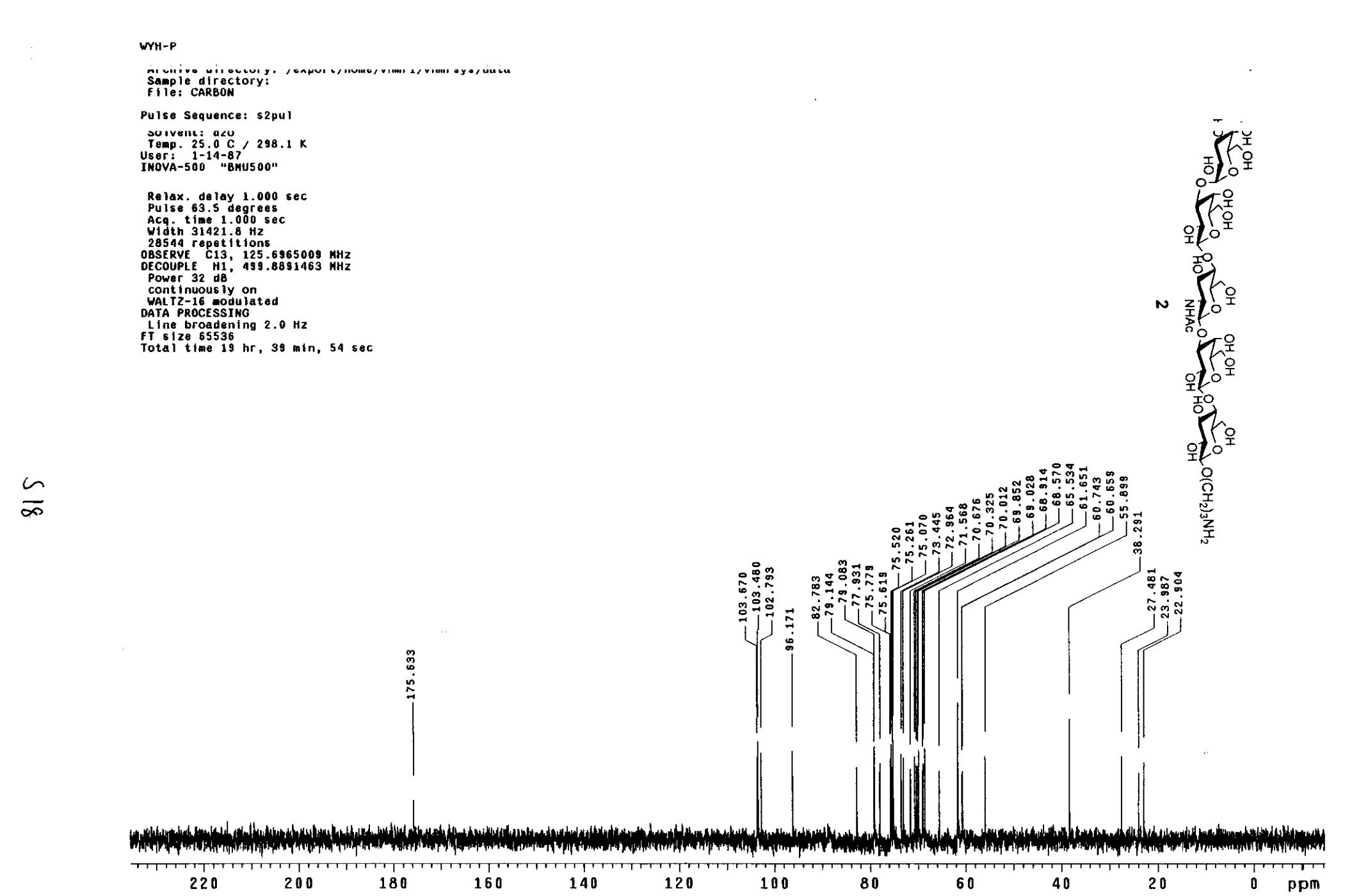
Archive directory: /export/home/vnmr1/vnmrsys/data
Sample directory:
File: PROTON

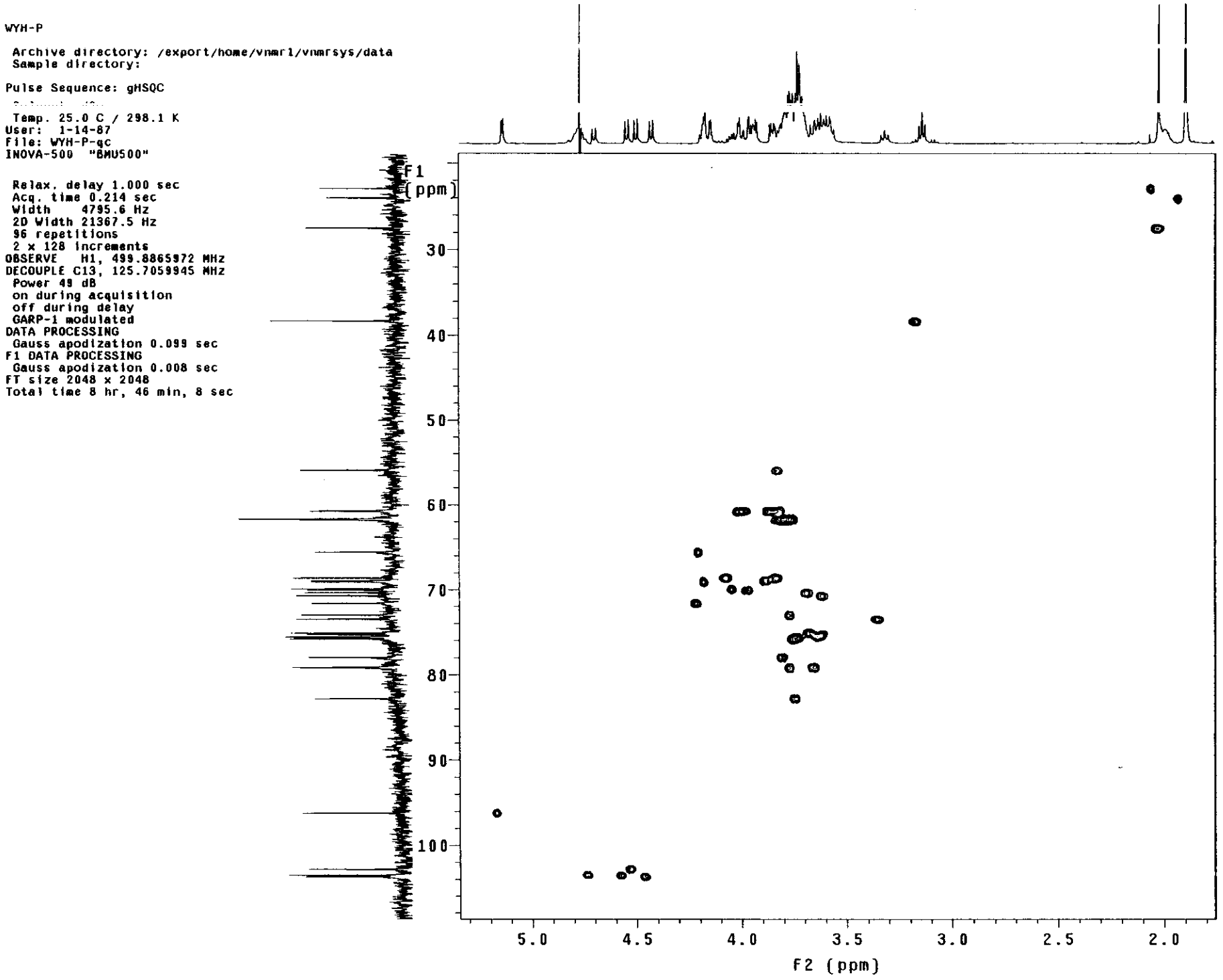
Pulse Sequence: s2pul

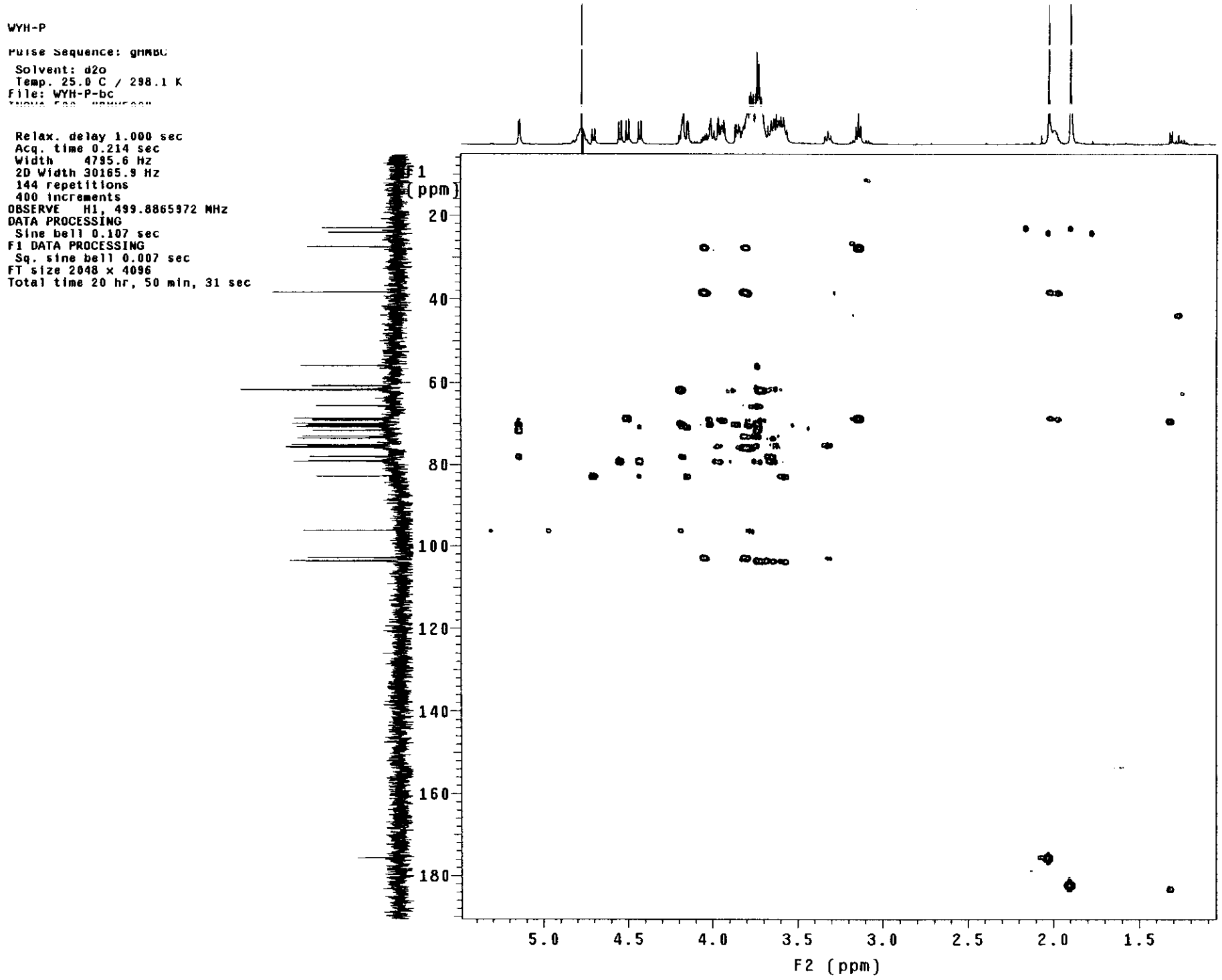
Solvent: d2o
Temp. 25.0 C / 298.1 K
INOVA-500 "BMU500"

Relax. delay 1.000 sec
Pulse 77.1 degrees
Acq. time 1.892 sec
Width 8552.5 Hz
128 repetitions
OBSERVE H1, 499.8865971 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 6 min, 11 sec




WYH-P
Archive directory: /export1/home/vimm1/vimmsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: d2o
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "BMU500"
Relax. delay 1.000 sec
Pulse 63.5 degrees
Acq. time 1.000 sec
Width 31421.8 Hz
28544 repetitions
OBSERVE C13, 125.6965009 MHz
DECOUPLE H1, 499.8891463 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 19 hr, 39 min, 54 sec
175.633
103.670
103.480
102.793
96.171
82.783
79.144
79.083
77.931
75.779
75.619
75.520
75.261
75.070
73.445
72.964
71.568
70.676
70.325
70.012
69.852
69.028
68.914
68.570
65.534
61.651
60.743
60.659
55.899
38.291
27.481
23.987
22.904
NHAc
O(CH2)3NH2
2
220
200
180
160
140
120
100
80
60
40
20
0
ppm


WYH-P
Archive directory: /export/home/vnmr1/vnmrsys/data
Sample directory:
Pulse Sequence: gHSQC
Temp. 25.0 C / 298.1 K
User: 1-14-87
File: WYH-P-gc
INOVA-500 "BMU500"
Relax. delay 1.000 sec
Acq. time 0.214 sec
Width 4795.6 Hz
2D Width 21367.5 Hz
96 repetitions
2 x 128 increments
OBSERVE H1, 499.8865972 MHz
DECOUPLE C13, 125.7059945 MHz
Power 49 dB
on during acquisition
off during delay
GARP-1 modulated
DATA PROCESSING
Gauss apodization 0.099 sec
F1 DATA PROCESSING
Gauss apodization 0.008 sec
FT size 2048 x 2048
Total time 8 hr, 46 min, 8 sec
F1 (ppm)
30
40
50
60
70
80
90
100
5.0
4.5
4.0
3.5
3.0
2.5
2.0
F2 (ppm)


WYH-P
Pulse Sequence: gHMBC
Solvent: d2o
Temp. 25.0 C / 298.1 K
File: WYH-P-bc
Relax. delay 1.000 sec
Acq. time 0.214 sec
Width 4795.6 Hz
2D Width 30165.9 Hz
144 repetitions
400 increments
OBSERVE H1, 499.8865972 MHz
DATA PROCESSING
Sine bell 0.107 sec
F1 DATA PROCESSING
Sq. sine bell 0.007 sec
FT size 2048 x 4096
Total time 20 hr, 50 min, 31 sec
F1 (ppm)
20
40
60
80
100
120
140
160
180
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
F2 (ppm)

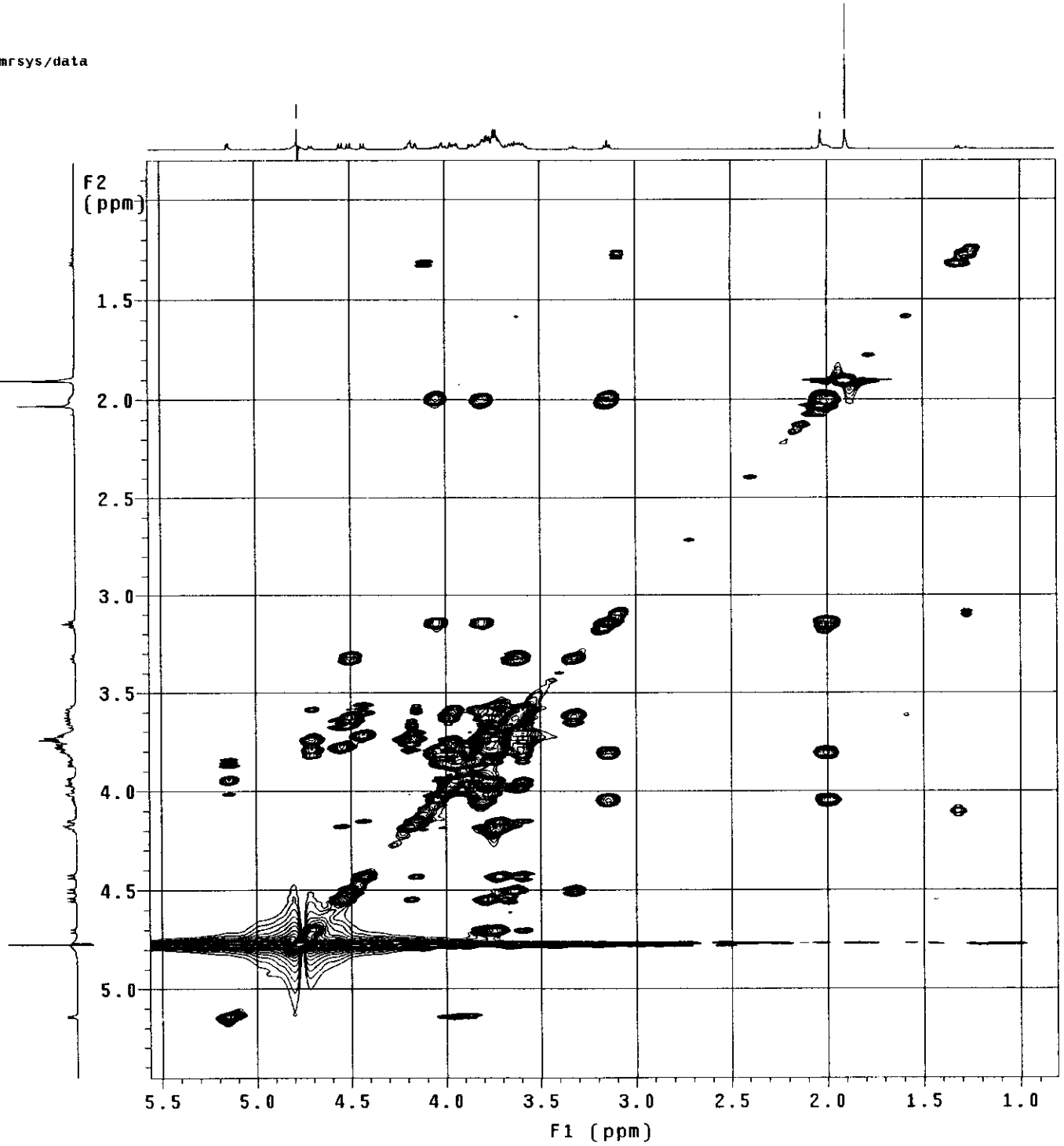
WYH-P

Archive directory: /export/home/vnmr1/vnmrsys/data
Sample directory:
File: PROTON

Pulse Sequence: TOCSY

Solvent: d2o
Temp. 25.0 C / 298.1 K
INOVA-500 "BMU500"

Relax. delay 0.800 sec
Mixing 0.060 sec
Acq. time 0.214 sec
Width 4795.6 Hz
2D Width 4795.6 Hz
64 repetitions
2 x 200 increments
OBSERVE H1, 499.8865978 MHz
DATA PROCESSING
Gauss apodization 0.099 sec
F1 DATA PROCESSING
Gauss apodization 0.018 sec
FT size 4096 x 4096
Total time 15 hr, 6 min, 37 sec



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