Supporting Information

Convergent Synthesis of a GPI Containing an Acylated Inositol

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Experimental

General methods. ^{1}H NMR spectra were recorded at 300 and 600 MHz with the chemical shifts reported in ppm (δ) downfield from tetramethylsilane (TMS). ^{13}C NMR spectra were recorded at 50 and 150 MHz with chemical shifts reported in ppm (δ) in reference to the solvent $CDCl_3$ (δ 77.16). Coupling constants (*J*) are reported in hertz (Hz). Thin layer chromatography (TLC) was performed on silica gel plates with detection by charring with phosphomolibdic acid in EtOH or 5% H_2SO_4 in EtOH. Commercial anhydrous solvents and other reagents were used without further purification.

Synthesis of 7. To the freshly dried MS 4A (3.0 g) was added **3** (780 mg, 0.487 mmol) and **6** (410 mg, 0.348 mmol) in dichloromethane/ethyl ether (1:1, 20 mL). After the mixture was stirred at rt for 1 h, it was cooled down to 0 °C, and NIS (220 mg, 0.97 mmol) was added. The mixture was stirred for another 30 min and then cooled down to –10 °C, whereupon TfOH (6.1 μL, 0.035 mmol) in dichloromethane was added. The mixture was warmed to 0 °C and stirred for another 30 min before triethylamine was added to quench the reaction. The molecular sieves were filtered off and the solution was diluted with ethyl ether. The organic layer was washed with water, dried with Na_2SO_4 and then concentrated. Column chromatography of the residue gave **7** (480 mg, 51%). $[\alpha]_D$ = +36.8 (*c* 1.0, $CHCl_3$). ^{1}H NMR ($CDCl_3$, 600 MHz): δ 7.68-7.77 (m, 4H), 7.00-7.38 (m, 78H), 6.87 (d, *J*

9.0 Hz, 2H), 5.96 (t, *J* 3.0 Hz, 1H), 5.72 (d, *J* 4.2 Hz, 1H), 5.26 (s, 2H), 4.91-5.00 (m, 5H), 4.76-4.79 (m, 3H), 4.73 (d, *J* 11.4 Hz, 1H), 4.72 (d, *J* 13.2 Hz, 1H), 4.68 (d, *J* 9.6 Hz, 1H), 4.64 (d, *J* 10.8 Hz, 1H), 4.62 (d, *J* 11.4 Hz, 1H), 4.57 (d, *J* 10.8 Hz, 1H), 4.27-4.55 (m, 17H), 4.20 (d, *J* 12.0 Hz, 1H), 4.07-4.14 (m, 4H), 4.02 (dd, *J* 11.4, 3.0 Hz, 1H), 3.70-3.97 (m, 16H), 3.58-3.65 (m, 4H), 3.55 (dd, *J* 9.6, 3.0 Hz, 1H), 3.49 (t, *J* 9.6 Hz, 1H), 3.45 (dd, *J* 10.8, 4.2 Hz, 1H), 3.37-3.41 (m, 2H), 3.31 (dd, *J* 11.4, 4.2 Hz, 1H), 3.25 (d, *J* 7.5 Hz, 1H), 3.17 (dd, *J* 10.8, 4.2 Hz, 1H), 2.38-2.41 (m, 2H), 1.60-1.64 (m, 2H), 1.25-1.32 (m, 24H), 1.06 (s, 9H), 0.90 (t, *J* 7.2 Hz, 3H). ^{13}C NMR (50 MHz, $CDCl_3$): δ 173.4, 159.5, 139.1, 139.0, 138.9, 138.8, 138.7, 138.6, 138.5, 138.4, 138.3, 138.1, 138.0, 137.9, 137.7, 136.0, 135.8, 134.1, 133.5, 130.4, 129.5, 129.2, 129.0, 128.8, 128.7, 128.6, 128.5, 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, 100.4, 99.2, 98.8, 97.4, 81.8, 81.0, 80.1, 80.0, 79.9, 79.2, 78.4, 76.3, 76.0, 75.9, 75.3, 75.0, 74.6, 74.4, 74.3, 73.8, 73.5, 73.3, 73.2, 73.0, 72.2, 72.1, 71.9, 71.8, 71.5, 69.7, 69.2, 69.1, 69.0, 68.9, 68.8, 66.4, 65.4, 63.2, 63.0, 55.3, 34.4, 32.0, 29.8, 29.6, 29.5, 29.4, 29.1, 26.9, 25.3, 22.8, 19.4, 14.2. MALDI-TOF-MS: Calcd for $C_{168}H_{191}N_3O_{27}Si$ 2710, Found 2733 ($M+Na^+$), 2749 ($M+K^+$).

Synthesis of 8. After the mixture of **7** (400 mg, 0.148 mmol) and CAN (400 mg, 0.73 mmol) in acetonitrile and water (9:1, 20 mL) was stirred at rt for 4 h, it was diluted with ethyl acetate, and the organic layer was washed with brine, dried with Na_2SO_4 and concentrated in a vacuum. Column chromatography of the residue gave **8** (200 mg, 0.077mmol, 52%), and 120 mg of **7** was recovered. TLC (acetone/hexane/dichloromethane 4:10:1): R_f = 0.50. $[\alpha]_D$ = +32.8 (*c* 1.0, $CHCl_3$). ^{1}H NMR ($CDCl_3$, 600 MHz): δ 7.66-7.74 (m, 4H), 7.00-7.34 (m, 76H), 5.70 (t, *J* 2.4 Hz, 1H), 5.29 (d, *J* 2.4 Hz, 1H), 5.26 (d, *J* 3.6 Hz, 1H), 5.23 (d, *J* 1.8 Hz, 1H), 4.94 (d, *J* 10.2 Hz, 1H), 4.91 (d, *J* 10.8 Hz, 1H), 4.89 (d, *J* 10.8 Hz, 1H), 4.85 (d, *J* 10.8 Hz, 1H), 4.82 (d, *J* 12.0 Hz, 1H), 4.69-4.76 (m, 5H), 4.65 (d, *J* 11.4 Hz, 1H), 4.61 (d, *J* 10.8 Hz, 1H), 4.19-4.51 (m, 17H), 4.08 (m, 1H), 3.98 (dd, *J* 11.4, 3.6 Hz, 1H), 3.59-3.92 (m, 16H), 3.50-3.53 (m, 2H), 3.40-3.46 (m, 3H), 3.33-3.36 (m, 2H), 3.24-3.28 (m, 2H), 2.39 (t, *J* 7.2 Hz, 2H), 1.64 (m, 2H), 1.21-1.34 (m, 24H), 1.02 (s, 9H), 0.87 (t, *J* 7.2 Hz, 3H). MALDI-TOF-MS: Calcd for $C_{160}H_{183}N_3O_{26}Si$ 2590, Found 2614 ($M+Na^+$).

Synthesis of 9. To a solution of **8** (70 mg, 0.027 mmol) and freshly prepared **5** in dry DCM/CH_3CN (3:1, 6 mL) was added 1*H*-tetrazole (0.38 mmol, 0.8 mL of 0.48 M solution in CH_3CN). After the mixture was stirred for 6 h at rt, MCPBA (100 mg, 0.58 mmol) was added. The mixture was stirred

for another 2 h and then concentrated. Column chromatography of the residue afforded **9** (70 mg, 83%) as a mixture of two diastereoisomers (1.6:1). TLC (acetone/hexane/DCM 4:10:1): R_f = 0.45. $[\alpha]_D$ = +16.5 (*c* 1.0, $CHCl_3$). ^{1}H NMR ($CDCl_3$, 600 MHz): δ (isomer-a) 7.58-7.68 (m, 4H), 5.74 (t, *J* 2.4 Hz, 1H), 5.31 (d, *J* 1.8 Hz, 1H), 5.15 (d, *J* 1.8 Hz, 1H), 5.02 (d, *J* 12.0 Hz, 1H), 2.27 (m, 2H), 0.96 (s, 9H), 0.81 (t, *J* 7.2 Hz, 3H); δ (isomer-b) 7.58-7.68 (m, 4H), 5.60 (t, *J* 2.4 Hz, 1H), 5.39 (d, *J* 4.2 Hz, 1H), 5.26 (d, *J* 1.8 Hz, 1H), 5.19 (d, *J* 1.8 Hz, 1H), 2.27 (m, 2H), 0.97 (s, 9H), 0.81 (t, *J* 7.2 Hz, 3H). ^{31}P NMR ($CDCl_3$): δ 8.98, 1.27. MALDI-TOF-MS: Calcd for $C_{188}H_{232}NO_{30}PSi$ 3043, Found 3066 ($M+Na^+$), 3082 ($M+K^+$).

Synthesis of 10a and 10b. After the solution of sodium methoxide in methanol (1 M, 1 mL) and **9** (10 mg, 0.0033 mmol) in DCM (2 mL) was stirred at rt for 2 days, the reaction mixture was diluted with ethyl acetate. The organic layer was washed with water, dried over Na_2SO_4 and concentrated. The residue was purified with preparative TLC to give two products **10a** and **10b** (1:1.6) in a quantitative yield. **10a**: TLC (acetone/hexane/DCM 3:10:3): R_f = 0.50. ^{1}H NMR ($CDCl_3$, 600 MHz): δ 5.38 (d, *J* 3.6 Hz, 1H), 5.31 (s, 1H), 5.24 (s, 1H), 4.73 (s, 1H). MALDI-TOF-MS: Calcd for $C_{172}H_{202}NO_{29}PSi$ 2804, Found 2827 ($M+Na^+$), 2843 ($M+K^+$). **10b**: TLC (acetone/hexane/DCM 3:10:3): R_f = 0.25. ^{1}H NMR ($CDCl_3$, 600 MHz): δ 5.36 (s, 1H), 5.23 (s, 1H), 4.96 (d, *J* 3.0 Hz, 1H), 4.66 (d, *J* 1.8 Hz, 1H). HMQC data ^{13}C (150 MHz)/^{1}H (600MHz): 102.9/4.96 (1-Glu), 99.3/5.36 (1-Man), 99.1/4.66 (1-Man), 98.8/5.23 (1-Man). MALDI-TOF-MS: Calcd for $C_{172}H_{202}NO_{29}PSi$ 2804, Found 2828 ($M+Na^+$), 2843 ($M+K^+$).

Synthesis of 11. After a mixture of 6-*O*-[(2,3,4-tri-*O*-benzyl-α-D-mannopyranosyl)-(1→2)-*O*-(3,4,6-tri-*O*-benzyl-α-D-mannopyranosyl)-(1→6)-*O*-(2,3,4-tri-*O*-benzyl-α-D-mannopyranosyl)-(1→4)-*O*-(2-azido-3,6-di-*O*-benzyl-2-deoxy-α-D-glucopyranosyl)]-2-*O*-hexadecanoyl-3,4,5-tri-*O*-benzyl-*myo*-inositol (109 mg, 0.046 mmol), imidazole (14 mg, 0.20 mmol), DMAP (2 mg, 0.018 mmol) and TBDMSCl (15 mg, 0.10 mmol) in anhydrous DMF (3 mL) was stirred at rt overnight, it was diluted with ethyl acetate. The organic layer was washed with brine, dried over Na_2SO_4 and then concentrated. Column chromatography of the residue gave 6-*O*-[(2,3,4-tri-*O*-benzyl-6-*O*-tert-butyl-dimethylsilyl-α-D-mannopyranosyl)-(1→2)-*O*-(3,4,6-tri-*O*-benzyl-α-D-mannopyranosyl)-(1→6)-*O*-(2,3,4-tri-*O*-benzyl-α-D-mannopyranosyl)-(1→4)-*O*-(2-azido-3,6-di-*O*-benzyl-2-deoxy-α-D-glucopyranosyl)]-2-*O*-hexadecanoyl-3,4,5-tri-*O*-benzyl-*myo*-inositol as a syrup (93 mg, 0.038

mmol, 82%). $[\alpha]_D$ = +34.8 (*c* 1.0, $CHCl_3$). ^{1}H NMR ($CDCl_3$, 600 MHz): δ 7.10-7.32 (m, 70H), 5.74 (t, *J* 2.4 Hz, 1H), 5.32 (br, 1H), 5.31 (d, *J* 2.4 Hz, 1H), 5.18 (d, *J* 1.2 Hz, 1H), 4.99 (d, *J* 10.8 Hz, 1H), 4.92 (d, *J* 10.8 Hz, 2H), 4.91 (d, *J* 10.8 Hz, 1H), 4.88 (d, *J* 11.4 Hz, 1H), 4.80 (d, *J* 1.8 Hz, 1H), 4.79 (d, *J* 10.2 Hz, 1H), 4.77 (d, *J* 10.8 Hz, 1H), 4.76 (d, *J* 10.8 Hz, 1H), 4.73 (d, *J* 10.8 Hz, 1H), 4.70 (d, *J* 10.8 Hz, 1H), 4.66 (d, *J* 10.8 Hz, 1H), 4.56 (d, *J* 12.0 Hz, 1H), 4.55 (d, *J* 11.4 Hz, 1H), 4.52 (d, *J* 10.8 Hz, 1H), 4.51 (d, *J* 12.0 Hz, 1H), 4.51 (d, *J* 11.4 Hz, 1H), 4.46 (d, *J* 12.0 Hz, 1H), 4.45 (d, *J* 10.8 Hz, 1H), 4.41-4.43 (m, 3H), 4.33-4.37 (m, 5H), 4.29 (d, *J* 12.0 Hz, 1H), 4.26 (d, *J* 11.4 Hz, 1H), 4.11 (t, *J* 2.4 Hz, 1H), 4.06 (t, *J* 9.6 Hz, 1H), 3.68-3.97 (m, 18H), 3.65 (dd, *J* 9.6, 3.0 Hz, 1H), 3.56 (dd, *J* 9.6, 2.4 Hz, 1H), 3.37-3.53 (m, 6H), 3.33 (d, *J* 9.6 Hz, 1H), 2.42 (t, *J* 7.2 Hz, 2H), 1.67 (m, 2H), 1.24-1.37 (m, 24H), 0.90 (s, 9H), 0.90 (t, *J* 7.2 Hz, 3H), 0.07 (s, 3H), 0.05 (s, 3H).

To the mixture of above product (200 mg, 0.081 mmol) and dibenzyl dicarbonate (363 mg, 1.50 mmol) in MeOH and DCM (1:2, 10 mL) was added triethylphosphine (145 μL, 1.0 mmol) under a nitrogen atmosphere at 0 °C. The reaction mixture was then warmed up to rt and stirred overnight. After removal of solvents in a vacuum, the residue was purified by flash column chromatography to afford **11** (136 mg, 0.053 mmol, 65%). $[\alpha]_D$ = +34.0 (*c* 1.0, $CHCl_3$). ^{1}H NMR ($CDCl_3$, 600 MHz): δ 5.50 (br s, 1 H), 5.44 (d, *J* 9.0 Hz, 1 H), 5.33 (d, *J* 2.4, 1 H), 5.20 (s, 1 H), 5.14 (s, 1H), 4.99 (d, *J* 12.0 Hz, 1 H), 4.77(s, 1 H), 3.36 (d, *J* 9.0 Hz, 1 H), 3.31 (d, *J* 9.6 Hz, 1 H), 2.30 (m, 2 H), 1.57 (m, 2 H), 1.25 (m, 24 H), 0.86 (s, 9H), 0.86 (t, *J* 7.2 Hz, 3H), 0.03 (s, 3H), 0.02 (s, 3H). MALDI-TOF-MS: Cacld for $C_{158}H_{187}NO_{28}Si$, 2576; Fond, 2599 ($M+Na^+$).

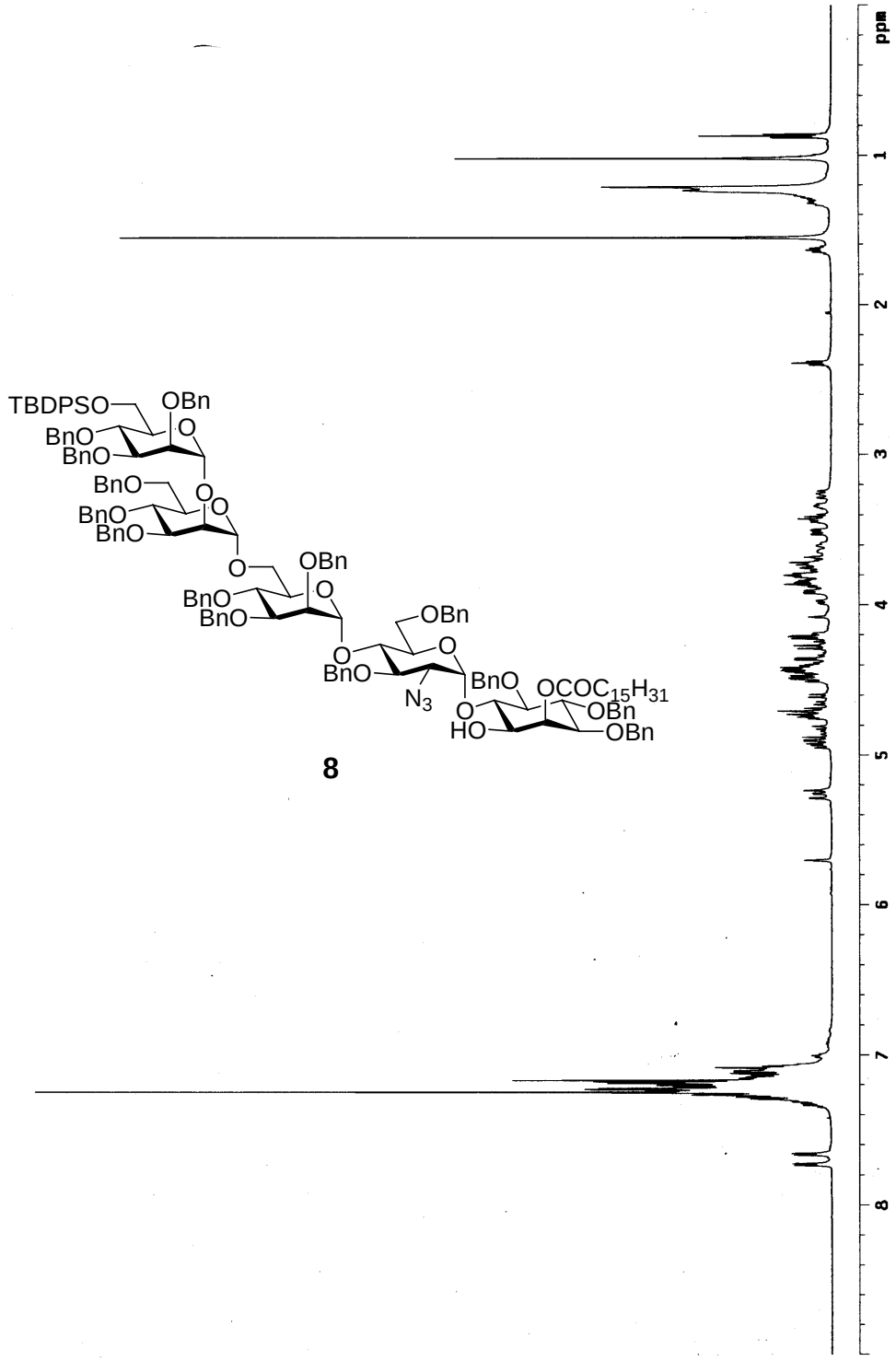
Synthesis of 12. To a mixture of **11** (100 mg, 0.039 mmol) and dibenzyl *N,N*-diisopropylphosphoramidite (55 mg, 0.16 mmol) in dry DCM (5 mL) under argon were added 1*H*-tetrazole (0.16 mmol, 0.47 mol/L in CH_3CN). After 0.5 h of stirring, **11** transformed completely to a less polar product shown on TLC. The solution was concentrated under reduced pressure and the residue was purified by flash column chromatography to give **12** (110 mg, 0.039 mmol, >99%). ^{1}H NMR ($CDCl_3$, 600 MHz): δ 5.70 (br s, 1 H), 5.58 (d, *J* 10.2 Hz, 1 H), 5.34 (d, *J* 3.0, 1 H), 5.18 (s, 1H), 5.12 (s, 1H), 2.24 (m, 2 H), 1.56 (m, 2 H), 1.25 (m, 24 H), 0.84 (s, 9H), 0.84 (t, *J* 7.2 Hz, 3H), 0.02 (s, 3H), 0.00 (s, 3H).

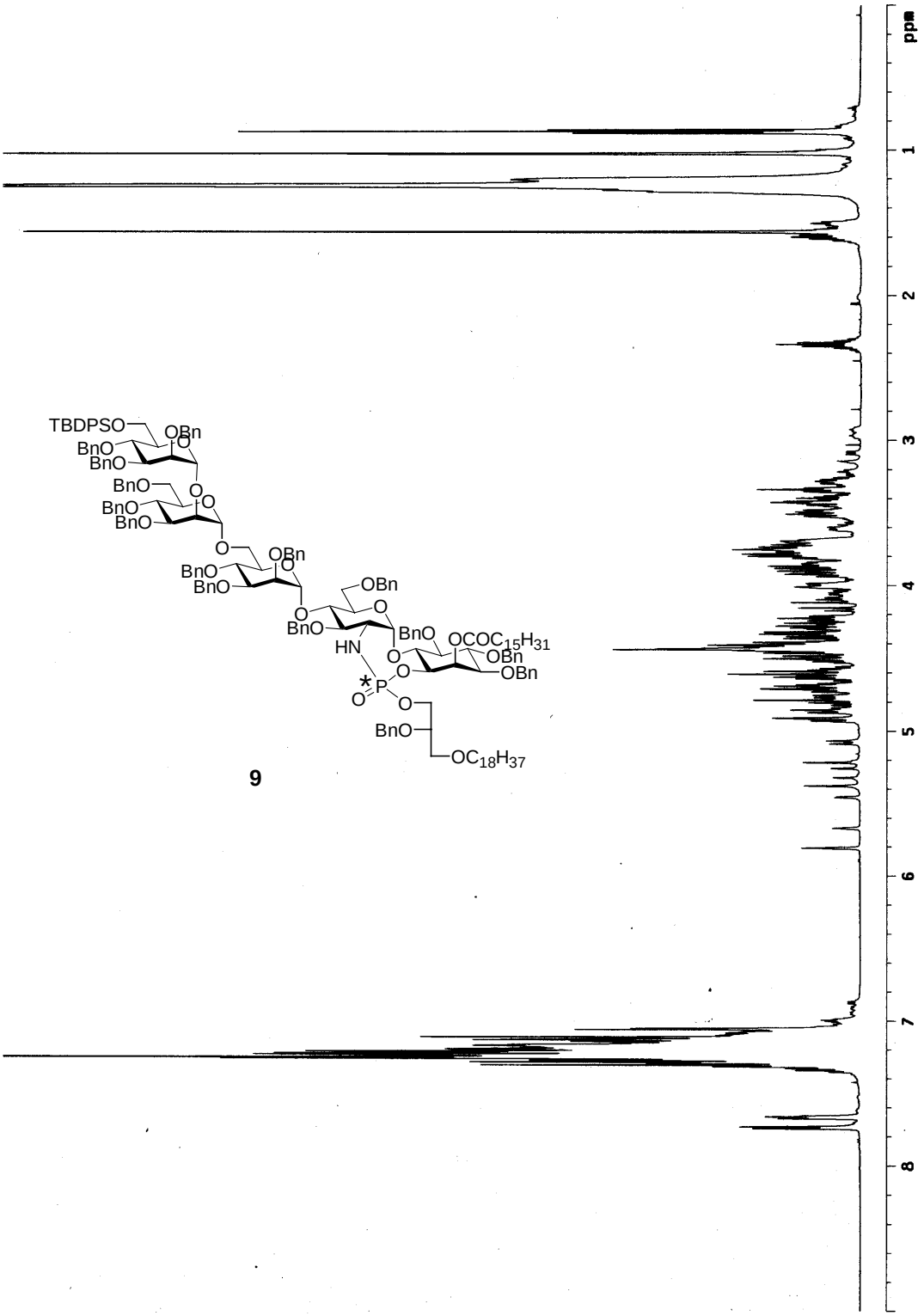
Synthesis of 16. After the mixture of **14** (130 mg, 0.11 mmol), **15** (0.60 mmol), 1*H*-tetrazole (0.050 mmol, 0.47 mmol/L in CH_3CN) and MS 4Å (200 mg) in dry CH_2Cl_2 (10 mL) was stirred at rt under

argon for 6 h, *t*-BuO_2H (5 mmol, 1 mL of 5 M solution in decane) was added at –20°C. The reaction mixture was warmed up to rt and stirred for another 1 h and finally concentrated under vacuum. Flash column chromatography of the residue produced the diastereoisomeric mixture of **16** (143 mg, 0.080 mmol, 76%) as a white solid. TLC (acetone/hexane 1:3): R_f = 0.41. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 5.85 (br, 1H), 5.38 (d, 1H), 2.45 (m, 2H), 2.30 (m, 2H), 1.56 (m, 2H), 0.88 (t, *J* 6.6 Hz, 3H). ^{31}P NMR ($CDCl_3$): 0.93, 0.58. FABMS: Calcd for $C_{104}H_{142}ClN_2O_{19}P$ 1789, Found 1813 ($M+Na^+$).

Synthesis of 22. After the mixture of MS 4A (1.0 g), the pseudodisaccharide **19** (40 mg, 26 μmol) and the disaccharide **21** (60 mg, 54 μmol) in DCM (4 mL) was stirred at rt for 1 h and cooled down to 0 °C, to it was then added NIS (45 mg, 0.2 mmol). The stirring was continued for another 30 min. Upon the mixture was cooled down to –10 °C, TfOH (1.0 μL, 5 μmol) in DCM (1 mL) was added. It was then warmed up to 0 °C and stirred for additional 30 min. Triethylamine was added to quench the reaction, and the molecular sieves were filtered off. The filtrate was diluted with DCM, washed with brine, water, dried with Na_2SO_4 and finally concentrated in a vacuum. Column chromatography of the residue afforded a diastereoisomeric mixture (1.4:1.0) of **22** as colorless syrup (35 mg, 54%), as well as an orthoester side product (15 mg, 23%). ^{1}H NMR ($CDCl_3$, 600 MHz): δ 5.84 (t, *J* = 2.0 Hz, 1 H, major isomer), 5.76 (t, *J* = 2 Hz, 1 H, minor isomer), 5.40-5.30 (m, 4 H), 4.10 (2 d, *J* = 12 Hz, 2 H), 2.36 (m, 4 H), 2.11-2.08 (m, 4 H), 1.58-1.48 (m, 2 H), 1.22-1.30 (m, 54 H), 1.02 (s, 9H), 0.87 (t, *J* = 7.2 Hz, 6 H).

Selected NMR and MS Spectra

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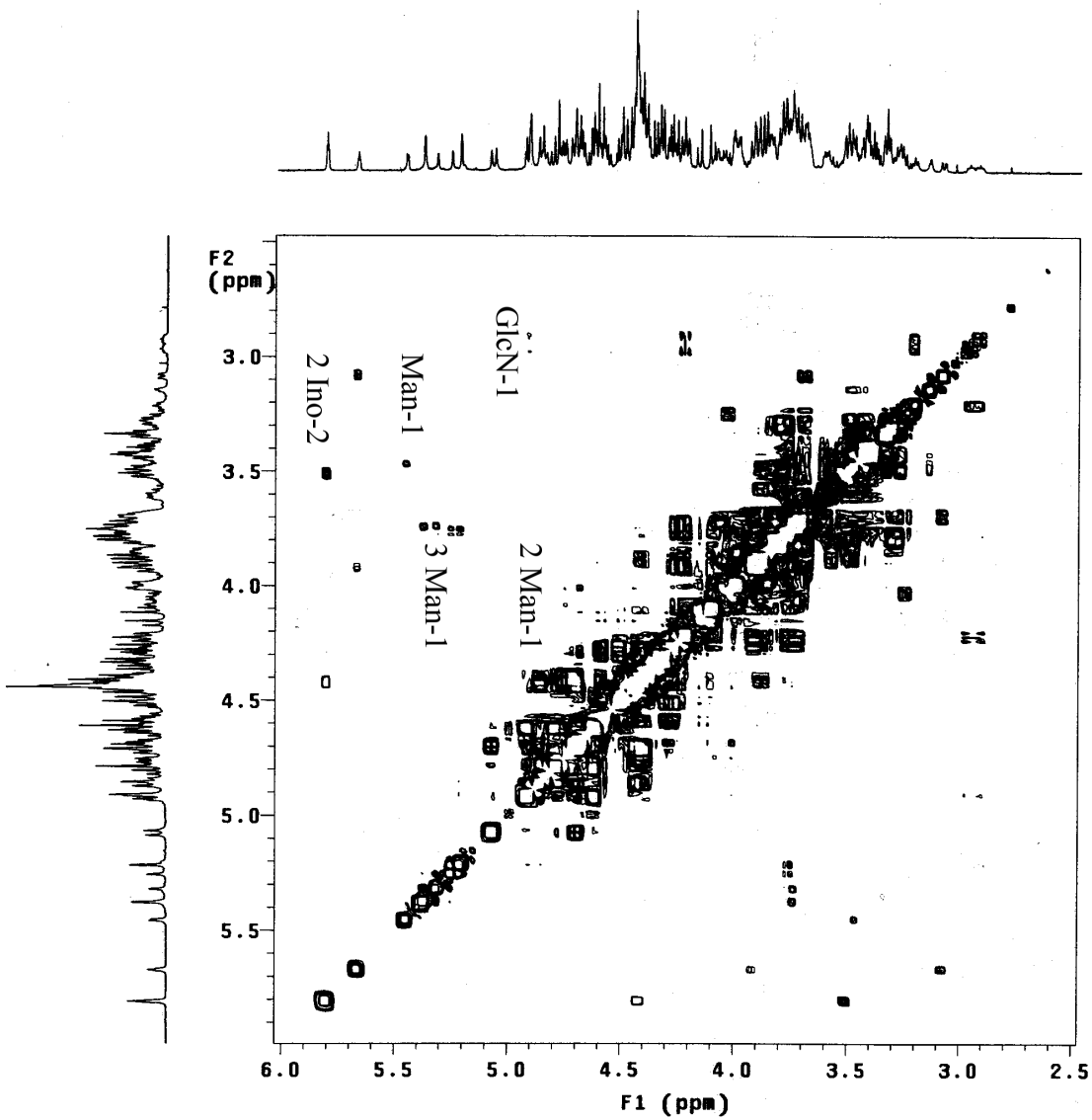

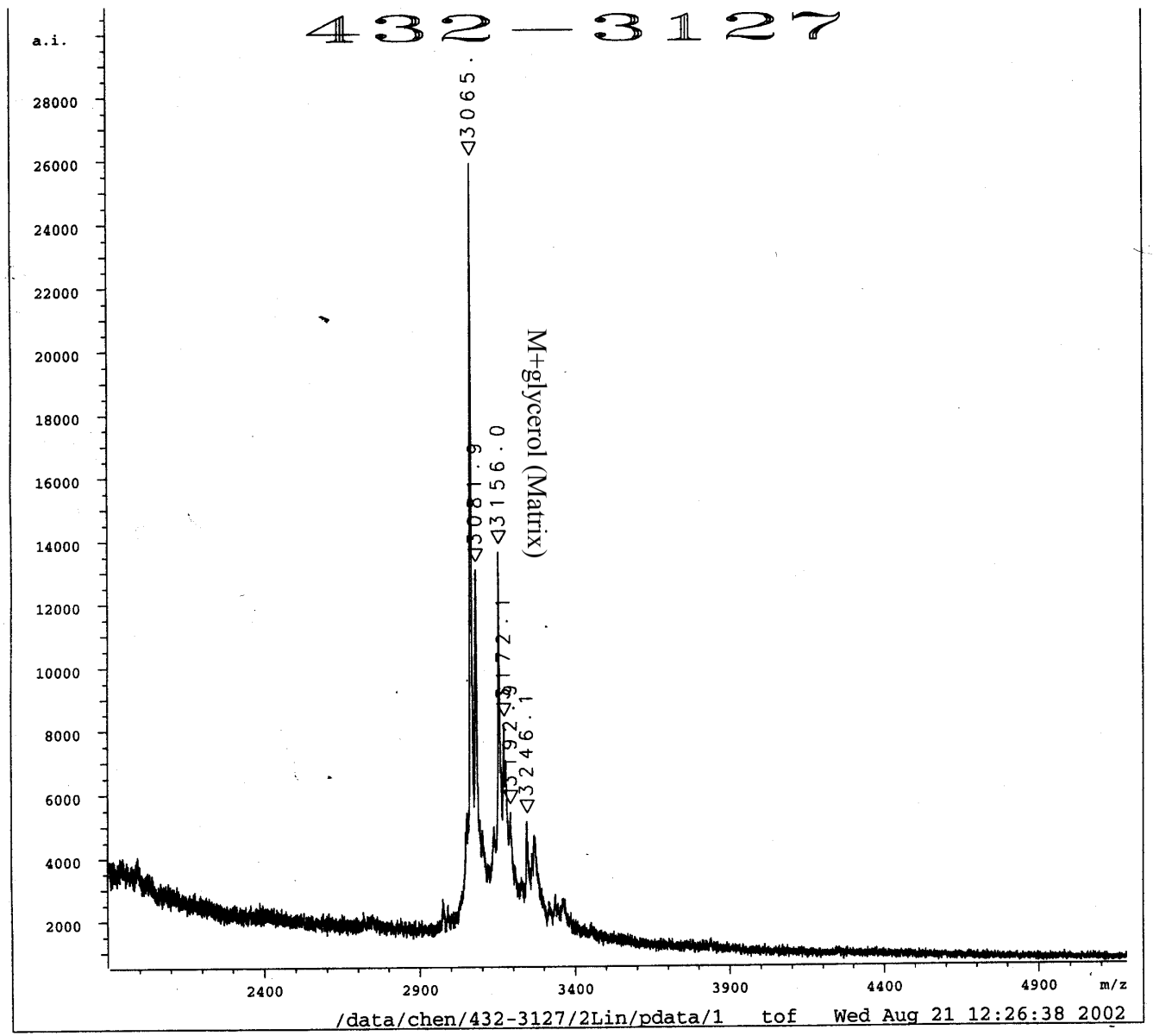


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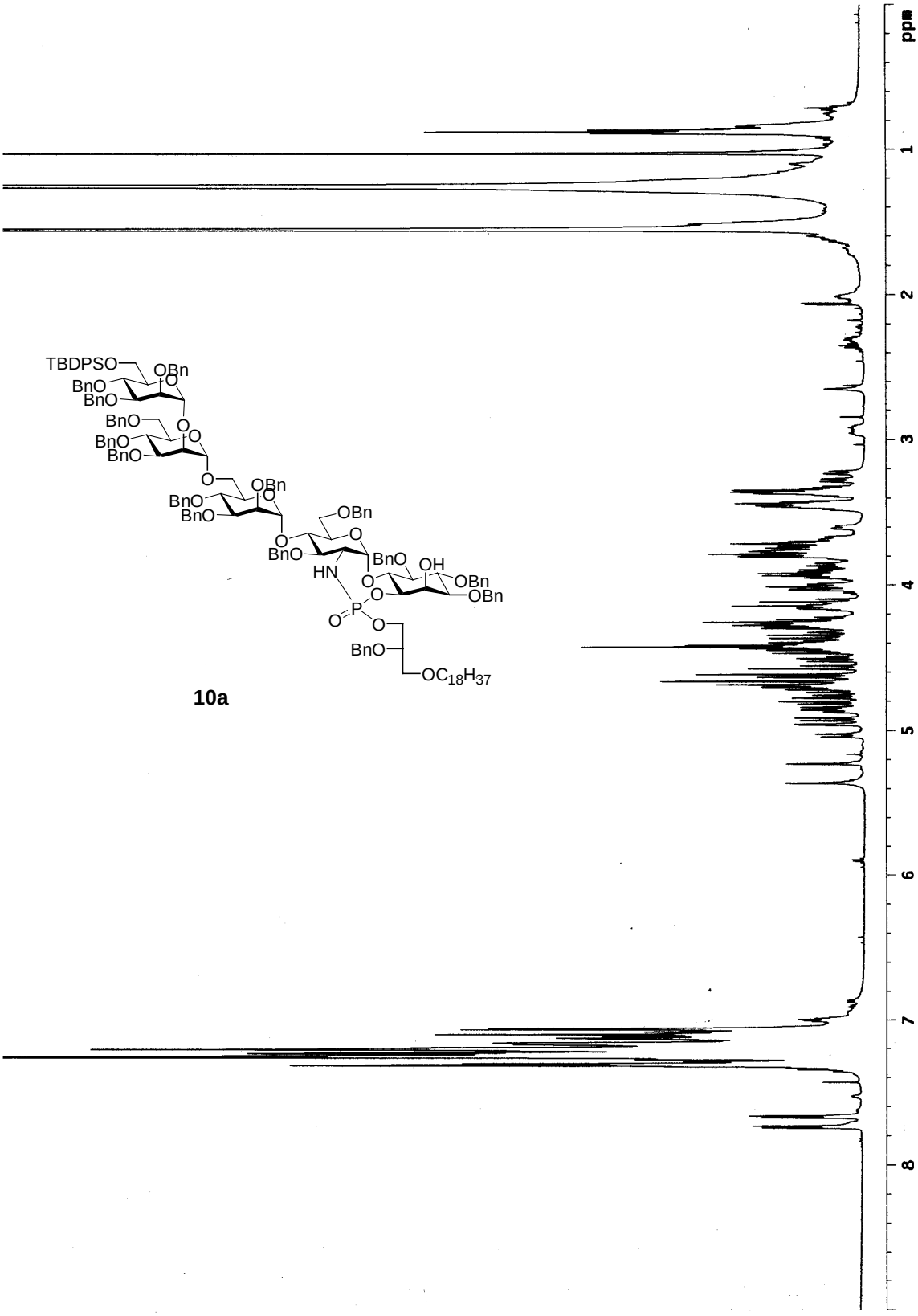
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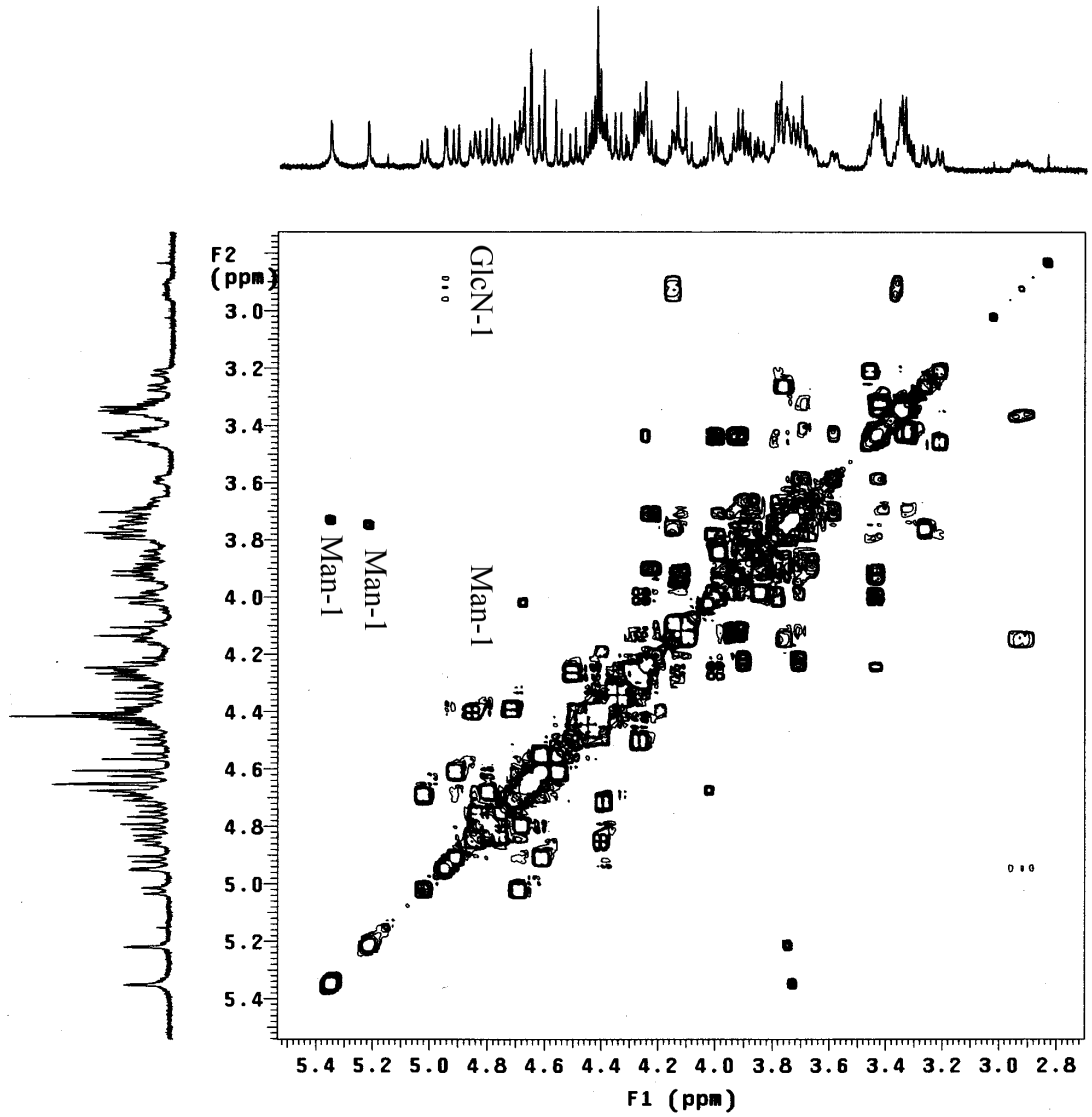

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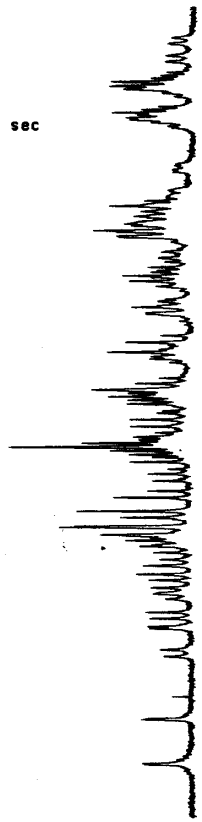

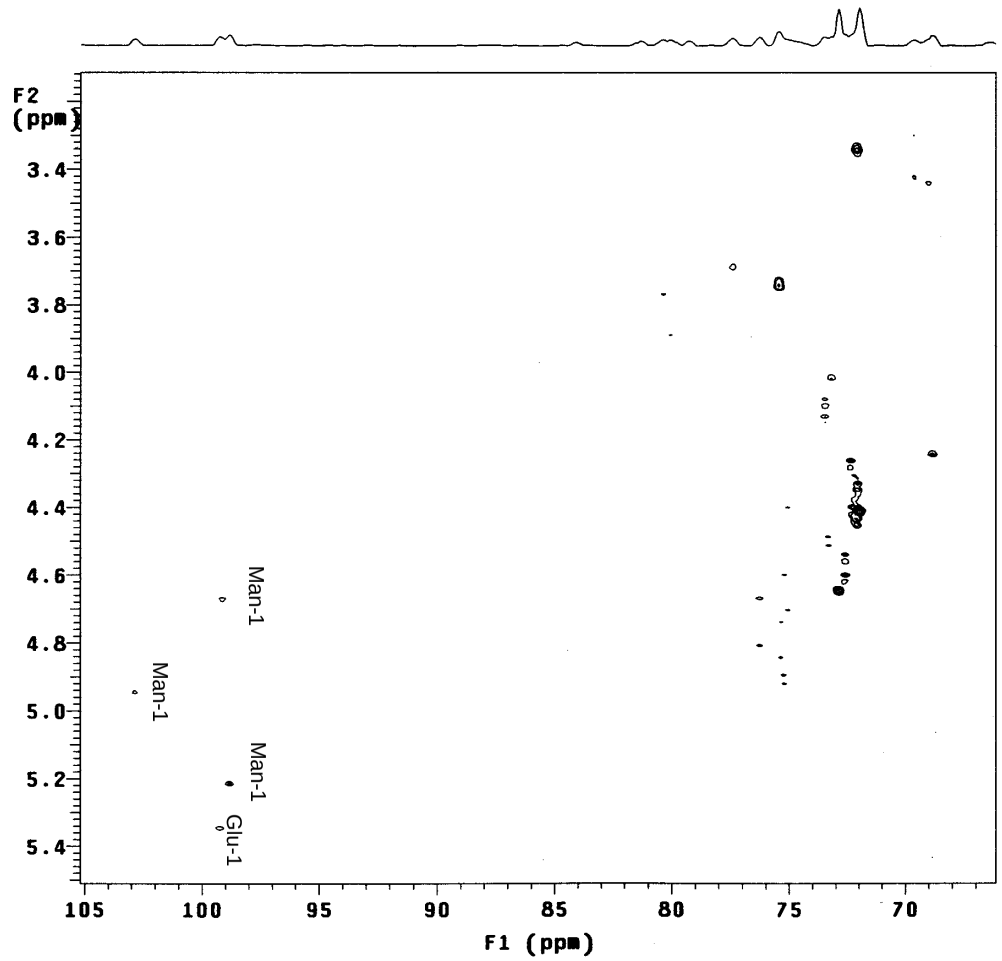

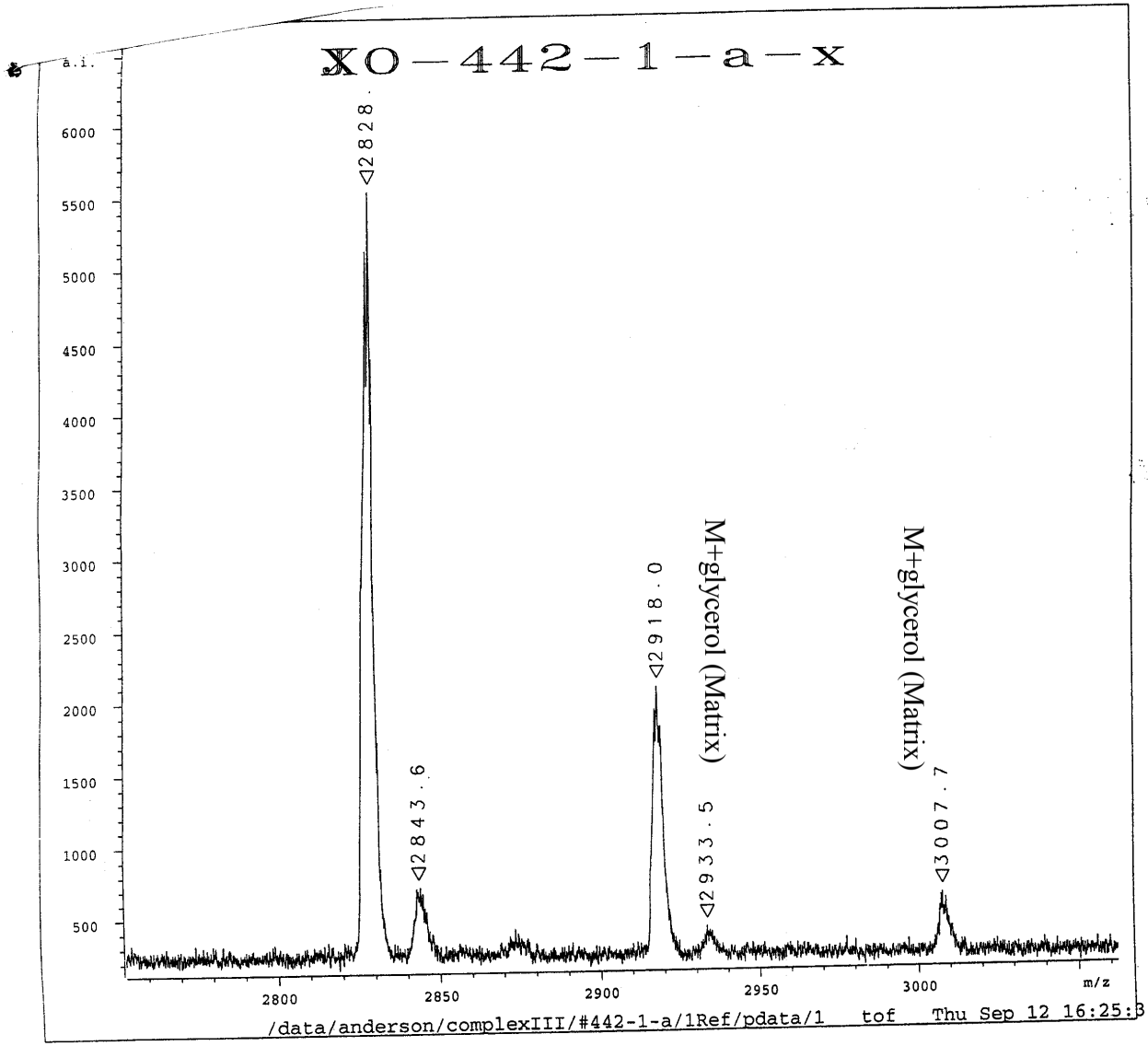


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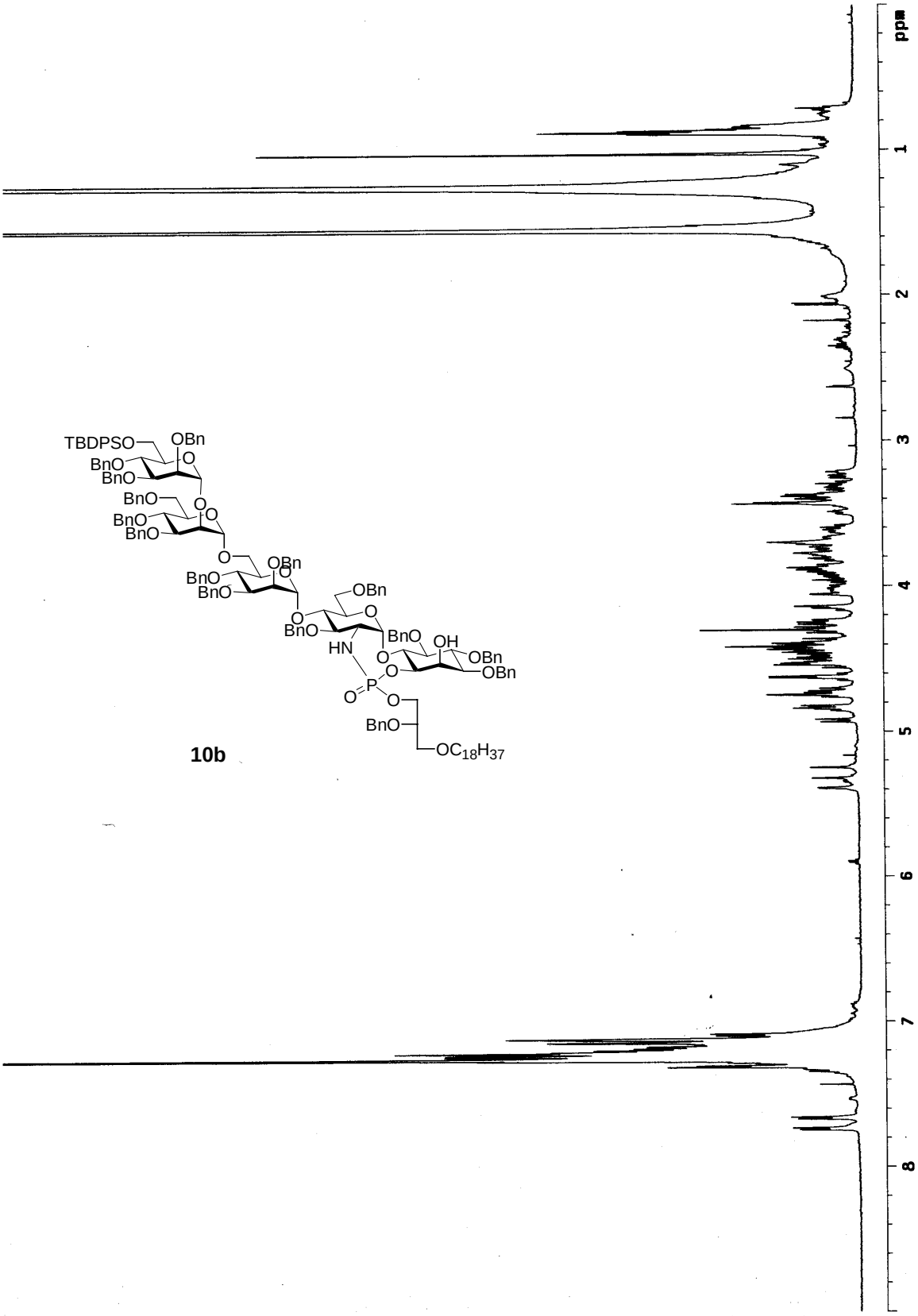
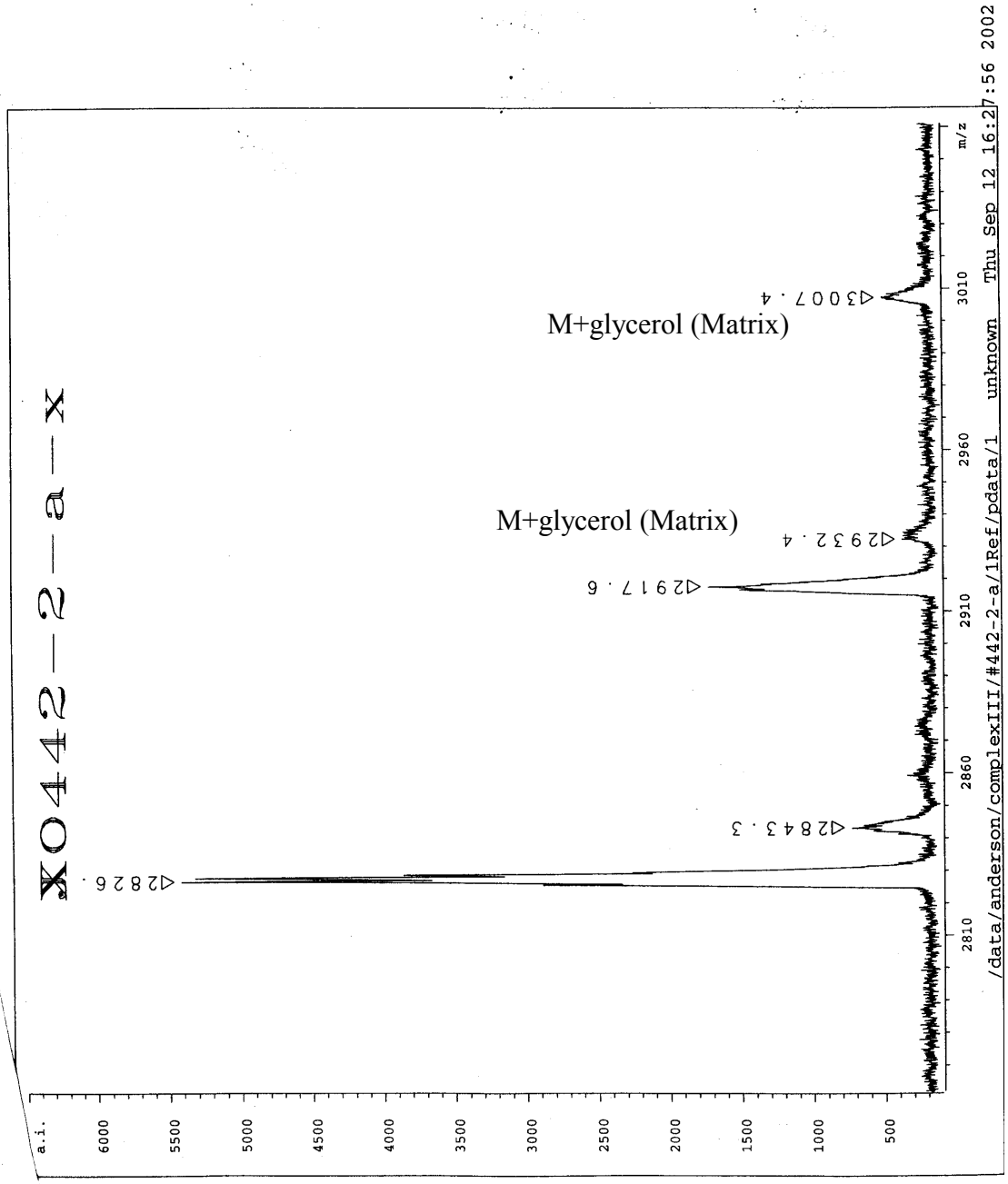
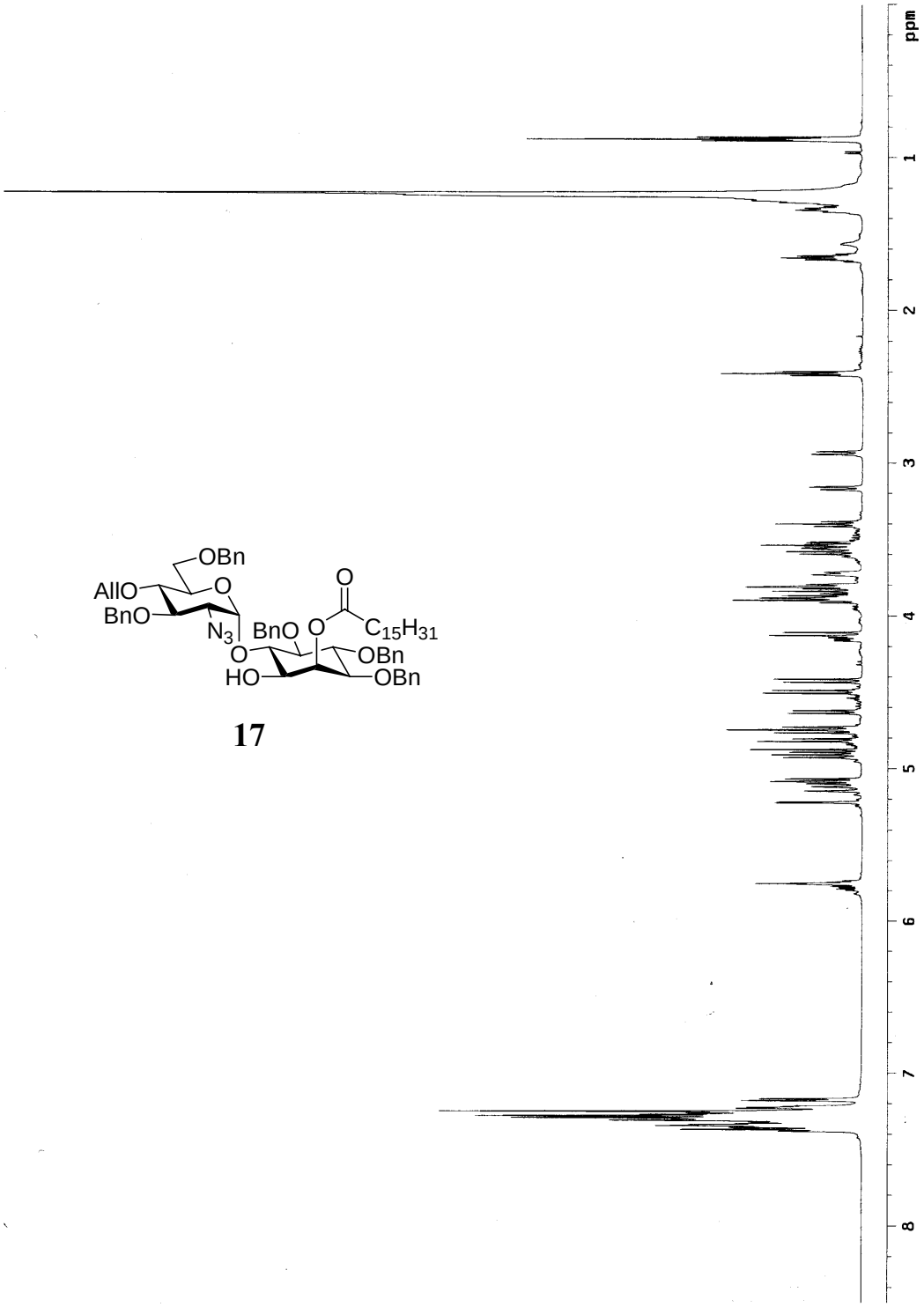
SMOPTS2          0
SMOPTS3          0
DW              0.50  [ns]
DELAY            0 [ns]
Uis1            19.00  [kV]
Uis2            15.50  [kV]
Uref1           20.00  [kV]
Ulens            9.30  [kV]
Uhimass         10.00  [kV]
Reffull         20.00  [kV]
UdetL            1.50  [kV]
UdetR            1.65  [kV]
Udef1            2.00  [kV]
REPHZ            3.00  [Hz]
ATTEN            71.0
ML1          735341.085
ML2             398.985
ML3              -0.009
HITURBO no
GDEON   yes
GDEDLY  medium
DEFLON  yes
RLNSBND no
LLNSBND no
UIS2BND no
DPCAL1          690.66
DPMASS          300.00  [Da]
RBNDVAL           0.33
LBNDVAL           0.28
IS2BNDV           0.91
CMT1    Cyto C -SDS PAGE-PreElectrp-acetyl-met (after c
        >on
CMT2    HCCA-Ziptip-negative
```

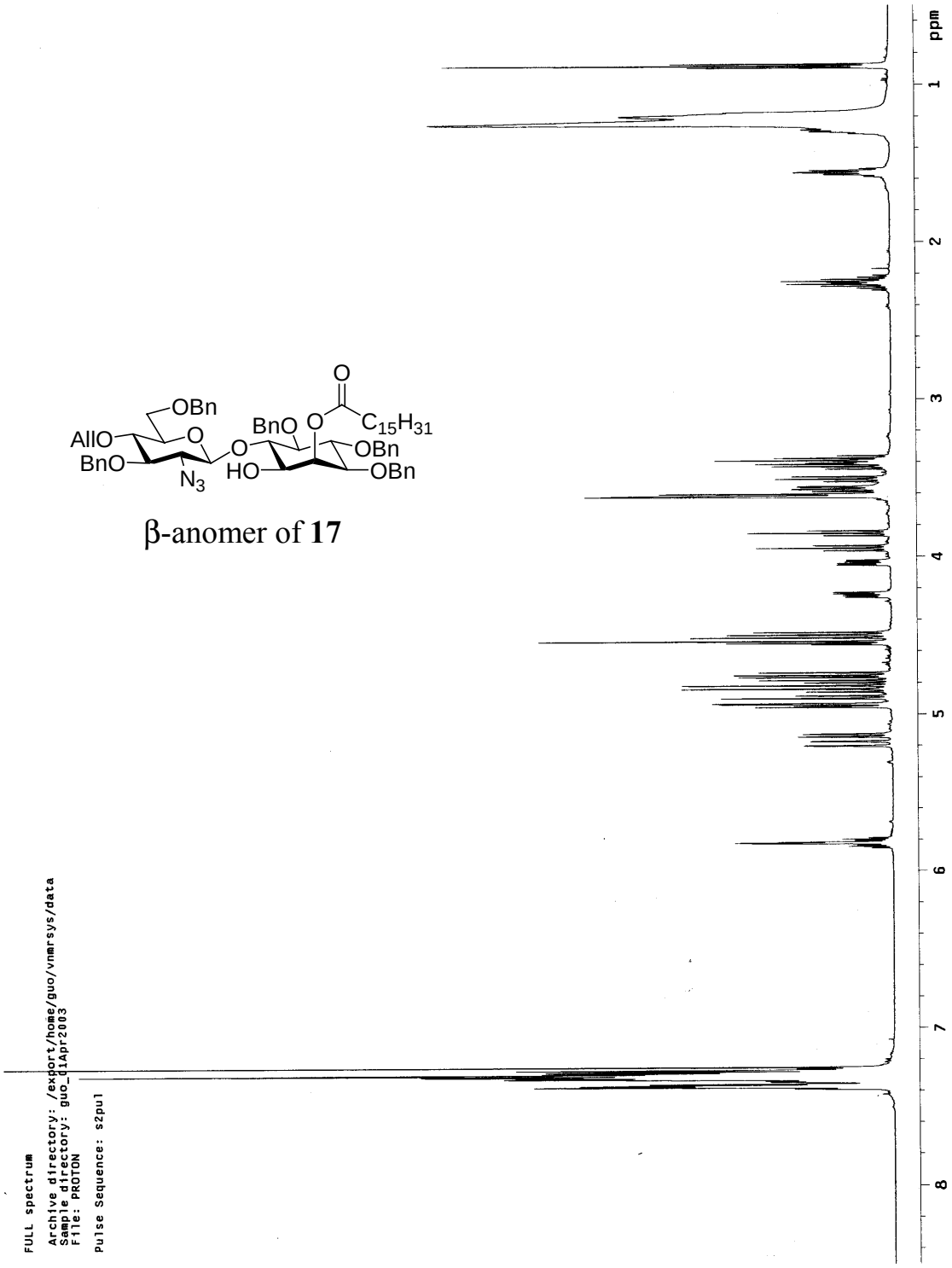

TBDPSO
OBn
BnO
BnO
BnO
BnO
BnO
O
O
O
O
OBn
BnO
BnO
OBn
O
O
BnO
HN
BnO
OH
O
OBn
O
OBn
O
P
O
O
BnO
OC18H37
10b
ppm
1
2
3
4
5
6
7
8

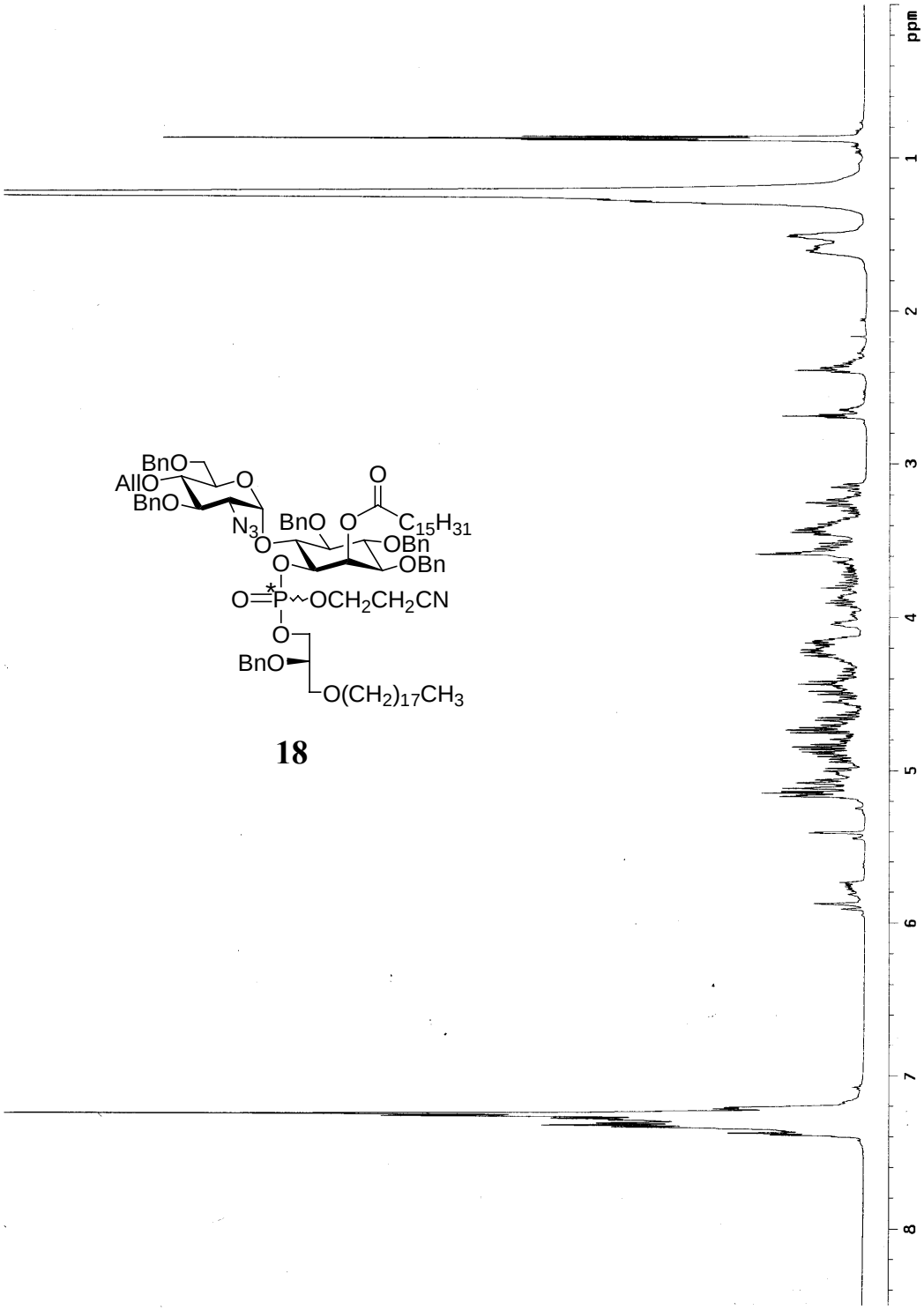

X0442-2-a-x
a.i.
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
42826.
42843.3
42917.6
42932.4
43007.4
M+glycerol (Matrix)
M+glycerol (Matrix)
2810
2860
2910
2960
3010
m/z
/data/anderson/complexIII/#442-2-a/1Ref/pdata/1 unknown Thu Sep 12 16:27:56 2002

17

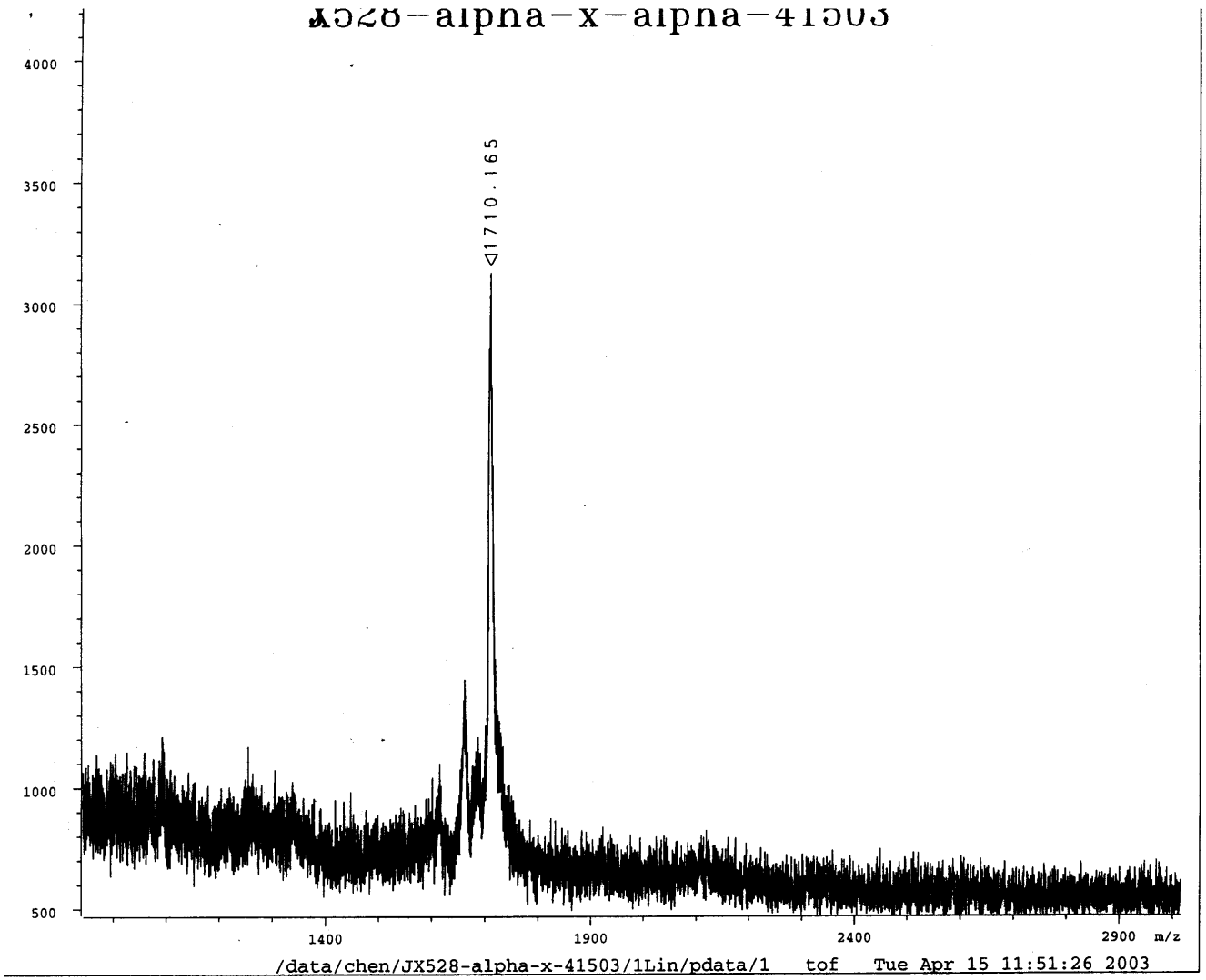


β-anomer of **17**

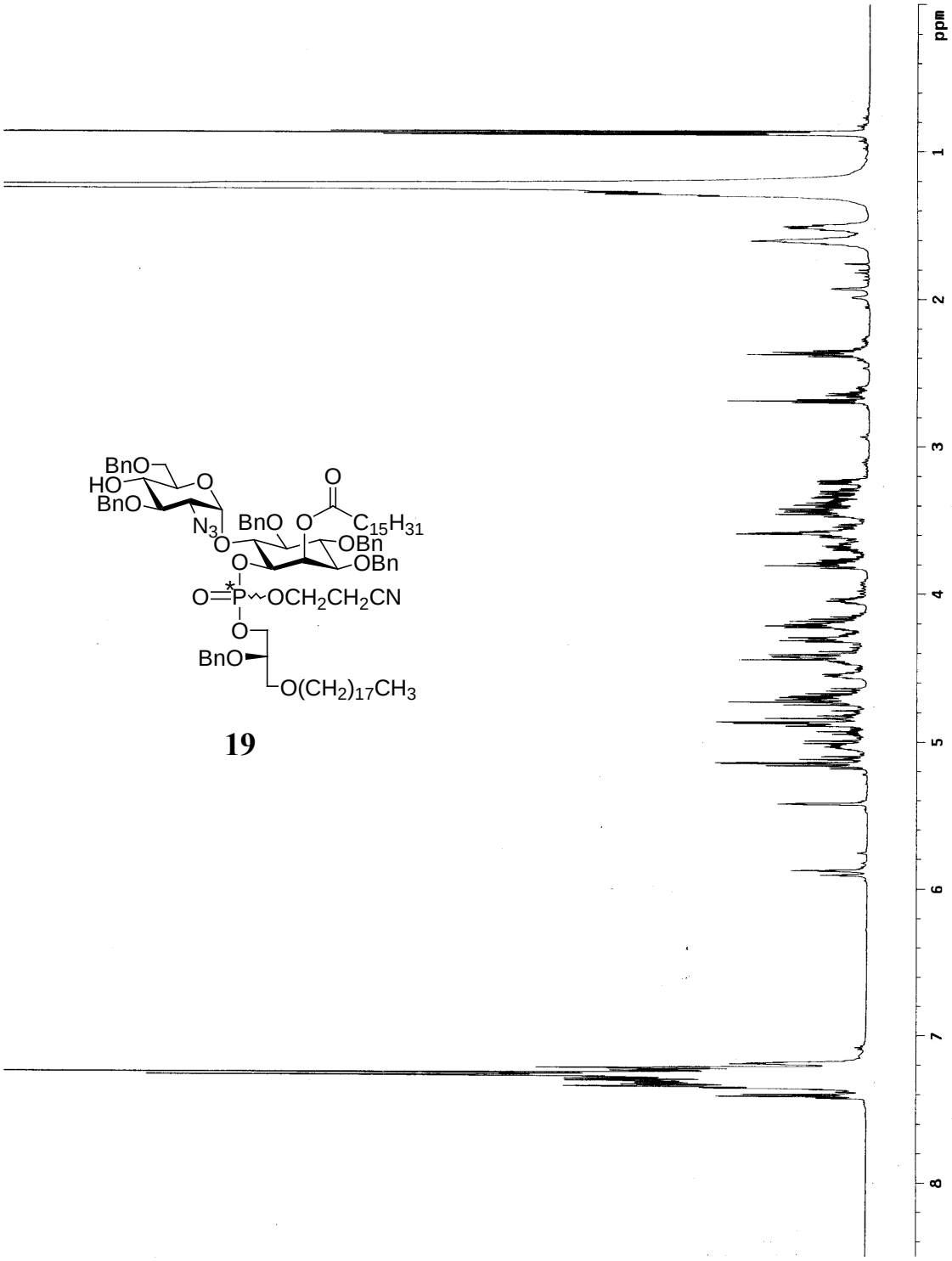



BnO
AllO
BnO
O
N3
O
BnO
O
O
C15H31
OBn
OBn
O
O
*
P
OCH2CH2CN
O
BnO
O(CH2)17CH3
ppm
1
2
3
4
5
6
7
8

18




```
INSTRUM  TOF
OpId     tof
SMPNAM   JX528-alpha-x-41503
AQ_DATE  Tue Apr 15 11:51:02 2003
PATH     /data/chen
POLARI   POS
AQOF_m   Linear
TD          200000
NoSHOTS        208
SMOSUM           0
SMOPTS1          0
SMOPTS2          0
SMOPTS3          0
DW              0.50  [ns]
DELAY           0 [ns]
Uis1           19.00  [kV]
Uis2           15.99  [kV]
Uref1          20.00  [kV]
Ulens           8.50  [kV]
Uhimass        10.00  [kV]
RefFull        20.00  [kV]
Udetl           1.50  [kV]
Udetk           1.65  [kV]
Udefl           2.00  [kV]
REPHZ           3.00  [Hz]
ATTEN           67.0
ML1        2186429.130
ML2            365.375
ML3             -0.034
NITURBO  no
GDSON    yes
GDEDLY   medium
DEFLON   yes
RLNSEND  no
LLNSEND  no
UIS2END  no
DPCAL1         690.66
DPMASS         600.00  [Da]
RENDVAL          0.33
LENDVAL          0.28
IS2ENDV          0.91
CMT1     JX528-alpha-x-alpha-41503
CMT2
```

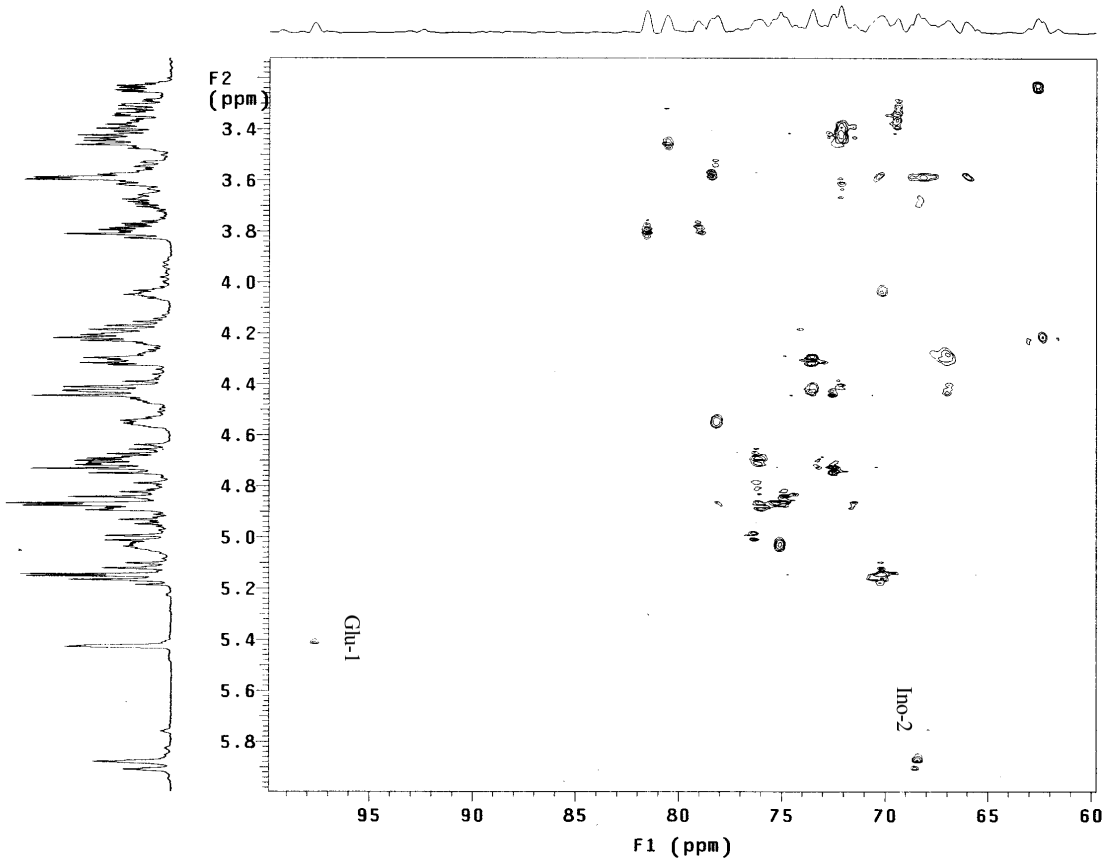

BnO
HO
BnO
O
N3
BnO
O
O
C15H31
OBn
OBn
O
O
*P
OCH2CH2CN
O
BnO
O(CH2)17CH3
19
ppm
1
2
3
4
5
6
7
8

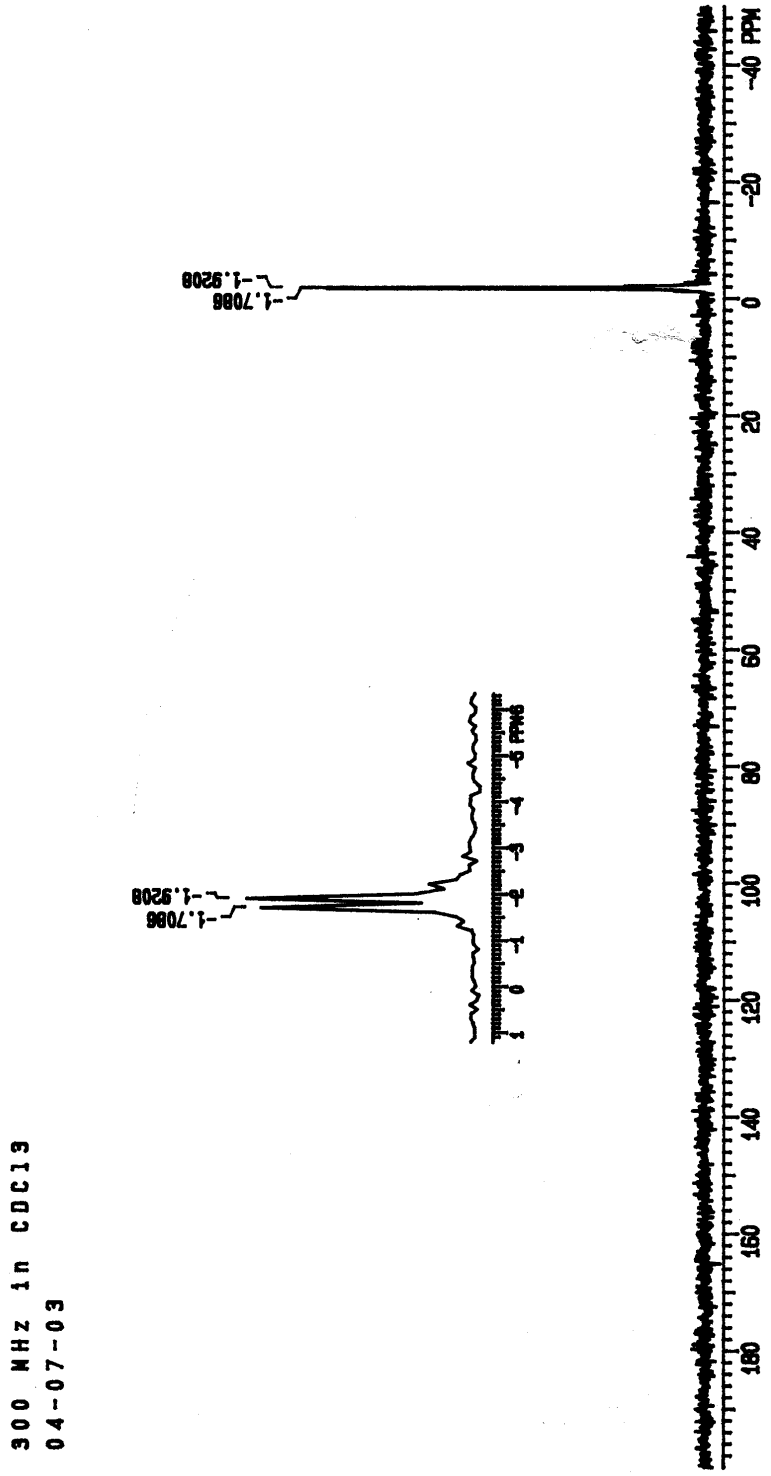
Archive directory: /export/home/guo/vnmrsys/data
Sample directory: JX0407HMQC_08Apr2003
File: HMQC

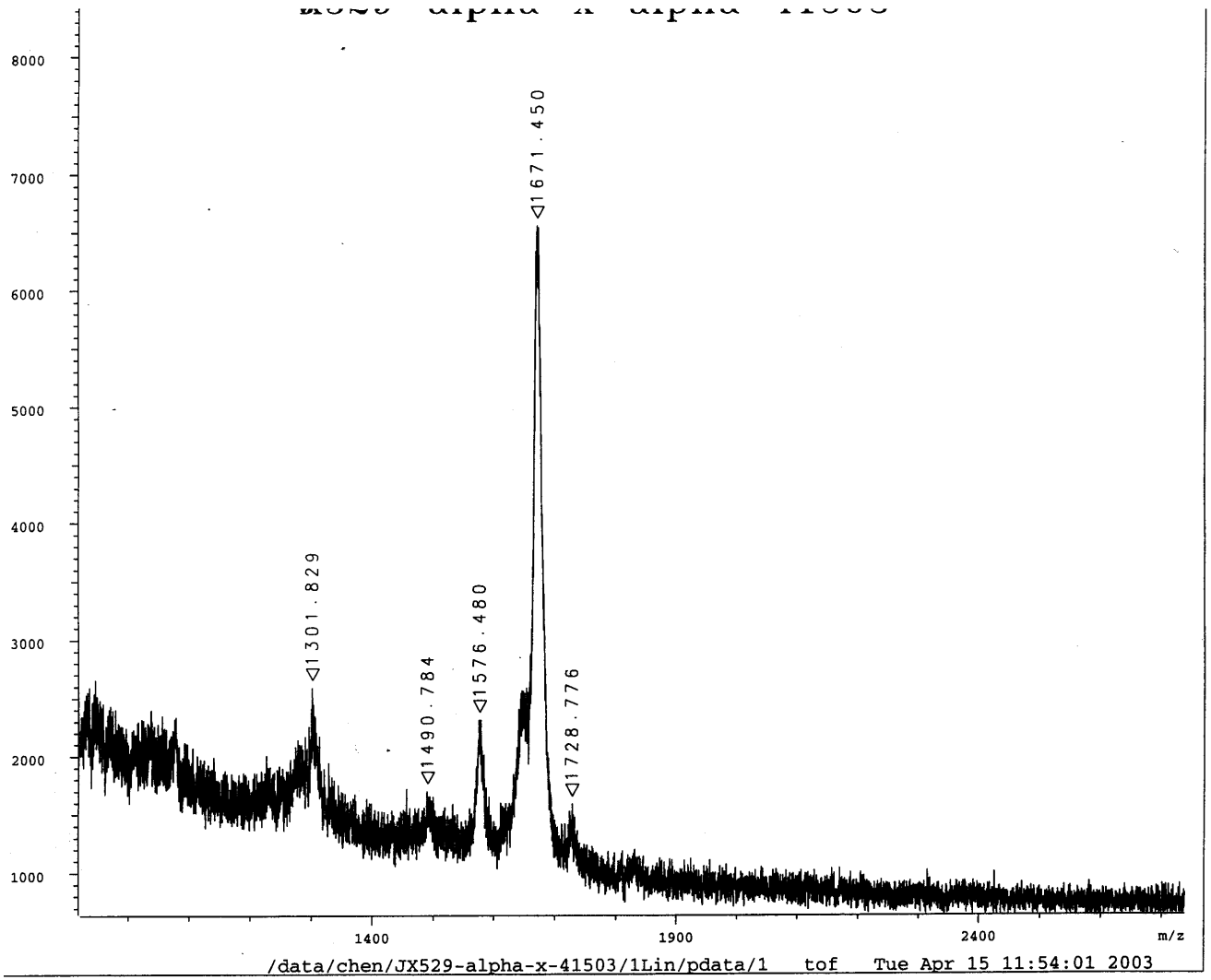
Pulse Sequence: HMQC

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-600 "larry"

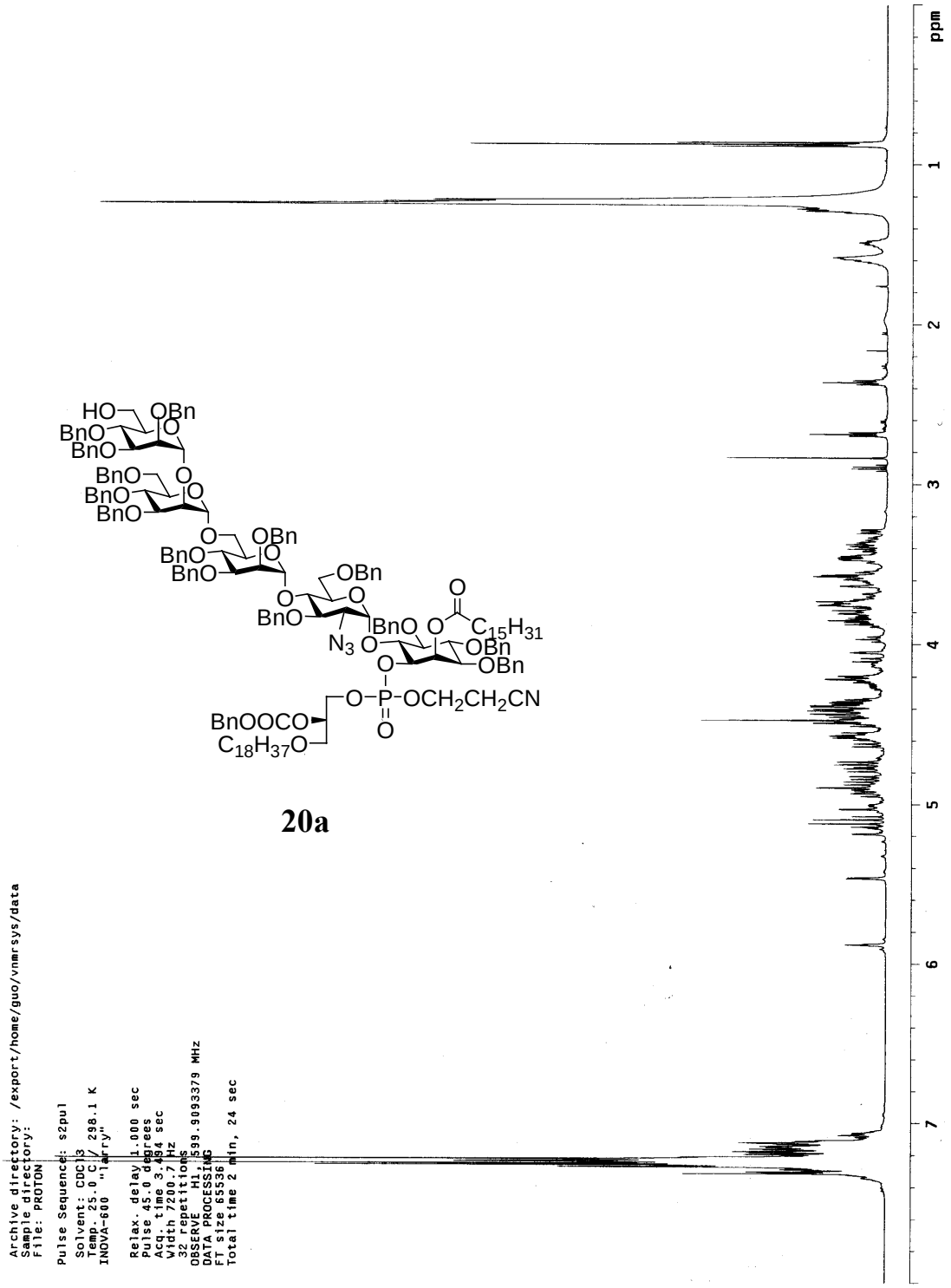
Relax. delay 1.000 sec
Acq. time 0.206 sec
Width 4964.0 Hz
2D Width 25641.0 Hz
16 repetitions
2 x 256 increments
OBSERVE H1, 599.9093379 MHz
DECOUPLE C13, 150.8586006 MHz
Power 41 dB
on during acquisition
off during delay
W40_HX modulated
DATA PROCESSING
Gauss apodization 0.095 sec
F1 DATA PROCESSING
Gauss apodization 0.018 sec
FT size 2048 x 4096
Total time 2 hr, 50 min, 23 sec




300 MHz in CDCl3
04-07-03
-1.7086
-1.9208
-1.7086
-1.9208
PPM
-40
-20
0
20
40
60
80
100
120
140
160
180




```
INSTRUM TOF
OpId    tof
SMPNAM  JX529-alpha-x-41503
AQ_DATE Tue Apr 15 11:53:04 2003
PATH    /data/chen
POLARI  POS
AQOP_m  Linear
TD          200000
NoSHOTS        227
SNOWUM           0
SMOPTS1          0
SMOPTS2          0
SMOPTS3          0
DW               0.50  [ns]
DELAY          0 [ns]
Uis1            19.00  [kV]
Uis2            15.99  [kV]
Uref1           20.00  [kV]
Ulens            8.50  [kV]
Uhimass         10.00  [kV]
RefFull         20.00  [kV]
UdetL            1.50  [kV]
UdetR            1.65  [kV]
Udef1            2.00  [kV]
REPHZ            3.00  [Hz]
ATTEN           67.0
ML1       2186429.130
ML2           365.375
ML3            -0.034
HYTURBO no
GDEON   yes
GDEDLY  medium
DEFLON  yes
RLNSBND no
LLNSBND no
UIS2BND no
DPCAL1          690.66
DPMASS          600.00  [Da]
RBNDVAL           0.33
LBNDVAL           0.28
IS2BNDV           0.91
CMT1    JX529-alpha-x-alpha-41503
CMT2
```


Archive directory: /export/home/guo/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-600 "larry"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.494 sec
Width 7200.7 Hz
32 repetitions
OBSERVE H1, 599.9993379 MHz
DATA PROCESSING
FT size 65536
Total time 2 min, 24 sec
ppm
1
2
3
4
5
6
7

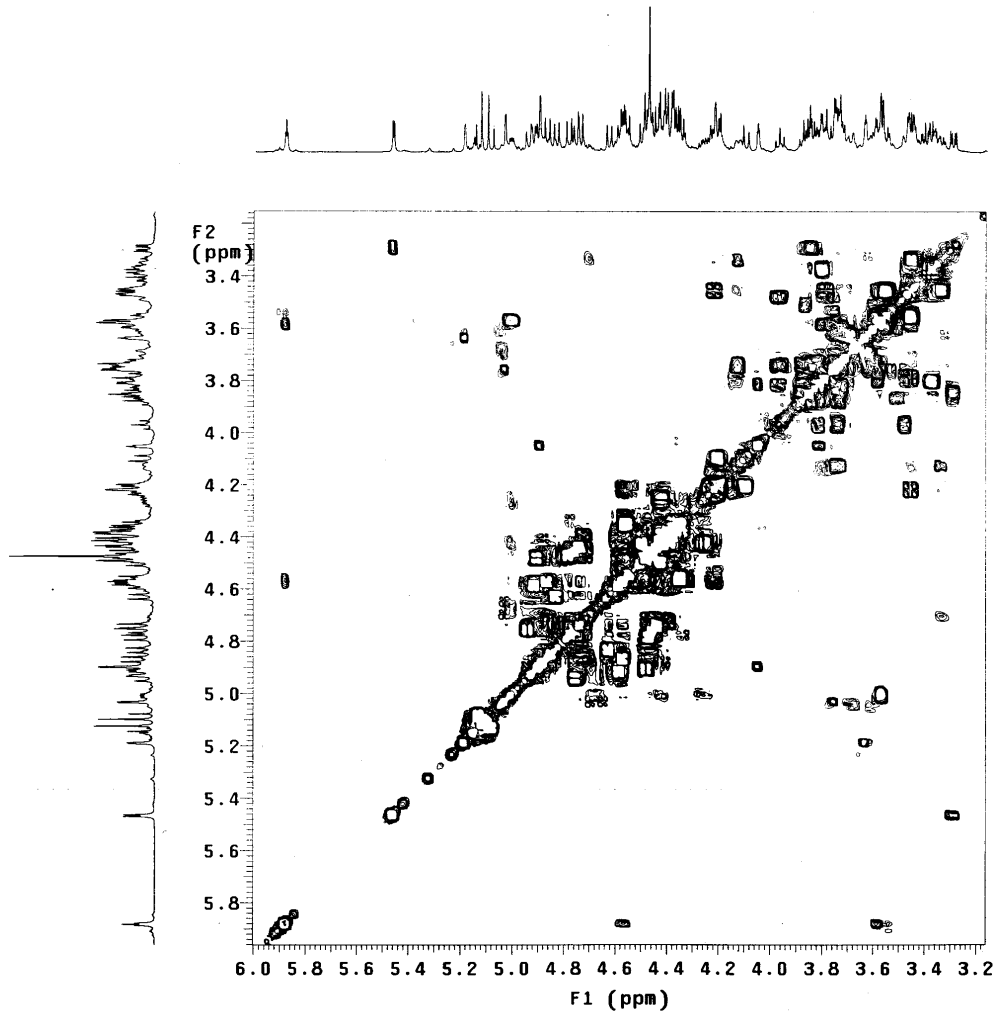
20a

Archive directory: /export/home/guo/vnmrsys/data
Sample directory: JXpenta1_28Apr2003
File: COSY

Pulse Sequence: COSY

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-600 "larry"

Relax. delay 1.000 sec
Acq. time 0.203 sec
Width 5049.2 Hz
2D Width 5049.2 Hz
8 repetitions
256 increments
OBSERVE H1, 599.9093379 MHz
DATA PROCESSING
Sq. sine bell 0.101 sec
F1 DATA PROCESSING
Sq. sine bell 0.051 sec
FT size 2048 x 2048
Total time 42 min, 53 sec

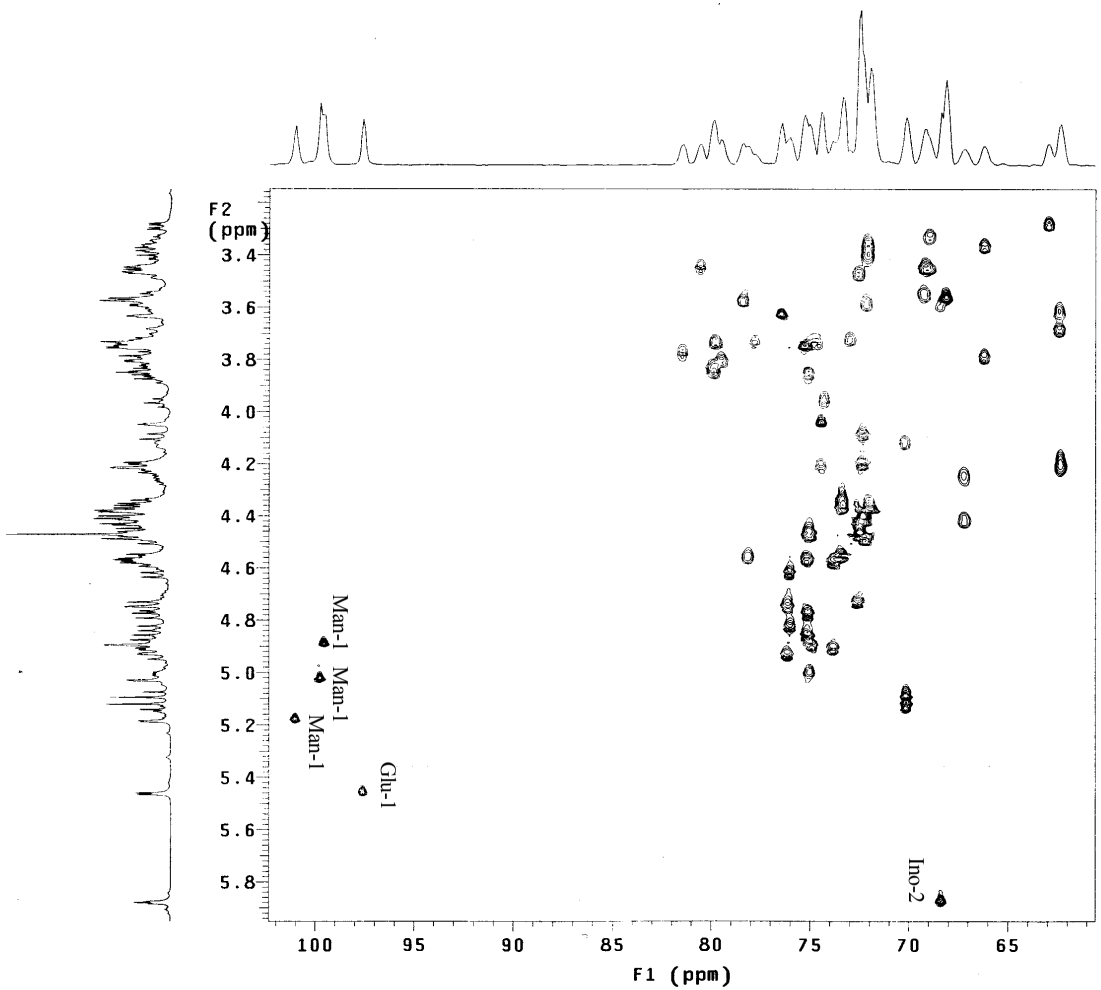


Archive directory: /export/home/guo/vnmrsys/data
Sample directory: JXpentahmqc_30Apr2003
File: HMQC

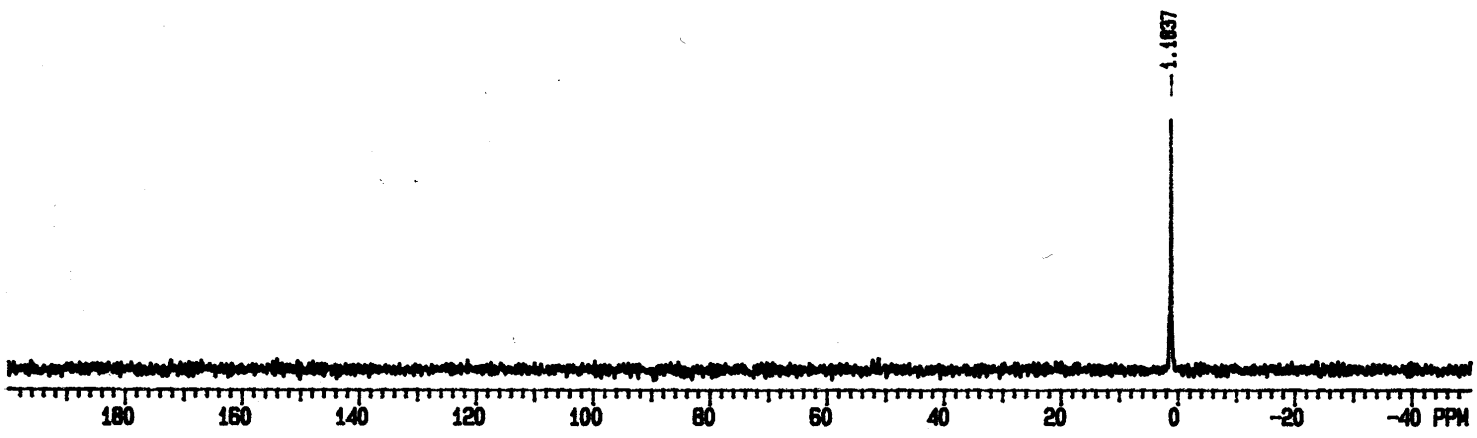
Pulse Sequence: HMQC

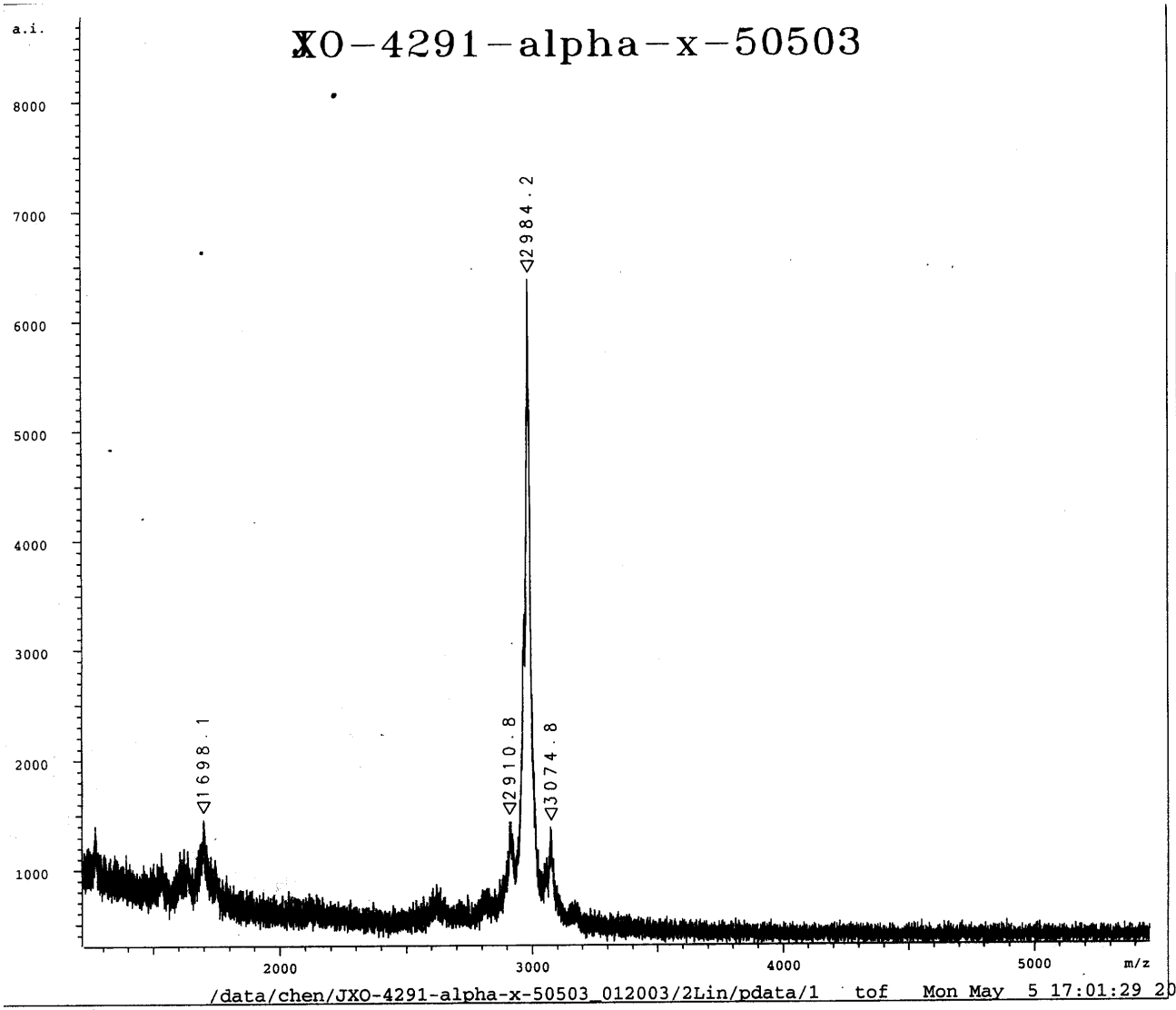
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-600 "larry"

Relax. delay 1.000 sec
Acq. time 0.202 sec
Width 5071.0 Hz
2D Width 25641.0 Hz
16 repetitions
2 x 256 increments
OBSERVE H1, 599.9093379 MHz
DECOUPLE C13, 150.8586006 MHz
Power 41 dB
on during acquisition
off during delay
W40_HX modulated
DATA PROCESSING
Gauss apodization 0.093 sec
F1 DATA PROCESSING
Gauss apodization 0.018 sec
FT size 2048 x 4096
Total time 2 hr, 49 min, 47 sec

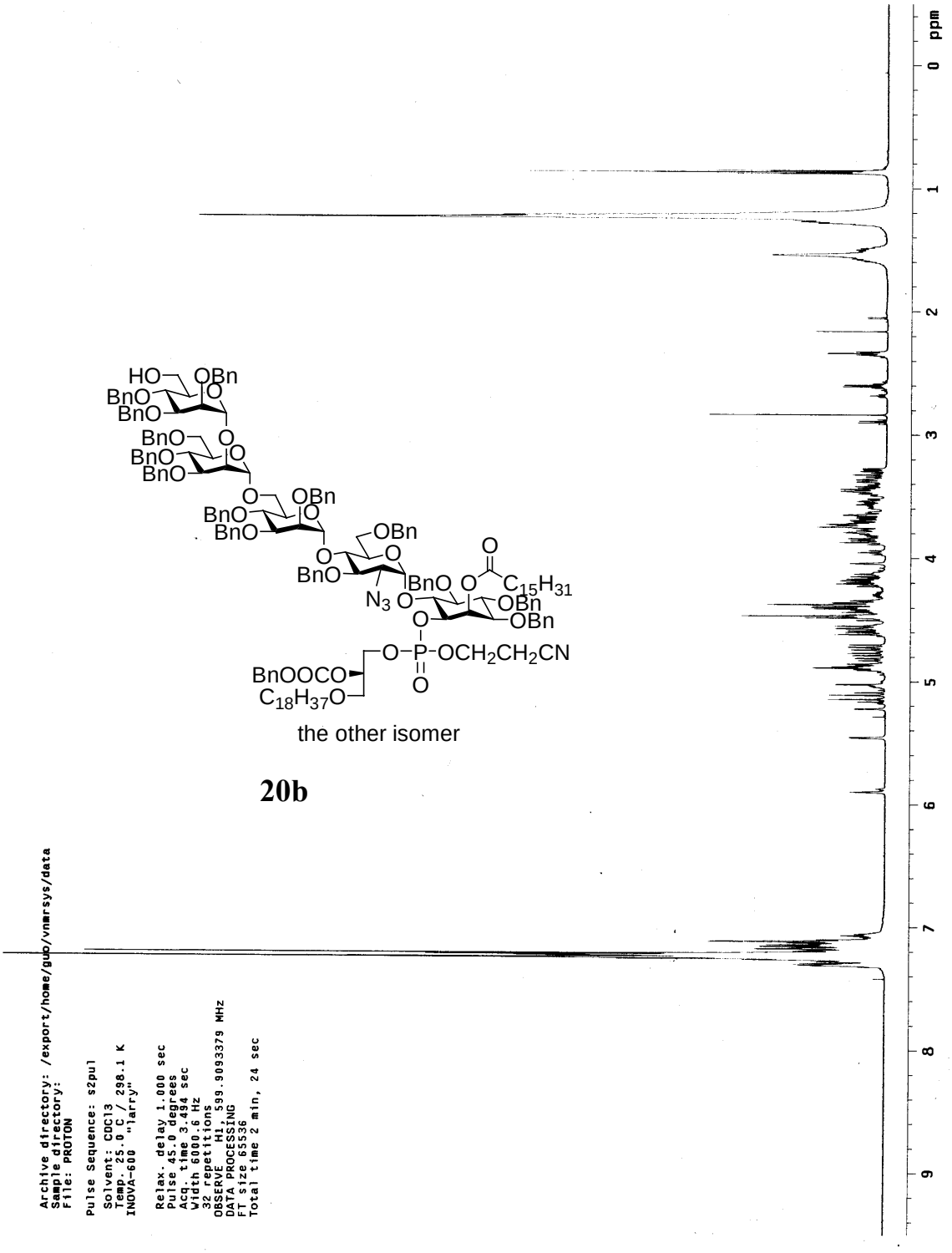


300 MHZ IN CDCL3
P-SPEC

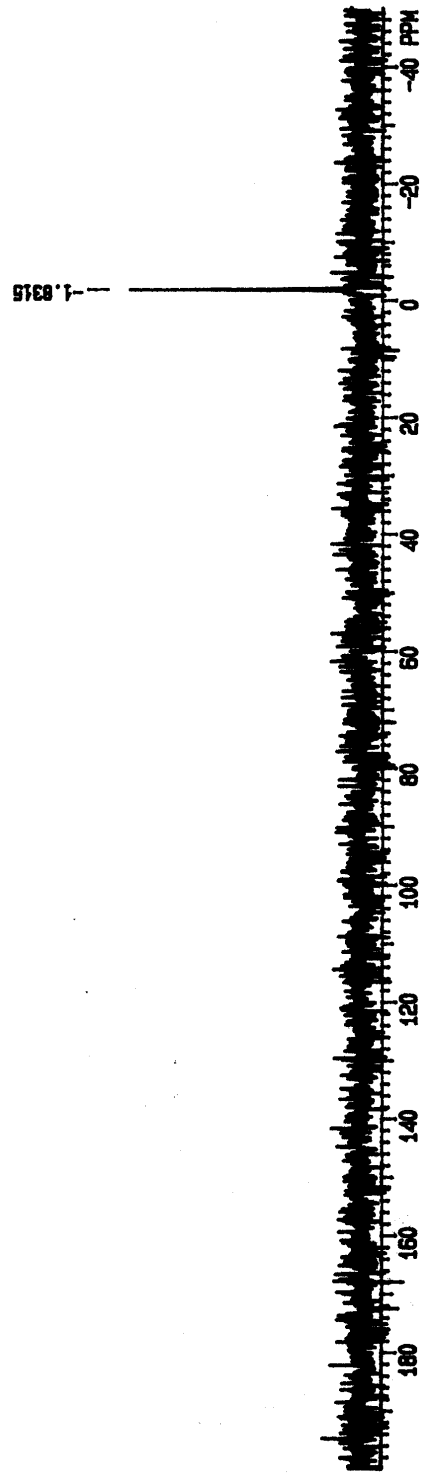


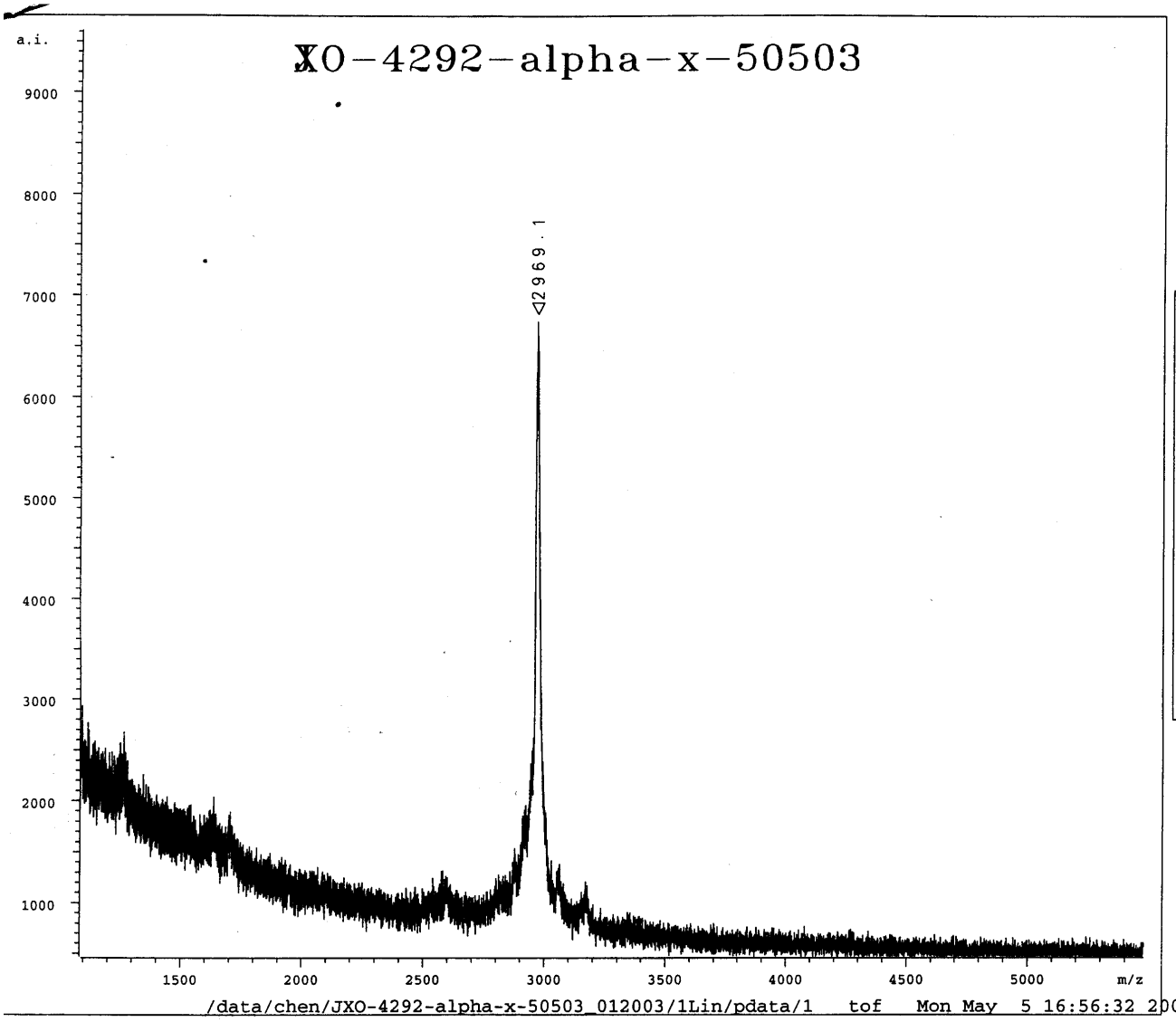



```
INSTRUM TOF
OpId    tof
SMPNAM  JXO-4291-alpha-x-50503_012003
AQ_DATE Mon May  5 17:00:49 2003
PATH    /data/chen
POLARI  POS
AQOP_m  Linear
TO          200000
NoSHOTS        215
SMONUM           0
SMOPTS1          0
SMOPTS2          0
SMOPTS3          0
DW               0.50  [ns]
DELAY          0 [ns]
Uis1            19.00  [kV]
Uis2            15.99  [kV]
Uref1           20.00  [kV]
Ulens            8.50  [kV]
Unimass         10.00  [kV]
RefFull         20.00  [kV]
UdetL            1.50  [kV]
UdetR            1.65  [kV]
Udef1            2.00  [kV]
REPHZ            3.00  [Hz]
ATTEN           70.0
ML1        2176752.387
ML2            295.311
ML3             -0.040
HITURBO no
GDEON   yes
GDEDLY  medium
DEFLON  yes
RLNSBND no
LLNSBND no
UIS2BND no
DPCAL1          690.66
DPMASS          600.00  [Da]
RBMDVAL           0.33
LBMDVAL           0.28
IS2BMDV           0.91
CMT1    JXO-4291-alpha-x-50503
CMT2
```

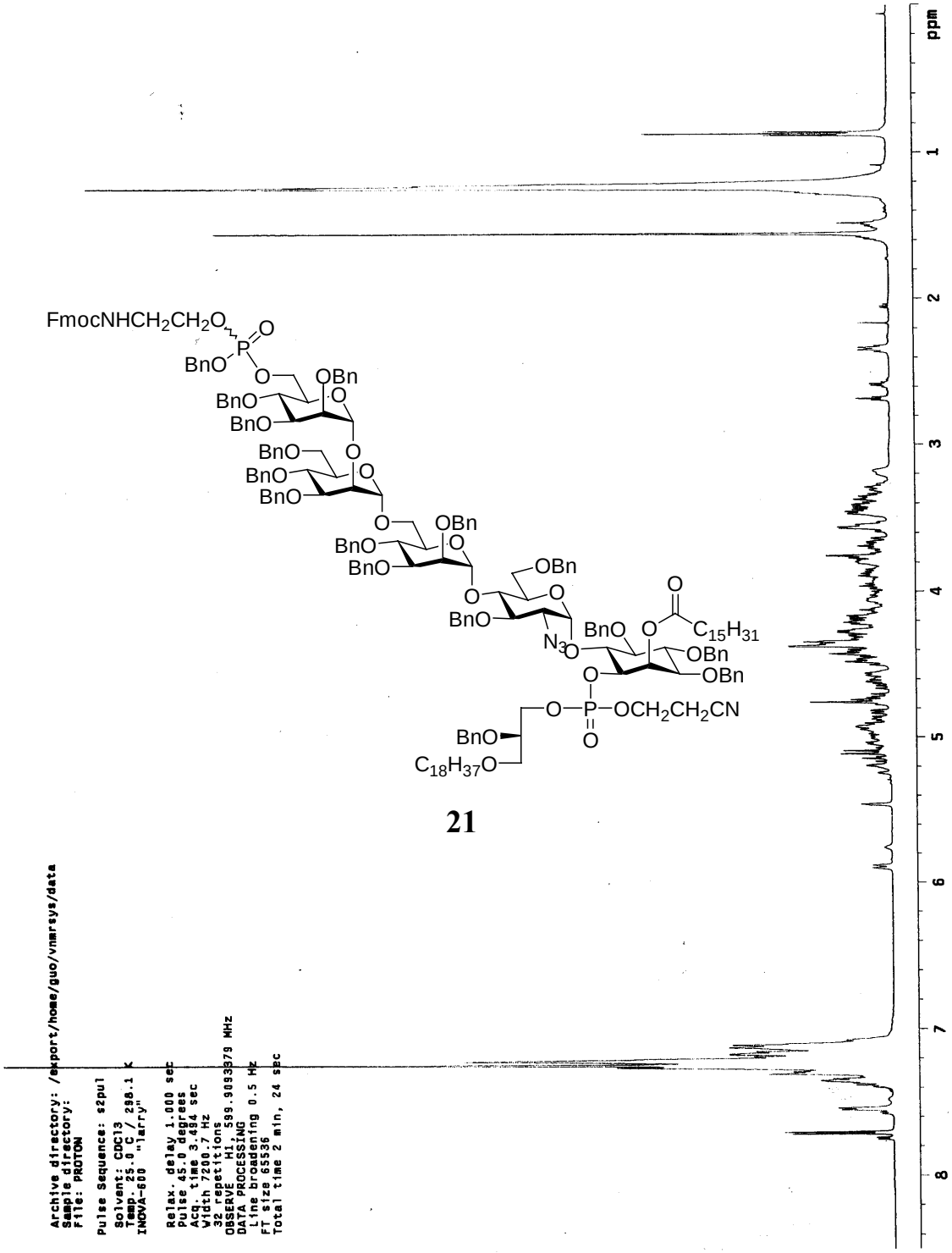

Archive directory: /export/home/gub/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-600 "larry"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.494 sec
Width 6000.6 Hz
32 repetitions
OBSERVE H1, 599.9093379 MHz
DATA PROCESSING
FT size 65536
Total time 2 min, 24 sec
ppm
0
1
2
3
4
5
6
7
8
9
HO
OBn
BnO
O
N3
C15H31
OCH2CH2CN
BnOOCO
C18H37O
the other isomer

20b

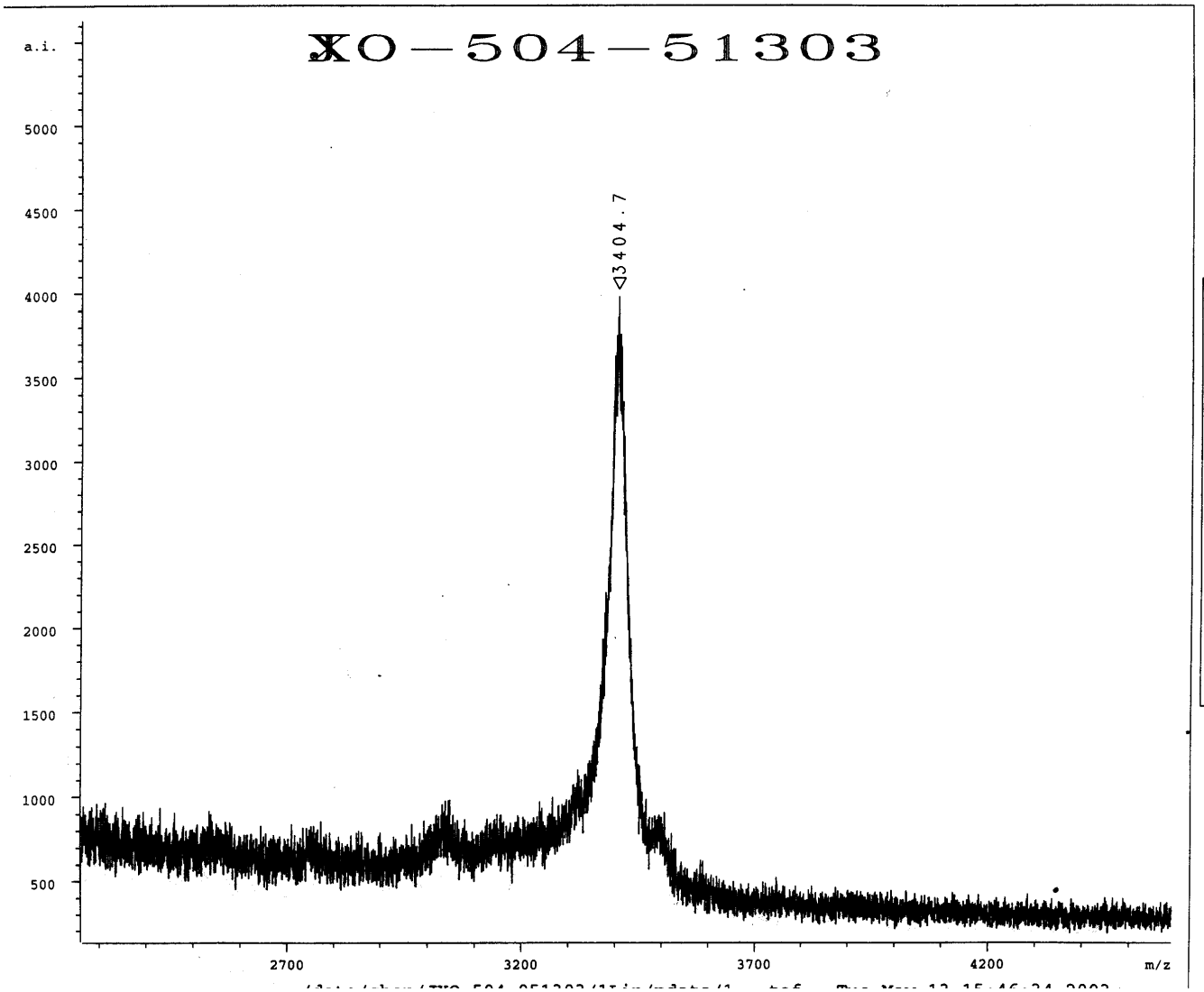

-1.8315
-40 PPM
-20
0
20
40
60
80
100
120
140
160
180



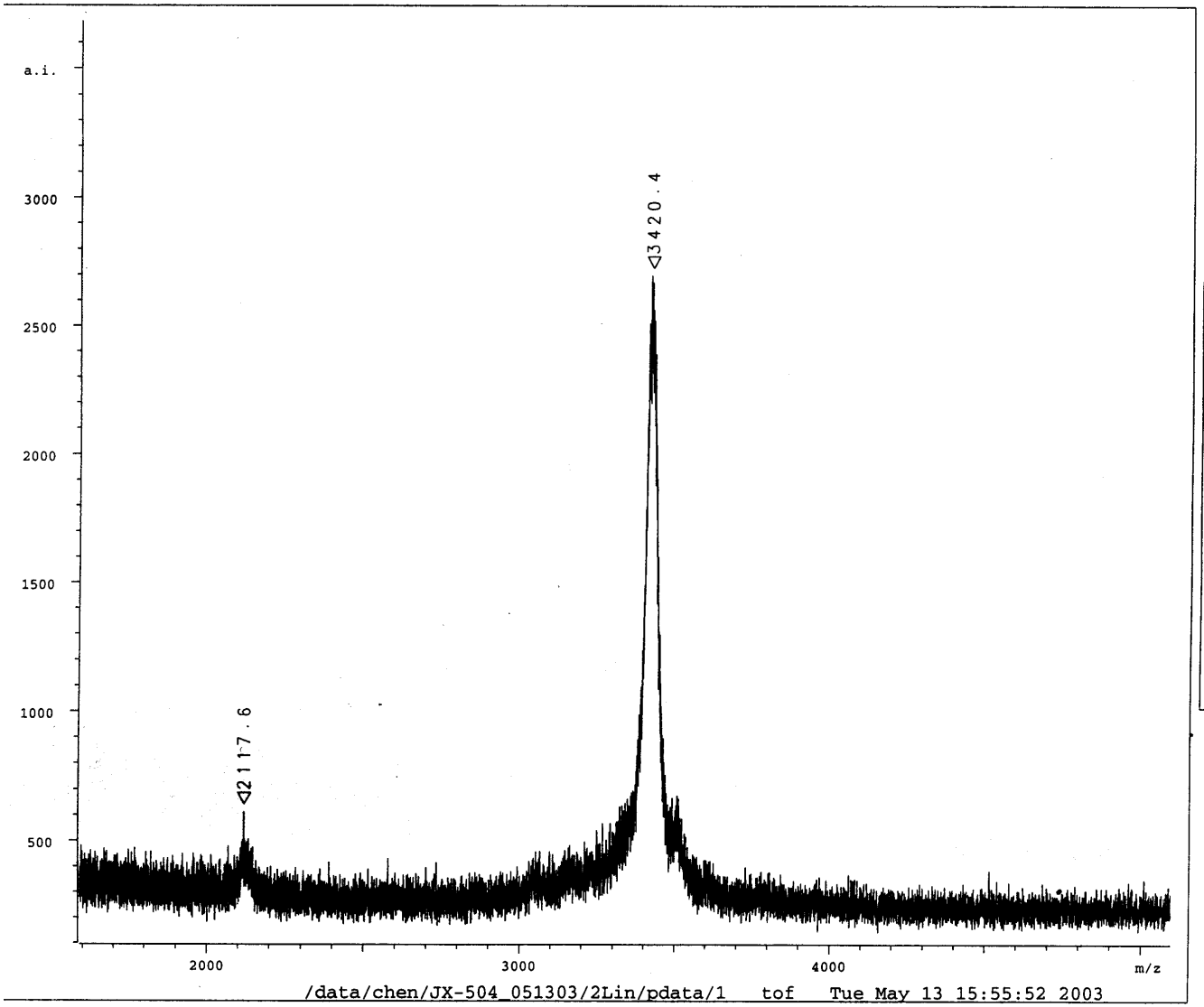

```
INSTRUM  TOF
OpId     tof
SMPNAM   JXO-4292-alpha-x-50503_012003
AQ_DATE  Mon May  5 16:56:02 2003
PATH     /data/chen
POLARI   POS
AQOP_m   Linear
TD          200000
NoSHOTS        250
SMONUM           0
SMOPTS1          0
SMOPTS2          0
SMOPTS3          0
DW              0.50  [ns]
DELAY           0 [ns]
Uis1           19.00  [kV]
Uis2           15.99  [kV]
Uref1          20.00  [kV]
Ulens           8.50  [kV]
Uhimass        10.00  [kV]
RefFull        20.00  [kV]
UdetL           1.50  [kV]
UdetR           1.65  [kV]
Udef1           2.00  [kV]
REPHZ           3.00  [Hz]
ATTEN          68.0
ML1        2176752.387
ML2            295.311
ML3             -0.040
HITURBO  no
GDEON    yes
GDEDLY   medium
DEFLON   yes
RLNSBND  no
LLNSBND  no
UIS2BND  no
DPCAL1         690.66
DPMASS         600.00  [Da]
RBNDVAL          0.33
LBNDVAL          0.28
IS2BNDV          0.91
CMT1     JXO-4292-alpha-x-50503
CMT2
```


Archive directory: /ekport/home/guo/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 / 298.1 K
INOVA-600 "Larry"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.494 sec
Width 7200.7 Hz
32 repetitions
OBSERVE H1, 599.9893879 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 2 min, 24 sec
ppm
1
2
3
4
5
6
7
8

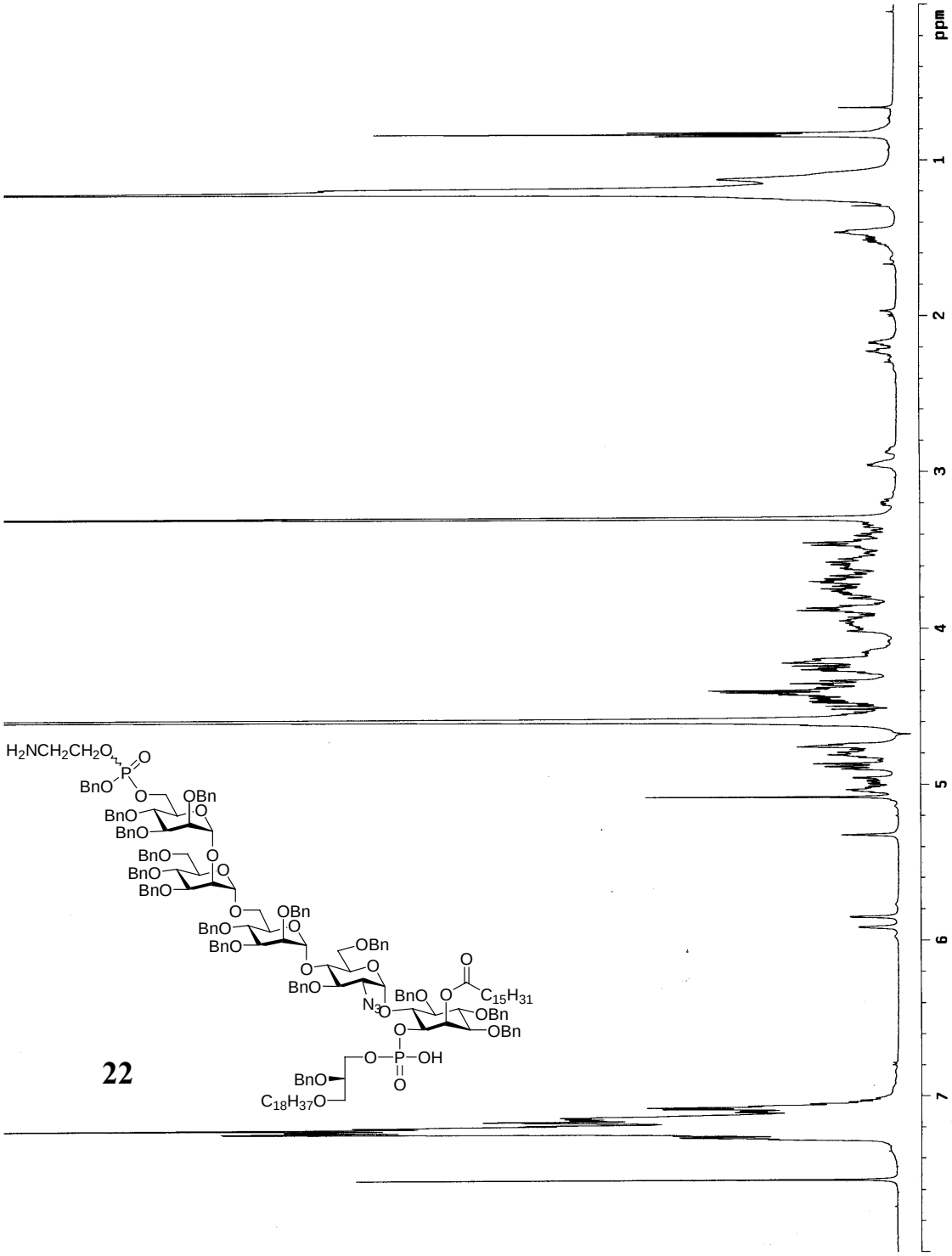
21




```
INSTRUM TOF
OpId    tof
SMPNAM  JXO-504_051303
AQ_DATE Tue May 13 15:45:45 2003
PATH    /data/chen
POLARI  POS
AQOP_m  Linear
TO          200000
NoSHOTS        150
SMONUM           0
SMOPTS1          0
SMOPTS2          0
SMOPTS3          0
DW               0.50  [ns]
DELAY          0 [ns]
Uis1            19.00  [kV]
Uis2            15.50  [kV]
Uref1           20.00  [kV]
Ulens            9.30  [kV]
Uhimass         10.00  [kV]
RefPul1         20.00  [kV]
UdetL            1.50  [kV]
UdetR            1.65  [kV]
Udef1            2.00  [kV]
REPHZ            3.00  [Hz]
ATTEN           66.0
ML1        2209187.686
ML2            430.683
ML3              0.008
HITURBO no
GDEON   yes
GDHOLY  medium
DEFLON  yes
RLNSBND no
LLNSBND no
UIS2BND no
DPCAL1         690.66
DPMASS         500.00  [Da]
RBNDVAL          0.33
LBNDVAL          0.28
IS2BNDV          0.91
CMT1    JXO-504-51303
CMT2
```




```
INSTRUM TOF
OpId    tof
SMPNAM  JX-504_051303
AQ_DATE Tue May 13 15:55:15 2003
PATH    /data/chen
POLARI  POS
AQOP_m  Linear
TD          200000
NoSHOTS        150
SMONUM           0
SMOPTS1          0
SMOPTS2          0
SMOPTS3          0
DW              0.50  [ns]
DELAY          0 [ns]
Uis1           19.00  [kV]
Uis2           15.50  [kV]
Uref1          20.00  [kV]
Ulens           9.30  [kV]
Uhimass        10.00  [kV]
RefFull        20.00  [kV]
UdetL           1.50  [kV]
UdetR           1.65  [kV]
Udef1           2.00  [kV]
REPHZ           3.00  [Hz]
ATTEN           65.0
NL1       2209187.686
NL2           430.683
NL3             0.008
HITURBO no
GDEON   yes
GDEDLY  medium
DEFLON  yes
RLNSEND no
LLNSEND no
UISZEND no
DPCAL1        690.66
DPMASS        500.00  [Da]
RBNDVAL         0.33
LBNDVAL         0.28
IS2BNDV         0.91
CMT1    JX-504-51303
CMT2
```

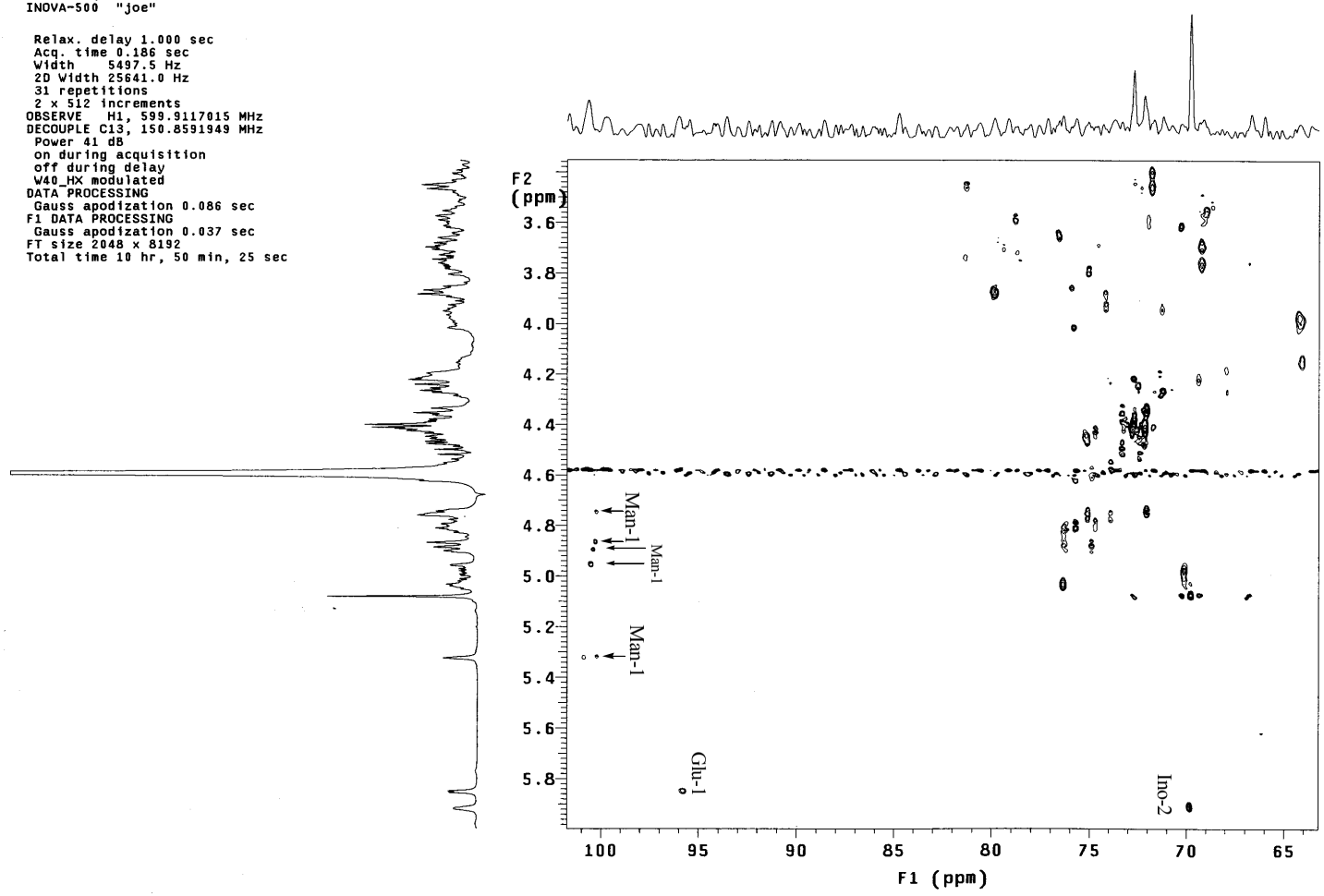

ppm
1
2
3
4
5
6
7
22

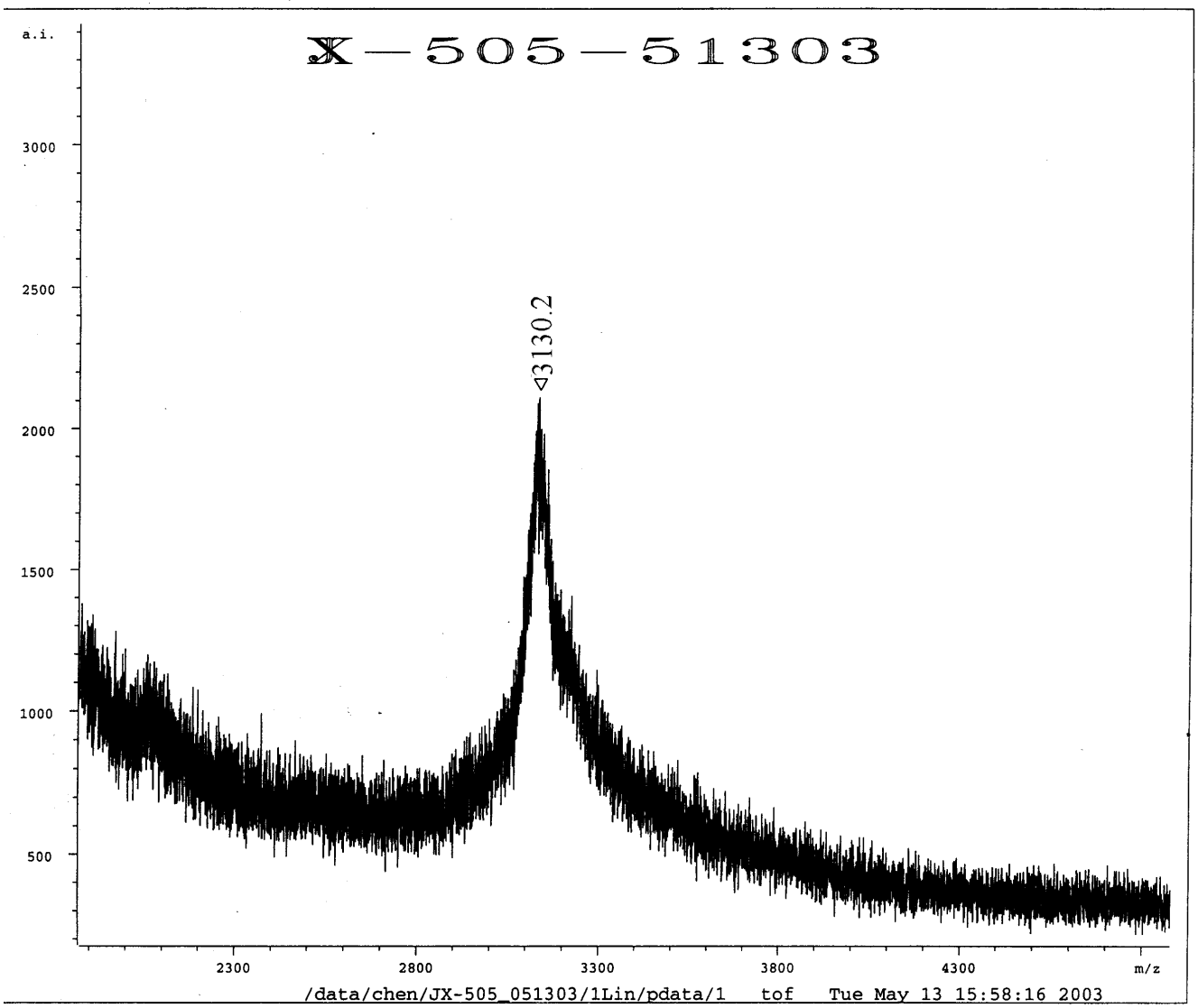
Archive directory: /export/home/guo/vnmrsys/data
Sample directory: JX05052_05May2003

Pulse Sequence: HMQC

Solvent: CD3OD
Temp. 25.0 C / 298.1 K
File: HMQC
INOVA-500 "joe"

Relax. delay 1.000 sec
Acq. time 0.186 sec
Width 5497.5 Hz
2D Width 25641.0 Hz
31 repetitions
2 x 512 increments
OBSERVE H1, 599.9117015 MHz
DECOUPLE C13, 150.8591949 MHz
Power 41 dB
on during acquisition
off during delay
W40_HX modulated
DATA PROCESSING
Gauss apodization 0.086 sec
F1 DATA PROCESSING
Gauss apodization 0.037 sec
FT size 2048 x 8192
Total time 10 hr, 50 min, 25 sec



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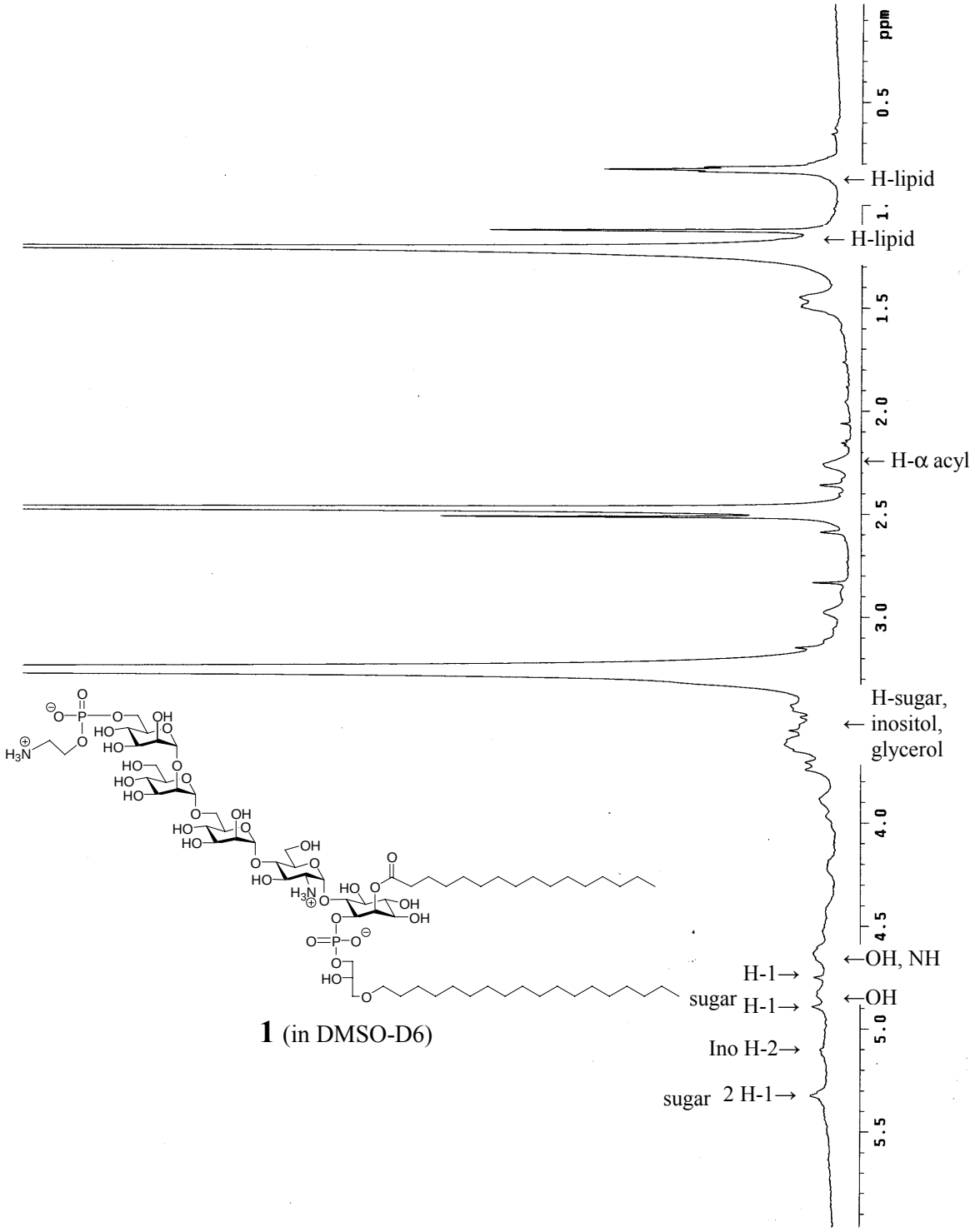
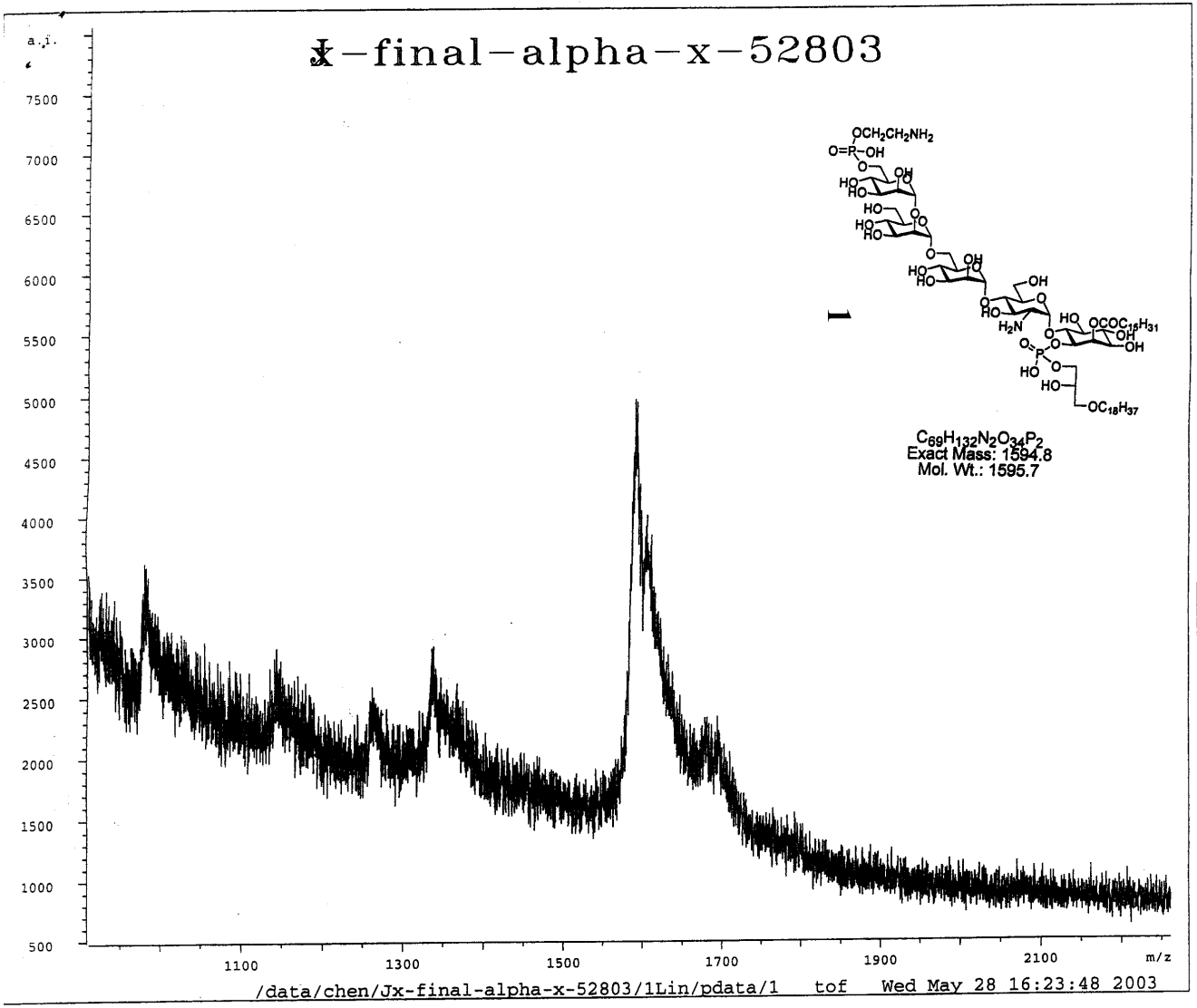
INSTRUM TOF
OpId    tof
SMPNAM  JX-505_051303
AQ_DATE Tue May 13 15:57:55 2003
PATH    /data/chen
POLARI  POS
AQOP_m  Linear
TD          200000
NoSHOTS        150
SMONUM           0
SMOPTS1          0
SMOPTS2          0
SMOPTS3          0
DW               0.50  [ns]
DELAY          0 [ns]
Uis1            19.00  [kV]
Uis2            15.50  [kV]
Uref1           20.00  [kV]
Ulens            9.30  [kV]
Uhimass         10.00  [kV]
RefFull         20.00  [kV]
UdetL            1.50  [kV]
UdetR            1.65  [kV]
Udef1            2.00  [kV]
REPHZ            3.00  [Hz]
ATTEN           59.0
ML1          2209187.686
ML2           430.683
ML3             0.008
HITURBO no
GDEON   yes
GDEDLY  medium
DEFLON  yes
RLNSBND no
LLNSBND no
UIS2BND no
DPCAL1          690.66
DPMASS          500.00  [Da]
RBNDVAL           0.33
LBNDVAL           0.28
IS2BNDV           0.91
CMT1    JX-505-51303
CMT2
```


ppm
0.5
1.
1.5
2.0
2.5
3.0
4.0
4.5
5.0
5.5
← H-lipid
← H-lipid
← H-α acyl
H-sugar,
← inositol,
glycerol
←OH, NH
←OH
H-1→
sugar H-1→
Ino H-2→
sugar 2 H-1→
1 (in DMSO-D6)

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INSTRUM TOF
OpId    tof
SMPNAM  Jx-final-alpha-x-52803
AQ_DATE Wed May 28 16:22:11 2003
PATH    /data/chen
POLARI  POS
AQOP_m  Linear
TD          200000
NoSHOTS        200
SMONUM           0
SMOPTS1          0
SMOPTS2          0
SMOPTS3          0
DW              0.50  [ns]
DELAY          0 [ns]
Uis1           19.00  [kV]
Uis2           15.99  [kV]
Uref1          20.00  [kV]
Ulens           8.50  [kV]
Uhimass        10.00  [kV]
RefFull        20.00  [kV]
UdetL           1.50  [kV]
UdetR           1.65  [kV]
Udef1           2.00  [kV]
REPHZ           3.00  [Hz]
ATTEN           67.0
ML1        2229736.203
ML2            541.112
ML3              0.031
HITURBO no
GDEON   yes
GDEDLY  medium
DEFLON  yes
RLNSBND no
LLNSBND no
UIS2BND no
DPCAL1         690.66
DPMASS         600.00  [Da]
RBNDVAL          0.33
LBNDVAL          0.28
IS2BNDV          0.91
CMT1    Jx-final-alpha-x-52803
CMT2
```