Supporting Information

An Expeditious Synthesis of Sialic Acid Derivatives by Copper(I)-Catalyzed Stereodivergent Propargylation of Unprotected Aldoses

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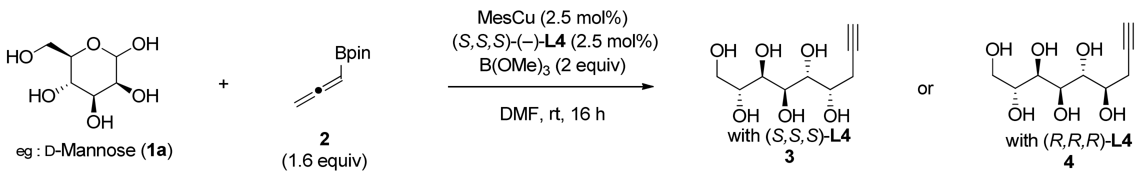
1. General Information

NMR spectra were recorded on JEOL JNM-LA500 (500 MHz for ^{1}H NMR and 125 MHz for ^{13}C NMR), JEOL ECX500 (500 MHz for ^{1}H NMR and 125 MHz for ^{13}C NMR), and JEOL ECX400 (400 MHz for ^{1}H NMR and 100 MHz for ^{13}C NMR). Chemical shifts were reported in ppm on the δ scale relative to residual $CHCl_3$ (δ = 7.26 for ^{1}H NMR and δ = 77.0 for ^{13}C NMR), CHD_2OD (δ = 3.31 for ^{1}H NMR and δ = 49.0 for ^{13}C NMR), or HDO (δ = 4.79 for ^{1}H NMR) as an internal reference. Infrared spectra (IR) were recorded on a JASCO FT/IR 410 Fourier transform infrared spectrophotometer. ESI-mass spectra were measured on a Waters ZQ4000 spectrometer (for LRMS) and a JEOL JMS-T100LC AccuTOF spectrometer (for HRMS). Preparative HPLC were conducted by using a JASCO HPLC system equipped with a UV-2075 spectrometer, PU-2086 pumps, a DG-2080-53 degasser, and an MX-2080-32 mixer. Reactions were carried out in dry solvents under argon atmosphere, unless otherwise stated. Reagents were purchased from Aldrich, Tokyo Chemical Industry Co., Ltd. (TCI), or Wako Pure Chemical Industries, Ltd., and used after purification by distillation or used without purification for solid substrates. Water for the HPLC analysis was purified using a Millipore MilliQ water purification system.

2. Copper-Catalyzed Stereodivergent Propargylation of Aldoses



2-1. General procedure for the stereodivergent propargylation of aldoses (Condition A)

A flame-dried 20-mL test tube was charged with mesitylcopper (0.5 mg, 0.0027 mmol), (*S,S,S*)-Ph-SKP (1.7 mg, 0.0026 mmol), and D-mannose **1a** (18 mg, 0.10 mmol) under argon atmosphere. $B(OMe)_3$ (22 µL, 0.20 mmol) and dry DMF (125 µL) were then added to this mixture. The mixture was stirred for 10 min at room temperature. Allenylboronate **2** (29 µL, 0.16 mmol) was added. After stirring for 16 h at room temperature, the reaction was quenched by the addition of MeOH and concentrated *in vacuo*. The process of MeOH addition followed by evaporation was repeated two-times to give a crude product. The diastereoselectivity was determined by ^{1}H NMR analysis.

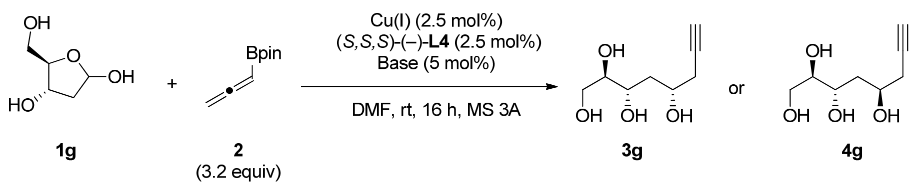
Products were purified by preparative reverse phase HPLC using a gradient of acetonitrile versus 0.1% TFA in water, affording **3a** as a white solid (19.8 mg, 90% yield). Preparative HPLC was carried out as follows: YMC-Triart C18 (20 mm I.D. × 250 mm) column using a linear gradient of 0-50% acetonitrile in 0.1% aqueous TFA over 30 min at room temperature with a flow rate of 7.0 mL min^{-1}.

The configurations of **3a** and **3j** were determined after converting to KDN (**6a**) and Neu5Ac (**6j**), respectively. The NMR data of synthesized KDN (**6a**) and Neu5Ac (**6j**) were identical to the reported ones (KDN: Nakamura, M.; Furuhata, K.; Yamasaki, T.; Ogura, H. *Chem. Pharm. Bull.* **1991**, *39* 3140., Neu5Ac: Lorpitthaya, R.; Suryawanshi, S. B.; Wang, S.; Pasunooti, K. K.; Cai, S.; Ma, J.; Liu, X.-W. *Angew. Chem. Int. Ed.* **2011**, *50*, 12054). The configurations of other products were tentatively assigned accordingly.

2-2. Optimization for the stereodivergent propargylation of 2-deoxy aldoses

The propargylation reaction between 2-deoxy-D-ribose (**1g**) and allenylboronate **2** was studied as a model reaction for 2-deoxy sugar substrates. Combinations of cationic copper salts and weak bases were examined to suppress protonolysis of allenylcopper species. A variety of mild bases, such as KOAc, PhCOONa, cesium pivalate, CF_3SO_3Na, and KOAc were examined, but the desired product was obtained only in trace amounts (Table S1, entries 2-6). Ultimately, CF_3COOK was identified as the optimum base, providing the product in 65% yield with an 18:1 diastereoselectivity (entry 7).

Table S1



Entry	Copper source	Base	Yield (%)[a]	3g : 4g[b]
1	MesCu	-	48	> 20 : 1
2	$CuClO_4(MeCN)_4$	KOAc	trace	-
3	$CuClO_4(MeCN)_4$	PhCOONa	trace	-
4	$CuClO_4(MeCN)_4$	Cesium pivalate	trace	-
5	$CuClO_4(MeCN)_4$	CF_3SO_3Na	trace	-
6	$CuClO_4(MeCN)_4$	KOAc	trace	-
7	$CuClO_4(MeCN)_4$	CF_3COOK	65	18 : 1

[a]Isolated yield. [b]Determined by ^{1}H NMR

2-3. General procedure for the stereodivergent propargylation of 2-deoxy aldoses (Condition B)

A flame-dried 20-mL test tube was charged with $CuClO_4(MeCN)_4$ (0.8 mg, 0.0025 mmol), (*S,S,S*)-Ph-SKP (1.7 mg, 0.0026 mmol), CF_3COOK (0.8 mg, 0.0053 mmol), MS 3A 40 mg and 2-deoxy-D-ribose (**1g**: 13.4 mg, 0.10 mmol) under argon atmosphere. $B(OMe)_3$ (22 μL, 0.20 mmol) and dry DMF (125 μL) were then added to this mixture. The mixture was stirred at room temperature for 10 min. Allenylboronate **2** (58 μL, 0.32 mmol) was added. After stirring for 16 h at room temperature, the reaction was quenched by the addition of MeOH and concentrated in *vacuo*. The process of MeOH addition followed by evaporation was repeated two-times to give a crude product. The diastereoselectivity was determined by ^1H NMR analysis. Products were purified by preparative reverse phase HPLC using a gradient of acetonitrile versus 0.1% TFA in water, affording **3g** as a white solid (11.3 mg, 65% yield). Preparative HPLC was carried out as follows: YMC-Triart C18 (20 mm I.D × 250 mm) column using a linear gradient of 0-50% acetonitrile in 0.1% aqueous TFA over 30 min at room temperature with a flow rate of 7.0 mL min^{-1}.

2-4. Gram-scale synthetic procedure for the stereodivergent propargylation of D-mannose

A flame-dried 20-mL bottle was charged with mesitylcopper (3.7 mg, 0.02 mmol), (*S,S,S*)-Ph-SKP (13.2 mg, 0.02 mmol), and D-Mannose **1a** (1.8 g, 10 mmol) under argon atmosphere. $B(OMe)_3$ (2.2 mL, 20 mmol) and dry DMF (6.3 mL) were then added to this mixture. The mixture was stirred for 10 min at room temperature. Allenylboronate **2** (2.7 mL, 15 mmol) was added. After stirring for 16 h at room temperature, the reaction was quenched by the addition of MeOH and concentrated *in vacuo*. Addition of MeOH-concentration process was repeated two-times to give a crude product. The crude solid was washed successively with EtOAc and MeOH to provide **3a** as a white solid (1.91 g, 87% yield).

2-5. Effect of $B(OMe)_3$

The ^1H NMR spectra in DMSO-d_6 of a sample containing D-mannose and 2 equiv of $B(OMe)_3$ (Figure S1) indicates the existence of complicated complexation between mannose and $B(OMe)_3$. The most notable difference between Figure S1 (mannose + 2 equiv of $B(OMe)_3$) and Figure S2 (mannose only) is the appearance of an aldehyde C-H proton (9.66 ppm) in Figure S1. The ratio of the aldehyde form to other species was determined to be 0.15% by ^1H NMR analysis using MeCN as internal standard. Thus, the addition of $B(OMe)_3$ significantly increased the aldehyde form. Although the concentration of the aldehyde form was still low, this observation indicates that the addition of $B(OMe)_3$ facilitates the propargylation reaction by stabilizing the aldehyde form of aldoses (see Fig. 2 in the text).

Figure S1. Mannose + 2 equiv of $B(OMe)_3$ + 1 equiv of MeCN

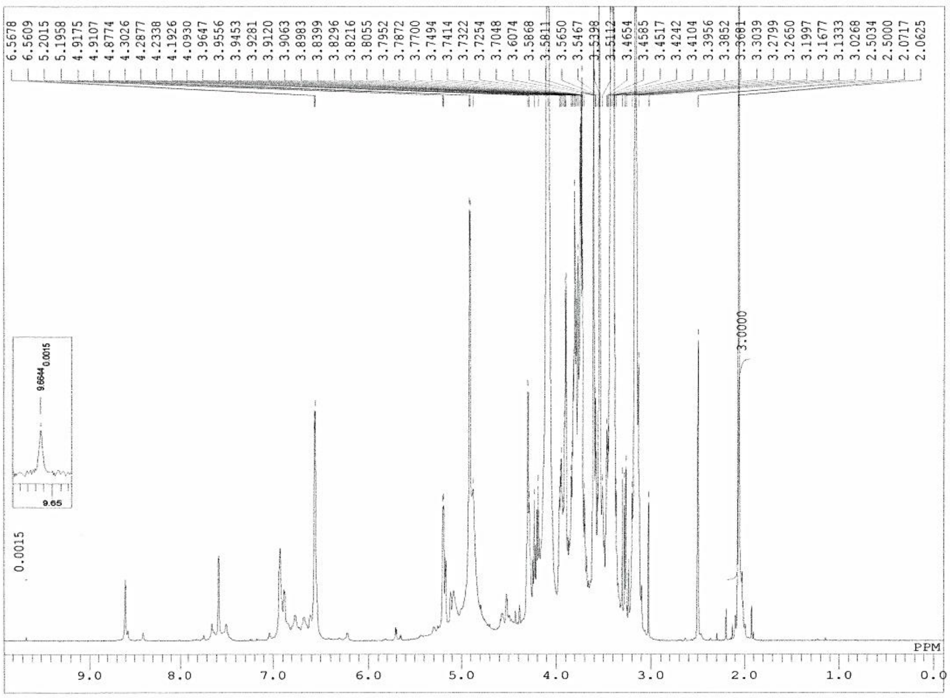
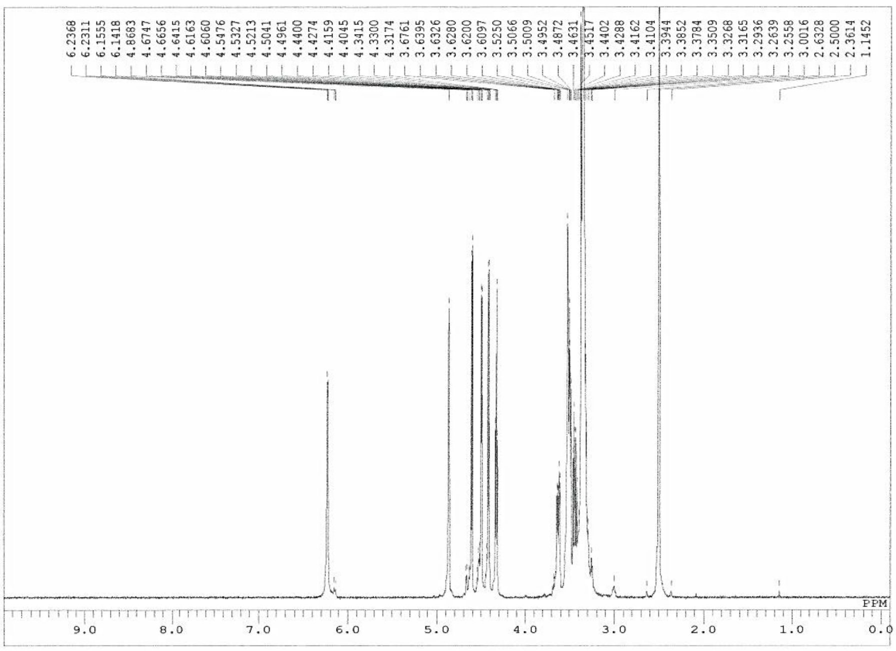
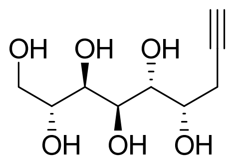


Figure S2. Mannose

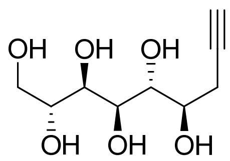


2-6. Characterization of propargylation products



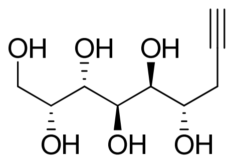
(2*R*,3*R*,4*R*,5*R*,6*S*)-non-8-yne-1,2,3,4,5,6-hexaol (3a)

A white solid, Yield: 90%. ^{1}H NMR (500 MHz, D_2O) δ 3.99 (t, *J* = 6.9 Hz, 1H), 3.79 (d, *J* = 9.6 Hz, 1H), 3.77-3.74 (m, 1H), 3.70 (d, *J* = 8.7 Hz, 1H), 3.67-3.63 (m, 1H), 3.60 (d, *J* = 9.6 Hz, 1H), 3.58-3.53 (m, 1H), 2.47-2.36 (m, 2H), 2.28 (t, *J* = 2.3 Hz, 1H); ^{13}C NMR (125 MHz, D_2O) δ 82.8, 71.8, 71.6, 70.1, 69.7, 68.5, 63.9, 23.6; IR (KBr): 3365, 3231, 1445, 1306, 1094, 1028, 849, 729 cm^{-1}; HRMS (ESI): m/z calcd for $C_9H_{16}O_6$ $[M+Na]^+$ 243.0840 Found 243.0842; $[\alpha]_D^{23.2}$ = +0.2 (*c* = 0.53, H_2O).



(2*R*,3*R*,4*R*,5*R*,6*R*)-non-8-yne-1,2,3,4,5,6-hexaol (4a)

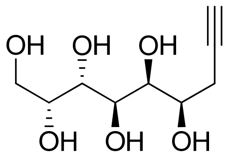
A white solid, Yield: 81%. ^{1}H NMR (500 MHz, D_2O) δ 3.88 (dt, *J* = 8.6, 4.6 Hz, 1H), 3.74-3.57 (m, 5H), 3.50 (dd, *J* = 11.7, 6.0 Hz, 1H), 2.42 (dt, *J* = 17.2, 3.5 Hz, 1H), 2.34 (ddd, *J* = 17.2, 8.0, 2.6 Hz, 1H), 2.22 (t, *J* = 2.6 Hz, 1H); ^{13}C NMR (125 MHz, D_2O) δ 82.7, 72.8, 71.5, 71.4, 71.2, 70.5, 70.2, 63.9, 21.7; IR (KBr): 3375, 2962, 2896, 1423, 1392, 1088, 1035, 752, 634 cm^{-1}; HRMS (ESI): m/z calcd for $C_9H_{16}O_6$ $[M+Na]^+$ 243.0840 Found 243.0835; $[\alpha]_D^{22.2}$ = +6.4 (*c* = 0.50, H_2O).



(2*R*,3*S*,4*R*,5*S*,6*S*)-non-8-yne-1,2,3,4,5,6-hexaol (3b)

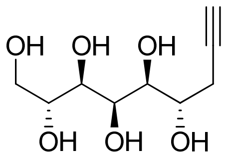
A white solid, Yield: 73%. ^{1}H NMR (500 MHz, D_2O) δ 3.83 (t, *J* = 6.5 Hz, 1H), 3.77 (d, *J* = 9.5 Hz, 1H), 3.75-3.69 (m, 2H), 3.53 (d, *J* = 6.3 Hz, 2H), 3.51 (d, *J* = 10.3 Hz, 1H), 2.52 (dt, *J* = 17.4, 2.5 Hz, 1H), 2.37 (ddd, *J* = 17.4, 5.4, 2.5 Hz, 1H), 2.22 (t, *J* = 2.5 Hz, 1H); ^{13}C NMR (125 MHz, CD_3OD) δ 75.1, 72.0, 71.3, 69.7, 68.0, 64.9, 40.9, 28.7; IR

(KBr): 3297, 1422, 1112, 1086, 1033 cm^{-1}; HRMS (ESI): m/z calcd for $C_9H_{16}O_6$ $[M+Na]^+$ 243.0840 Found 243.0842; $[\alpha]_D^{22.4}$ = +4.4 (*c* = 0.50, H_2O).



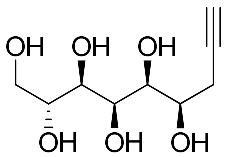
(2*R*,3*S*,4*R*,5*S*,6*R*)-non-8-yne-1,2,3,4,5,6-hexaol (4b)

A white solid, Yield: 66%. ^{1}H NMR (500 MHz, D_2O) δ 3.80-3.73 (m, 3H), 3.60 (d, *J* = 9.2 Hz, 1H), 3.54-3.49 (m, 3H), 2.44-2.40 (m, 1H), 2.34-2.28 (m, 1H), 2.22 (brs, 1H); ^{13}C NMR (125 MHz, CD_3OD) δ 81.9, 73.6, 73.2, 71.7, 71.4, 71.2, 64.9, 24.3; IR (KBr): 3398, 2925, 1433, 1103, 1055, 680 cm^{-1}; HRMS (ESI): m/z calcd for $C_9H_{16}O_6$ $[M+Na]^+$ 243.0840 Found 243.0832; $[\alpha]_D^{22.1}$ = +6.1 (*c* = 0.35, H_2O).



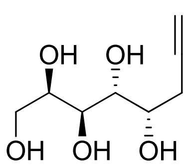
(2*R*,3*R*,4*R*,5*S*,6*S*)-non-8-yne-1,2,3,4,5,6-hexaol (3c)

A white solid, Yield: 76%. ^{1}H NMR (500 MHz, D_2O) δ 3.86 (t, *J* = 2.9 Hz, 1H), 3.74-3.70 (m, 1H), 3.66-3.57 (m, 4H), 3.49 (dd, *J* = 11.4, 5.9 Hz, 1H), 2.45 (dt, *J* = 17.3, 3.4 Hz, 1H), 2.36 (ddd, *J* = 17.3, 6.3, 2.3 Hz, 1H), 2.21 (t, *J* = 2.3 Hz, 1H); ^{13}C NMR (125 MHz, D_2O) δ 81.9, 74.6, 73.6, 71.8, 71.7, 69.2, 68.8, 63.1, 23.0; IR (KBr): 3280, 2918, 1427, 1096, 1028, 667 cm^{-1}; HRMS (ESI): m/z calcd for $C_9H_{16}O_6$ $[M+Na]^+$ 243.0840 Found 243.0835; $[\alpha]_D^{22.9}$ = +2.0 (*c* = 0.51, H_2O).



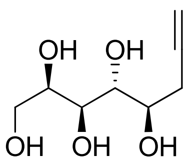
(2*R*,3*R*,4*R*,5*S*,6*R*)-non-8-yne-1,2,3,4,5,6-hexaol (4c)

A white solid, Yield: 72%. (inseparable mixture of **3c** and **4c**) For **4c**: ^{1}H NMR (500 MHz, D_2O) δ 3.95 (dd, *J* = 5.7, 1.7 Hz, 1H), 3.89 (td, *J* = 6.9, 2.9 Hz, 1H), 3.80-3.60 (m, 5H), 2.52 (dd, *J* = 6.9, 2.9 Hz, 1H), 2.27 (t, *J* = 2.9 Hz, 1H); ^{13}C NMR (125 MHz, D_2O) δ 82.0, 74.7, 73.9, 71.8, 71.7, 70.6, 70.0, 63.5, 23.7; IR (KBr): 3387, 2925, 1675, 1204, 1076 cm^{-1}; HRMS (ESI): m/z calcd for $C_9H_{16}O_6$ $[M+Na]^+$ 243.0840 Found 243.0832.



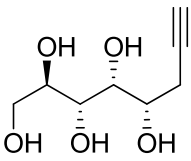
(2*R*,3*S*,4*R*,5*S*)-oct-7-yne-1,2,3,4,5-pentaol (3d)

A white solid, Yield: 84%. ^{1}H NMR (500 MHz, D_2O) δ 4.00 (t, *J* = 7.1 Hz, 1H), 4.89 (t, *J* = 6.4 Hz, 1H), 3.65-3.56 (m, 4H), 2.47 (ddd, *J* = 16.9, 7.5, 2.6 Hz, 1H), 2.40 (ddd, *J* = 16.9, 6.7, 2.6 Hz, 1H), 2.32 (t, *J* = 2.6 Hz, 1H); ^{13}C NMR (125 MHz, D_2O) δ 82.6, 71.6, 71.2, 71.0, 70.2, 69.1, 63.9, 23.7; IR (KBr): 3388, 3217, 1452, 1389, 1294, 1231, 1105, 1052, 735, 657 cm^{-1}; HRMS (ESI): m/z calcd for $C_8H_{14}O_5$ $[M+Na]^+$ 213.0734 Found 213.0737; $[\alpha]_D^{22.8}$ = +4.3 (*c* = 0.15, MeOH).



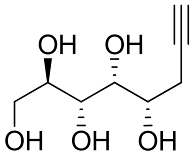
(2*R*,3*S*,4*R*,5*R*)-oct-7-yne-1,2,3,4,5-pentaol (4d)

A white solid, Yield: 81%. ^{1}H NMR (500 MHz, D_2O) δ 3.87 (dt, *J* = 8.6, 4.5 Hz, 1H), 3.80-3.77 (m, 1H), 3.66 (dd, *J* = 8.1, 5.1 Hz, 1H), 3.53-3.48 (m, 3H), 2.41 (dt, *J* = 17.2, 2.9 Hz, 1H), 2.33 (ddd, *J* = 17.2, 7.9, 2.9 Hz, 1H), 2.21 (t, *J* = 2.9 Hz, 1H); ^{13}C NMR (125 MHz, D_2O) δ 82.6, 72.9, 71.6, 71.5, 71.2, 71.0, 63.7, 21.8; IR (KBr): 3326, 2952, 2900, 1458, 1411, 1222, 1095, 1048, 1030, 860, 695, 654 cm^{-1}; HRMS (ESI): m/z calcd for $C_8H_{14}O_5$ $[M+Na]^+$ 213.0734 Found 213.0731 ; $[\alpha]_D^{20.8}$ = +9.4 (*c* = 0.86, H_2O).



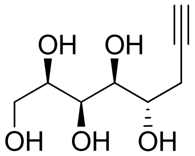
(2*R*,3*S*,4*R*,5*S*)-oct-7-yne-1,2,3,4,5-pentaol (3e)

A white solid, Yield: 95%.^{1}H NMR (500 MHz, D_2O) δ 3.76 (dd, *J* = 11.2, 6.2 Hz, 1H), 3.71 (dd, *J* = 6.2, 2.0 Hz, 1H), 3.67 (dd, *J* = 11.8, 2.9 Hz, 1H), 3.63-3.60 (m, 1H), 3.51-3.47 (m, 2H), 2.40 (ddd, *J* = 17.4, 4.8, 2.3 Hz, 1H), 2.30 (ddd, *J* = 17.4, 6.4, 2.3 Hz, 1H), 2.22 (t, *J* = 2.3 Hz, 1H); ^{13}C NMR (125 MHz, D_2O) δ 81.7, 72.0, 71.9, 71.8, 71.7, 71.2, 63.5, 23.4; IR (KBr): 3430, 3285, 1434, 1089, 1042cm^{-1}; HRMS (ESI): m/z calcd for $C_8H_{14}O_5$ $[M+Na]^+$ 213.0734 Found 213.0737 ; $[\alpha]_D^{21.5}$ = -0.4 (*c* = 0.52, H_2O).



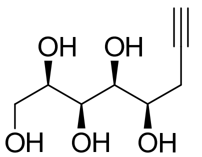
(2*R*,3*S*,4*R*,5*R*)-oct-7-yne-1,2,3,4,5-pentaol (4e)

A white solid, Yield: 93%. ^{1}H NMR (500 MHz, D_2O) δ 3.72-3.64 (m, 4H), 3.60 (ddd, *J* = 8.8, 6.3, 2.8 Hz, 1H), 3.51 (dd, *J* = 11.9, 6.3 Hz, 1H), 2.51 (dt, *J* = 17.3, 2.6 Hz, 1H), 2.37 (ddd, *J* = 17.3, 5.6, 2.6 Hz, 1H), 2.22 (t, *J* = 2.6 Hz, 1H); ^{13}C NMR (125 MHz, D_2O) δ 82.2, 71.9, 71.9, 71.5, 69.8, 68.9, 63.9, 23.9; IR (KBr): 3305, 2949, 1286, 1082, 1041, 644 cm^{-1}; HRMS (ESI): m/z calcd for $C_8H_{14}O_5$ $[M+Na]^+$ 213.0734 Found 213.0737; $[\alpha]_D^{22.7}$ = -4.8 (*c* = 0.48, H_2O).



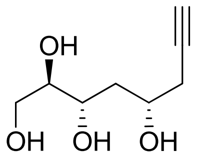
(2*R*,3*S*,4*S*,5*S*)-oct-7-yne-1,2,3,4,5-pentaol (3f)

A white solid, Yield: 65%. ^{1}H NMR (400 MHz, D_2O) δ 3.75-3.64 (m, 3H), 3.60-3.57 (m, 1H), 3.52-3.43 (m, 2H), 2.49 (dt, *J* = 17.3, 2.3 Hz, 1H), 2.36 (ddd, *J* = 17.3, 6.0, 2.3 Hz, 1H), 2.22 (t, *J* = 2.3 Hz, 1H); ^{13}C NMR (100 MHz, D_2O) δ 82.0, 73.7, 73.0, 72.0, 70.2, 69.0, 63.0, 23.4; IR (KBr): 3389, 2934, 1421, 1067, 657 cm^{-1}; HRMS (ESI): m/z calcd for $C_8H_{14}O_5$ $[M+Na]^+$ 213.0734 Found 213.0741; $[\alpha]_D^{22.6}$ = +6.4 (*c* = 0.91, H_2O).



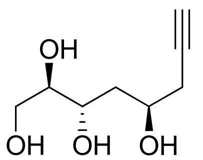
(2*R*,3*S*,4*S*,5*R*)-oct-7-yne-1,2,3,4,5-pentaol (4f)

A white solid, Yield: 60%. ^{1}H NMR (500 MHz, D_2O) δ 3.81-3.79 (m, 1H), 3.70-3.67 (m, 1H), 3.61-3.55 (m, 3H), 3.51-3.47 (dd, *J* = 11.5, 6.9 Hz, 1H), 2.41-2.32 (m, 2H), 2.24 (m, 1H); ^{13}C NMR (125 MHz, D_2O) δ 81.9, 73.2, 72.5, 71.9, 71.8, 70.4, 63.4, 23.5; IR (KBr): 3388, 1420, 1067, 669 cm^{-1}; HRMS (ESI): m/z calcd for $C_8H_{14}O_5$ $[M+Na]^+$ 213.0727 Found 213.0734; $[\alpha]_D^{22.8}$ = +2.9 (*c* = 0.68, H_2O).



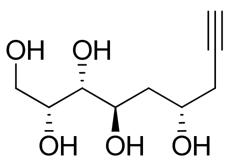
(2*R*,3*S*,5*S*)-oct-7-yne-1,2,3,5-tetraol (3g)

A white solid, Yield: 65%. ^{1}H NMR (500 MHz, CD_3OD) δ 3.97 (dq, *J* = 8.1, 5.7 Hz, 1H), 3.74-3.67 (m, 2H), 3.56 (dd, *J* = 11.3, 6.5 Hz, 1H), 3.46 (dt, *J* = 6.3, 2.9 Hz, 1H), 2.41-2.30 (m, 2H), 2.28 (t, *J* = 2.9 Hz, 1H), 1.99 (ddd, *J* = 14.3, 4.6, 2.9 Hz, 1H), 1.66-1.60 (dt, *J* = 14.3, 9.2 H, 1H); ^{13}C NMR (125 MHz, CD_3OD) δ 81.7, 76.3, 72.4, 71.4, 70.0, 64.4, 39.5, 27.8; IR (KBr): 3376, 2923, 1677, 1424, 1204, 1071, 651 cm^{-1}; HRMS (ESI): m/z calcd for $C_8H_{14}O_4$ [M+Na]$^+$ 197.0785 Found 197.0781; $[\alpha]_D^{22.9}$ = -1.6 (*c* = 0.57, MeOH).



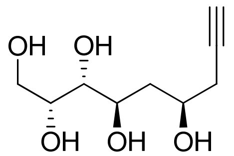
(2*R*,3*S*,5*R*)-oct-7-yne-1,2,3,5-tetraol (4g)

A white solid, Yield: 53%. ^{1}H NMR (500 MHz, CD_3OD) δ 3.98 (dddd, *J* = 9.2, 6.3, 2.9 Hz, 1H), 3.786 (ddd, *J* = 9.8, 6.3, 2.4 Hz, 1H), 3.71 (dd, *J* = 11.2, 3.9 Hz, 1H), 3.56 (dd, *J* = 11.2, 6.6 Hz, 1H), 3.48 (dt, *J* = 6.6, 3.9 Hz, 1H), 2.40 – 2.31 (m, 2H), 2.27 (t, *J* = 2.8 Hz, 1H), 1.77 (ddd, *J* = 14.4, 9.8, 2.7 Hz, 1H), 1.69 (ddd, *J* = 14.4, 9.8, 2.7 Hz, 1H); ^{13}C NMR (125 MHz, CD_3OD) δ 81.9, 76.6, 71.3, 70.2, 67.8, 64.7, 40.2, 28.7; IR (KBr): 3375, 2921, 1420, 1064, 652 cm^{-1}; HRMS (ESI): m/z calcd for $C_8H_{14}O_4$ [M+Na]$^+$ 197.0785 Found 197.0781; $[\alpha]_D^{20.6}$ = -28.2 (*c* = 0.64, H_2O).



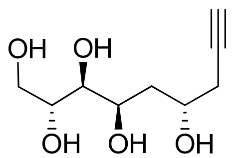
(2*R*,3*R*,4*R*,6*S*)-non-8-yne-1,2,3,4,6-pentaol (3h)

A white solid, Yield: 74%. ^{1}H NMR (500 MHz, CD_3OD) δ 4.03-3.97 (m, 1H), 3.87 (ddd, *J* = 7.6, 6.7, 2.2 Hz, 2H), 3.64-3.58 (m, 2H), 3.36 (dd, *J* = 7.6, 2.2 Hz, 1H), 2.41-2.31 (m, 2H), 2.27 (t, *J* = 2.7 Hz, 1H), 1.88 (ddd, *J* = 14.3, 9.8, 2.4 Hz, 1H), 1.69 (ddd, *J* = 14.3, 9.8, 2.4 Hz, 1H); ^{13}C NMR (100 MHz, D_2O) δ 82.5, 74.4, 71.9, 71.2, 68.3, 66.8, 63.7, 39.2, 27.6; IR (KBr): 3280, 3192, 2952, 1466, 1422, 1065, 1030, 707 cm^{-1}; HRMS (ESI): m/z calcd for $C_9H_{16}O_5$ $[M+Na]^+$ 227.0890 Found 227.0887; $[\alpha]_D^{22.8}$ = +23.8 (c = 0.49, H_2O).



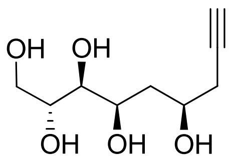
(2*R*,3*R*,4*R*,6*R*)-non-8-yne-1,2,3,4,6-pentaol (4h)

A white solid, Yield: 67%. ^{1}H NMR (500 MHz, CD_3OD) δ 4.00 (dq, J = 8.0, 5.6 Hz, 1H), 3.87-3.81 (m, 2H), 3.61 (d, J = 6.6 Hz, 2H), 3.37 (dd, J = 7.5, 2.1 Hz, 1H), 2.42-2.31 (m, 2H), 2.28 (t, J = 2.6 Hz, 1H), 2.02 (ddd, J = 14.2, 4.7, 3.0 Hz, 1H), 1.55 (ddd, J = 14.2, 9.1, 8.1 Hz, 1H); ^{13}C NMR (125 MHz, CD_3OD) δ 81.7,84.6.9, 71.8, 71.7, 70.1, 64.8, 40.2, 27.9; IR (neat): 3375, 1422, 1069 cm^{-1}; HRMS (ESI): m/z calcd for $C_9H_{16}O_5$ $[M+Na]^+$ 227.0890 Found 227.0882, $[\alpha]_D^{21.5}$ = +9.4 (c = 0.53, H_2O).



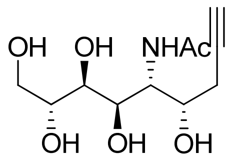
(2*R*,3*S*,4*R*,6*S*)-non-8-yne-1,2,3,4,6-pentaol (3i)

A white solid, Yield: 59%. ^{1}H NMR (400 MHz, CD_3OD) δ 4.13-4.09 (m, 1H), 4.00-3.91 (m, 1H), 3.78 (dd, J = 10.9, 3.5 Hz, 1H), 3.67 (ddd, J = 8.0, 5.9, 3.5 Hz, 1H), 3.60 (dd, J = 10.9, 5.9 Hz, 1H), 3.33 (s, 1H), 2.36 (dd, J = 6.2, 2.7 Hz, 2H), 2.26 (t, J = 2.7 Hz, 1H), 1.94 (ddd, J = 14.3, 10.4, 2.6 Hz, 1H), 1.53 (ddd, J = 14.3, 9.8, 2.6 Hz, 1H); ^{13}C NMR (125 MHz, CD_3OD) δ 82.0, 75.5, 73.2, 71.3, 68.3, 68.0, 65.1, 41.2, 28.6; IR (neat): 3290, 1420, 1092, 1074, 1025 cm^{-1}; HRMS (ESI): m/z calcd for $C_9H_{16}O_5$ $[M+Na]^+$ 227.0890 Found 227.0900; $[\alpha]_D^{23.8}$ = +31.9 (c = 0.28, MeOH).



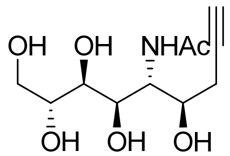
(2*R*,3*S*,4*R*,6*R*)-non-8-yne-1,2,3,4,6-pentaol (4i)

A white solid, Yield: 52%. ^{1}H NMR (500 MHz, CD_3OD) δ 4.06 (dt, J = 7.4, 1.8 Hz, 1H), 3.94 (dq, J = 11.0, 5.8 Hz, 1H), 3.78 (dd, J = 11.0, 3.5 Hz, 1H), 3.67 (ddd, J = 8.1, 6.0, 3.5 Hz, 1H), 3.60 (dd, J = 8.1, 5.7 Hz, 1H), 3.36 (dd, J = 10.4, 5.2 Hz, 1H), 2.42-2.33 (m, 2H), 2.28 (t, *J* = 2.6 Hz, 1H), 1.85-1.82 (m, 2H); ^{13}C NMR (125 MHz, CD_3OD) δ 81.7, 74.5, 73.0, 71.4, 69.9, 69.5, 65.1, 40.3, 27.9; IR (KBr): 3280, 2918, 1432, 1089, 1033, 638 cm^{-1}; HRMS (ESI): m/z calcd for $C_9H_{16}O_5$ $[M+Na]^+$ 227.0890 Found 227.0900; $[\alpha]_D^{22.4}$ = +6.5 (*c* = 0.26, H_2O).



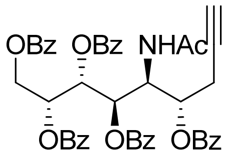
N-((4*S*,5*R*,6*R*,7*S*,8*R*)-4,6,7,8,9-pentahydroxynon-1-yn-5-yl)acetamide (3j)

A white solid, Yield: 70%. ^{1}H NMR (500 MHz, D_2O) δ 4.13 (t, *J* = 6.9 Hz, 1H), 3.94 (d, *J* = 10.4 Hz, 1H), 3.78 (d, *J* = 10.4 Hz, 1H), 3.68 (dd, *J* = 12.0, 2.9 Hz, 1H), 3.61-3.57 (m, 1H), 3.47 (dd, *J* = 12.0, 6.3 Hz, 1H), 3.31 (d, *J* = 9.2 Hz, 1H), 2.26-2.22 (m, 3H), 1.89 (s, 3H); ^{13}C NMR (125 MHz, CD_3OD) δ 174.8, 81.8, 72.4, 71.4, 71.2, 69.7, 68.7, 65.2, 54.7, 25.3, 22.6; IR (KBr): 3499, 3362, 1623, 1541, 1074 cm^{-1}; HRMS (ESI): m/z calcd for $C_{11}H_{19}NO_6$ $[M+Na]^+$ 284.1105 Found 284.1106; $[\alpha]_D^{22.8}$ = -28.9 (*c* = 0.67, H_2O).



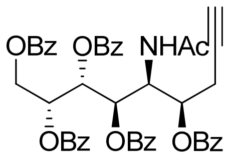
N-((4*R*,5*R*,6*R*,7*S*,8*R*)-4,6,7,8,9-pentahydroxynon-1-yn-5-yl)acetamid (4j)

A white solid, Yield: 51%. ^{1}H NMR (500 MHz, D_2O) δ 4.10 (dd, *J* = 8.9, 5.7 Hz, 1H), 3.98 (ddd, *J* = 7.7, 5.7, 4.3 Hz, 1H), 3.84-3.82 (m, 1H), 3.67 (dd, *J* = 11.9, 2.8 Hz, 1H), 3.57 (ddd, *J* = 9.1, 6.3, 2.8 Hz, 1H), 3.46 (dd, *J* = 11.9, 6.3 Hz, 1H), 3.38 (dd, *J* = 9.1, 0.8 Hz, 1H), 2.41 (ddd, *J* = 17.0, 4.1, 2.6 Hz, 1H), 2.30 (ddd, *J* = 17.0, 7.7, 2.6 Hz, 1H), 2.24 (t, *J* = 2.6 Hz, 1H), 1.87 (s, 3H); ^{13}C NMR (125 MHz, CD_3OD) δ 174.9, 82.1, 71.7, 71.1, 70.2, 69.4, 63.7, 54.3, 22.7, 22.6; IR (neat): 3293, 1639, 1547, 1424, 1378, 1203, 1078, 1031 cm^{-1}; HRMS (ESI): m/z calcd for $C_{11}H_{19}NO_6$ $[M+Na]^+$ 284.1105 Found 284.1118; $[\alpha]_D^{23.3}$ = -4.4 (*c* = 0.84, MeOH).



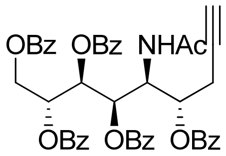
(2*R*,3*R*,4*R*,5*S*,6*S*)-5-acetamidonon-8-yne-1,2,3,4,6-pentayl pentabenzoate (3k-Bz)

A white solid, Yield: 55%. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.10-8.09 (m, 2H), 7.95-7.93 (m, 2H), 7.86-7.84 (m, 4H), 7.70-7.69 (m, 2H), 7.68 (t, *J* = 7.5 Hz, 1H), 7.50-7.43 (m, 5H), 7.37 (t, *J* = 7.5 Hz, 1H), 7.33-7.26 (m, 7H), 7.10 (t, *J* = 7.5 Hz, 2H), 6.12 (d, *J* = 6.9 Hz, 1H), 6.05 (d, *J* = 9.8 Hz, 1H), 5.86 (dd, *J* = 7.3, 2.0 Hz, 2H), 5.20 (dd, *J* = 13.0, 6.3 Hz, 1H), 5.11 (dd, *J* = 8.2, 6.3 Hz, 1H), 4.67 (dd, *J* = 11.9, 4.4 Hz, 1H), 4.48 (dd, *J* = 11.9, 6.9 Hz, 1H), 2.75 (ddd, *J* = 17.0, 7.1, 2.6 Hz, 1H), 2.68 (ddd, *J* = 17.0, 6.0, 2.6 Hz, 1H), 2.01 (s, 3H), 1.93 (t, *J* = 2.6 Hz, 1H); ^{13}C NMR (125 MHz, acetone-d6) δ170.4, 166.3, 166.1, 165.9, 165.8, 134.3, 134.2, 134.0, 133.9, 133.7, 131.0, 130.9, 130.9, 130.8, 130.7, 130.6, 130.5, 130.3, 130.2, 129.4, 129.3, 129.2, 129.1, 129.0, 79.8, 72.5, 72.0, 71.9, 71.0, 70.4, 69.5, 64.3, 50.5, 50.4, 22.9, 21.9; IR (neat): 3390, 1721, 1683, 1259, 1092, 1067, 708 cm^{-1}; HRMS (ESI): m/z calcd for $C_{46}H_{39}NO_{11}$ $[M+Na]^+$ 804.2416 Found 804.2399; $[\alpha]_D^{22.9}$ = -13.9 (*c* = 0.65, MeOH).

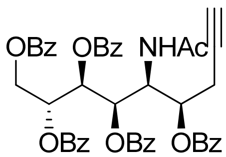


(2*R*,3*R*,4*R*,5*S*,6*R*)-5-acetamidonon-8-yne-1,2,3,4,6-pentayl pentabenzoate (4k-Bz)

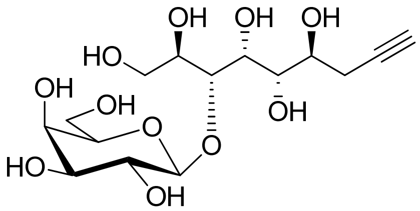
A white solid, Yield: 40%. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.10 (d, *J* = 7.4 Hz, 2H), 7.95 (d, *J* = 7.4 Hz, 2H), 7.86-7.80 (m, 6H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.49 (dd, *J* = 13.1, 7.4 Hz, 2H), 7.42 (dd, *J* = 10.8, 4.4 Hz, 4H), 7.32-7.24 (m, 8H), 5.93-5.89 (m, 4H), 5.26 (dd, *J* = 11.3, 5.6 Hz, 1H), 5.16 (dd, *J* = 9.8, 6.3 Hz, 1H), 4.73 (dd, *J* = 11.9, 4.3 Hz, 1H), 4.48 (dd, *J* = 11.9, 7.4 Hz, 1H), 2.75 (ddd, *J* = 17.3, 5.0, 2.6 Hz, 1H), 2.64 (ddd, *J* = 17.3, 5.7, 2.6 Hz, 1H), 1.92 (t, *J* = 2.6 Hz, 1H), 1.90 (s, 3H); ^{13}C NMR (125 MHz, acetone-d6) δ170.9, 166.3, 166.1, 166.0, 165.9, 134.2, 134.1, 134.0, 133.9, 133.8, 130.9, 130.8, 130.6, 130.5, 130.4, 130.3, 130.2, 130.1, 129.3, 129.2, 129.1, 129.0, 79.6, 73.2, 72.8, 71.4, 70.9, 70.2, 64.5, 50.4, 22.8, 22.3; IR (neat): 3376, 1719, 1683, 1246, 1092, 1067, 708 cm^{-1}; HRMS (ESI): m/z calcd for $C_{46}H_{39}NO_{11}$ $[M+Na]^+$ 804.2416 Found 804.2399; $[\alpha]_D^{22.8}$ = -17.4 (*c* = 0.45, MeOH).



(2*R*,3*S*,4*R*,5*S*,6*S*)-5-acetamidonon-8-yne-1,2,3,4,6-pentayl pentabenzoate (3l-Bz)
A white solid, Yield: 45%. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.05-7.99 (m, 4H), 7.847.82 (m, 2H), 7.73 (dd, *J* = 8.1, 7.5 Hz, 4H), 7.54 (dt, *J* = 7.5, 1.0 Hz, 2H), 7.45-7.35 (m, 8H), 7.26-7.17 (m, 5H), 5.99-5.92 (m, 2H), 5.85 (dt, *J* = 5.9, 3.0 Hz, 1H), 5.23 (dd, *J* = 13.2, 6.3 Hz, 1H), 5.16 (dd, *J* = 7.3, 1.8 Hz, 1H), 4.87 (dd, *J* = 12.3, 3.0 Hz, 1H), 4.59 (dd, *J* = 12.3, 5.9 Hz, 1H), 2.72 (ddd, *J* = 17.2, 6.4, 2.7 Hz, 1H), 2.65 (ddd, *J* = 17.2, 5.9, 2.7 Hz, 1H), 2.11 (s, 3H), 1.86 (t, *J* = 2.7 Hz, 1H); ^{13}C NMR (125 MHz, acetone-d6) δ171.1, 166.5, 166.1, 165.8, 165.7, 134.2, 134.1, 134.1, 134.0, 133.9, 130.8, 130.7, 130.6,130.6, 130.6, 130.5, 130.4, 130.4, 130.2, 129.4, 129.4, 129.3, 129.2, 129.2, 129.0, 79.8, 72.4, 72.0, 71.3, 71.2, 70.4, 63.2, , 51.9, 51.8, 23.0, 22.2; IR (neat): 3418, 1717, 1653, 1261, 1093, 709 cm^{-1}; HRMS (ESI): m/z calcd for $C_{46}H_{39}NO_{11}$ $[M+Na]^{+}$ 804.2416 Found 804.2399; $[\alpha]_D^{22.8}$ = +12.6 (*c* = 0.44, MeOH).

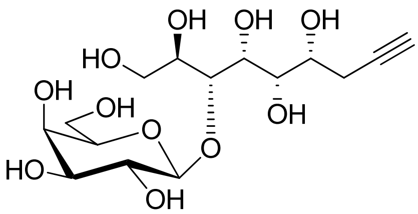


(2*R*,3*S*,4*R*,5*S*,6*R*)-5-acetamidonon-8-yne-1,2,3,4,6-pentayl pentabenzoate (4l-Bz)
A white solid, Yield: 51%. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.11-8.07 (m, 2H), 8.07-8.03 (m, 2H), 8.03-7.99 (m, 2H), 7.92-7.88 (m, 2H), 7.84 (d, *J* = 8.2 Hz, 2H), 7.58 (dd, *J* = 15.3, 7.5 Hz, 2H), 7.53 (dd, *J* = 13.0, 6.9 Hz, 2H), 7.44-7.33 (m, 10H), 7.19 (t, *J* = 7.7 Hz, 2H), 6.33 (dd, *J* = 8.2, 3.1 Hz, 1H), 5.81-5.72 (m, 4H), 5.01-4.95 (m, 1H), 4.79 (dd, *J* = 12.4, 2.7 Hz, 1H), 4.49 (dd, *J* = 12.4, 5.0 Hz, 1H), 2.67-2.56 (m, 2H), 1.78 (s, 3H), 1.70 (t, *J* = 2.1 Hz, 1H); ^{13}C NMR (125 MHz, acetone-d6) δ 170.9, 166.5, 166.5, 166.4, 166.3, 134.5, 134.2, 134.2, 134.0, 133.9, 130.8, 130.8, 130.7, 130.6, 130.6, 130.5, 130.3, 130.3, 130.2, 129.5, 129.3, 129.2, 129.2, 100.8, 79.7, 72.3, 72.1, 71.8, 70.6, 63.1, 51.5, 22.7, 22.5; IR (neat): 3384, 1711, 1674, 1241, 1090, 1066, 1024, 706 cm^{-1}; HRMS (ESI): m/z calcd for $C_{46}H_{39}NO_{11}$ $[M+Na]^{+}$ 804.2416 Found 804.2399; $[\alpha]_D^{23.1}$ =+19.8 (*c* = 1.17, MeOH).



(2*R*,3*R*,4*R*,5*S*,6*S*)-3-(((2*S*,3*R*,4*S*,5*R*,6*R*)-3,4,5-trihydroxy-6-(hydroxymethyl)tetrahydro-2*H*-pyran-2-yl)oxy)non-8-yne-1,2,4,5,6-pentaol (3m)

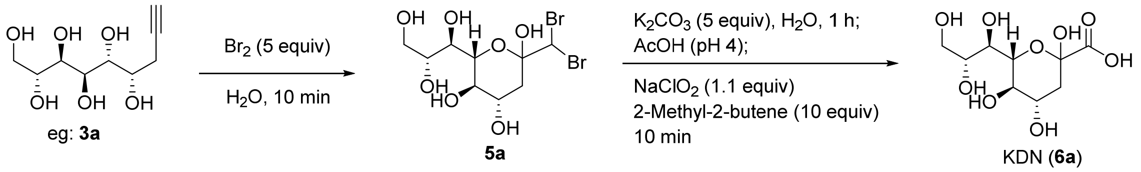
A white solid, Yield: 56%. [1]H NMR (500 MHz, D_2O) δ 4.39 (d, *J* = 8.0 Hz, 1H), 3.93 (s, 1H), 3.78-3.38 (m, 12H), 2.45 (dt, *J* = 17.3, 2.5 Hz, 1H), 2.35 (ddd, *J* = 17.3, 6.1, 2.5 Hz, 1H), 2.23 (t, *J* = 2.5 Hz, 1H); [13]C NMR (125 MHz, D_2O) δ 104.1, 82.2, 82.1, 75.9, 73.7, 73.2, 72.1, 71.9, 71.8, 69.3, 69.1, 68.9, 62.7, 61.7, 23.1; IR (neat): 3398, 1642, 1424, 1074 cm^{-1}; HRMS (ESI): m/z calcd for $C_{15}H_{26}O_{11}$ $[M+Na]^+$ 405.1368 Found 405.1367; $[\alpha]_D^{23.0}$ = +7.4 (*c* = 0.87, H_2O).



(2*R*,3*R*,4*R*,5*S*,6*R*)-3-(((2*S*,3*R*,4*S*,5*R*,6*R*)-3,4,5-trihydroxy-6-(hydroxymethyl)tetrahydro-2*H*-pyran-2-yl)oxy)non-8-yne-1,2,4,5,6-pentaol (4m)

A white solid, Yield: 45%. [1]H NMR (500 MHz, D_2O) δ 4.34 (d, *J* = 7.5 Hz 1H), 3.90-3.87 (m, 1H), 3.81-3.47 (m, 11H), 3.38-3.35 (m, 1H), 2.38 (ddd, *J* = 9.4, 7.4, 2.5 Hz, 1H), 2.33-2.26 (m, 1H), 2.23 (t, *J* = 2.0 Hz, 1H). [13]C NMR (125 MHz, D_2O) δ 103.7, 82.5, 79.8, 75.6, 73.5, 73.2, 71.9, 71.8, 71.7, 70.6, 70.0, 69.2, 62.7, 61.5, 23.8; IR (neat): 3409, 1643, 1423, 1075 cm^{-1}; HRMS (ESI): m/z calcd for $C_{15}H_{26}O_{11}$ $[M+Na]^+$ 405.1368 Found 405.1367; $[\alpha]_D^{23.1}$ = +6.7 (*c* = 1.09, H_2O).

3. Rapid Synthesis of Sialic Acid Derivatives



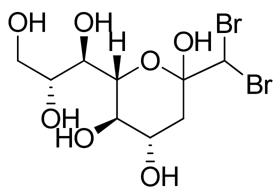
3-1. General procedure for sialic acid synthesis

3-Deoxy-D-glycero-β-D-galacto-2-nonulosonic acid (6a)

To a 100 mL round bottom flask containing compound **3a** (1.1 g, 5 mmol) in 22.5 mL H_2O was added Br_2 (2.0 g, 25 mmol). The resulting reaction mixture was stirred for 10 min at room temperature to afford 5**a**. Excess amounts of bromine were removed *via* extraction with hexane (30 mL, 3 times). The aqueous solution containing the product was used directly into the next step without purification.

To a solution of **5a** in H_2O (22.5 mL) was added K_2CO_3 (3.46 g, 25 mmol). The mixture was stirred for 1 h at room temperature until TLC analysis indicated completion of the reaction. Subsequently, CH_3COOH was added to the reaction mixture until pH = 4. To the mixture were added *t*BuOH (22.5 mL) and 2-methyl-2-butene (5.3 mL, 50 mmol). $NaClO_2$ (497 mg, 5.5 mmol) dissolved in 5 mL water was added dropwise, and the mixture was stirred for 10 min at room temperature. The solvent was evaporated, and the resulting crude residue was passed through a Dowex 1X8 resin (formate form) using aqueous formic acid solution (0-1 M) as an eluent. The solvent was removed *via* lyophilization to afford 6**a** as white powder (1.02 g, 76% yield).

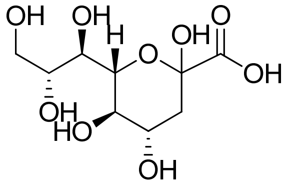
3-2. Characterization of sialic acids



(4*S*,5*R*,6*R*)-2-(dibromomethyl)-6-((1*R*,2*R*)-1,2,3-trihydroxypropyl)tetrahydro-2H-pyran-2,4,5-triol (5a)

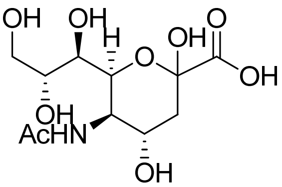
^{1}H NMR (500 MHz, D_2O) δ 5.81 (s, 1H), 3.90-3.85 (m, 2H), 3.81 (d, *J* = 9.8 Hz, 2H), 3.70-3.68 (m, 1H), 3.63 (dd, *J* = 10.9, 5.7 Hz, 1H), 3.44 (t, *J* = 9.75 Hz, 1H), 2.42 (dd, *J* = 12.6, 5.2 Hz, 1H) 1.59 (t, *J* = 12.6 Hz, 1H); ^{13}C NMR (125 MHz, CD_3OD) δ 97.9, 73.7, 72.3, 71.8, 71.0, 69.9, 65.0, 53.9, 39.5;IR (KBr): 3376, 1655, 1420, 1066, 1034;

HRMS (ESI): m/z calcd for $C_9H_{16}Br_2O_7$ $[M+Na]^+$ 418.9135 Found 418.9146; $[\alpha]_D^{23.5}$ = -12.7 (c = 0.49, MeOH).



KDN (6a)

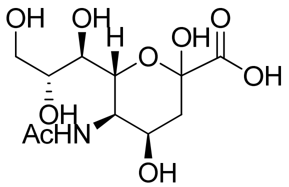
^{1}H NMR (500 MHz, D_2O) δ 3.93-3.88 (m, 2H), 3.79-3.75 (m, 2H), 3.67-3.64 (m, 1H), 3.57 (dd, J = 12.0, 6.3 Hz, 1H), 3.49 (t, J = 9.8 Hz, 1H), 2.18 (dd, J = 13.2, 4.6 Hz, 1H), 1.74 (t, J = 13.2 Hz, 1H); ^{13}C NMR (125 MHz, D_2O) δ 173.9, 95.9, 72.4, 71.1, 70.7, 69.3, 68.5, 63.9, 39.2; IR (KBr): 3399, 1743, 1440, 1281, 1210, 691 cm^{-1} HRMS (ESI): m/z calcd for $C_9H_{16}O_9$ $[M-H]^-$ 267.0721 Found 267.0721. $[\alpha]_D^{25}$ = -42 (c = 1, H_2O).



Neu5Ac (6j)

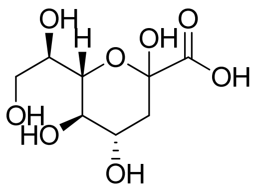
The reaction was conducted by following the general procedure, using **3j** (26.1 mg, 0.1 mmol), Br_2 (40 mg, 0.5 mmol) in H_2O (0.5 mL). The reaction mixture was stirred at room temperature for 5 min. Excess amounts of bromine were removed *via* extraction with hexane. The aqueous solution containing the product was used directly into the next step. K_2CO_3 (69.1 mg, 0.5 mmol) was added to the aqueous solution (0.5 mL) containing the crude product. The reaction was stirred for 30 min. Then CH_3COOH was added dropwise until pH = 4, and Pinnick oxidation using $NaClO_2$ (9.9 mg, 0.11 mmol), *t*BuOH (0.5 mL), and 2-methyl-2-butene (0.11 mL, 1 mmol) was carried out in one pot. The solution was evaporated, and the resulting crude residue was passed through a Dowex 1X8 resin (formate form) using aqueous formic acid solution (0-1 M) as an eluent. The solvent was removed *via* lyophilization to afford **6j** as white powder (22.9 mg, 74% yield).

^{1}H NMR (500 MHz, D_2O) δ 3.94-3.86 (m, 2H), 3.77 (t, J = 10.2 Hz, 1H), 3.69 (dd, J = 11.9, 2.6 Hz, 1H), 3.60 (ddd, J = 9.1, 6.4, 2.6 Hz, 1H), 3.46 (dd, J = 11.9, 6.4 Hz, 1H), 3.38 (dd, J = 9.1, 0.7 Hz, 1H), 2.11 (dd, J = 13.0, 4.9 Hz, 1H), 1.89 (s, 3H), 1.70 (dd, J = 13.0, 11.6 Hz, 1H); ^{13}C NMR (125 MHz, D_2O) δ 175.6, 174.0, 96.0, 71.1, 70.9, 68.9, 67.4, 63.9, 52.8, 39.5, 22.8; IR (KBr): 3433, 3398, 1719, 1637, 1559, 1457, 1128, 1069, 1036 cm^{-1} HRMS (ESI): m/z calcd for $C_{11}H_{19}NO_9$ $[M-H]^-$ 308.0987 Found 308.0994, $[\alpha]_D^{23.4}$ = -13.2 (*c* = 0.27, H_2O).



4-*epi*-Neu5Ac (6j')

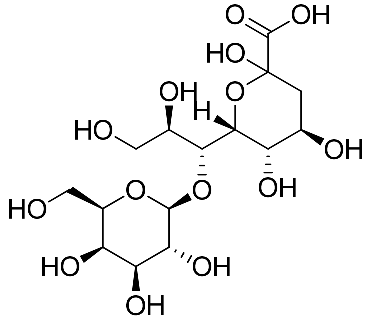
Using **4j** (26.1 mg, 0.1 mmol), the reaction was conducted by following the procedure for preparing **6j**. The corresponding product was obtained as a pale pink solid (20.1 mg, 65% from **4j**). Mixture of anomers. For the major isomer: ^1H NMR (500 MHz, D_2O) δ 4.20 (d, *J* = 10.8 Hz, 1H), 4.05-3.94 (m, 2H), 3.64 (dd, *J* = 11.8, 2.4 Hz, 1H), 3.61 (ddd, *J* = 9.0, 5.6, 2.4 Hz, 1H), 3.50-3.40 (m, 2H), 2.02 (dd, *J* = 14.9, 3.3 Hz, 1H), 1.97 (dd, *J* = 14.9, 3.3 Hz, 1H), 1.87 (s, 3H); ^{13}C NMR (125 MHz, D_2O) δ 174.9, 174.0, 95.8, 70.7, 69.1, 66.7, 66.4, 63.9, 48.3, 36.8, 22.6; IR (KBr): 3397, 1750, 1735, 1654, 1637, 1628, 1125, 1089, 1031 cm^{-1} HRMS (ESI): m/z calcd for $C_{11}H_{19}NO_9$ $[M-H]^-$ 308.0987 Found 308.0994,



(4*S*,5*R*,6*S*)-6-((*R*)-1,2-dihydroxyethyl)-2,4,5-trihydroxytetrahydro-2*H*-pyran-2-carboxylic acid (6d)

Using **4d** (19 mg, 0.1 mmol), the reaction was conducted by following the procedure for preparing **6j**. The corresponding product was obtained as a white solid. (19.5 mg, 82% from **4d**).

^1H NMR (500 MHz, D_2O) δ 3.91-3.88 (m, 1H), 3.81 (ddd, *J* = 11.6, 9.2, 5.1 Hz, 1H), 3.62-3.58 (m, 1H), 3.53 (dd, *J* = 11.6, 7.6 Hz, 1H), 3.49-3.41 (m, 2H), 2.07 (dd, *J* = 13.0, 5.1 Hz, 1H), 1.65 (t, *J* = 13.0, 1H); ^{13}C NMR (125 MHz, D_2O) δ 174.5, 96.1, 73.3, 70.8, 69.3, 69.3, 63.5, 39.3; IR (KBr): 3406, 1685, 1438, 1403, 1207, 1142, 624 cm^{-1}; HRMS (ESI): m/z calcd for $C_8H_{14}O_8$ $[M-H]^-$ 237.0615 Found 237.0626; $[\alpha]_D^{22.6}$ = -4.5 (*c* = 6.78, H_2O)



(4*R*,5*S*,6*R*)-6-((1*R*,2*R*)-2,3-dihydroxy-1-(((2*S*,3*R*,4*S*,5*R*,6*R*)-3,4,5-trihydroxy-6-(hydroxymethyl)tetrahydro-2*H*-pyran-2-yl)oxy)propyl)-2,4,5-trihydroxytetrahydro-2*H*-pyran-2-carboxylic acid (6m)

The reaction was conducted by following the general procedure, using **3m** (100 mg, 0.26 mmol), KBr (74.8 mg, 0.63 mmol), and Oxone (193 mg, 0.63 mmol) in H_2O (2.4 mL). The reaction mixture was stirred at room temperature for 5 min. Inorganic salts were roughly removed using C_{18} reverse phase column, and the crude product was used directly for the next step without further purification. K_2CO_3 (144 mg, 1.0 mmol) was added to the crude product dissolved in H_2O (2.4 mL). The reaction was stirred for 30 min. Then CH_3COOH was added dropwise until pH = 4, and Pinnick oxidation using $NaClO_2$ (25.9 mg, 0.29 mmol), *t*BuOH (2.4 mL), and 2-methyl-2-butene (275 μL, 0.90 mmol) was carried out in one pot. The crude product was passed through a Dowex 1X8 resin (acetate form) using aqueous CH_3COOH solution (0-2 M) as an eluent. Products were purified by preparative reverse phase HPLC using a gradient of acetonitrile versus 0.1% TFA in water, affording **6m** as a white solid (59.3 mg, 53% from **3m**). Preparative HPLC was carried out as follows: YMC-Triart C18 (20 mm I.D × 250 mm) column using a linear gradient of 0-50% acetonitrile in 0.1% aqueous TFA over 30 min at room temperature with a flow rate of 7.0 mL min^{-1}.

^{1}H NMR (400 MHz, D_2O) δ 4.33 (dd, *J* = 7.7, 2.1 Hz, 1H), 4.17 (dd, *J* = 9.3, 2.1 Hz, 1H), 3.83-3.76 (m, 5H), 3.63-3.48 (m, 6H), 3.40-3.35 (m, 1H), 2.92 (ddd, *J* = 18.1, 5.1, 2.2 Hz, 1H), 2.41 (dd, *J* = 18.1, 2.2 Hz, 1H); ^{13}C NMR (125 MHz, D_2O) δ 180.4, 103.5, 87.1, 76.8, 75.6, 73.2, 71.7, 71.4, 69.3, 68.5, 67.9, 62.8, 61.9, 40.3; IR (neat): 3389, 1758, 1638, 1077, 1043; HRMS (ESI): m/z calcd for $C_{15}H_{26}O_{14}$ $[M-H]^-$ 429.1249 Found 429.1266; $[\alpha]_D^{21.3}$ = +21.5 (*c* = 2.28, H_2O).

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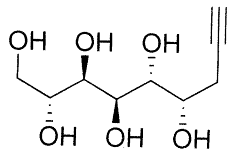
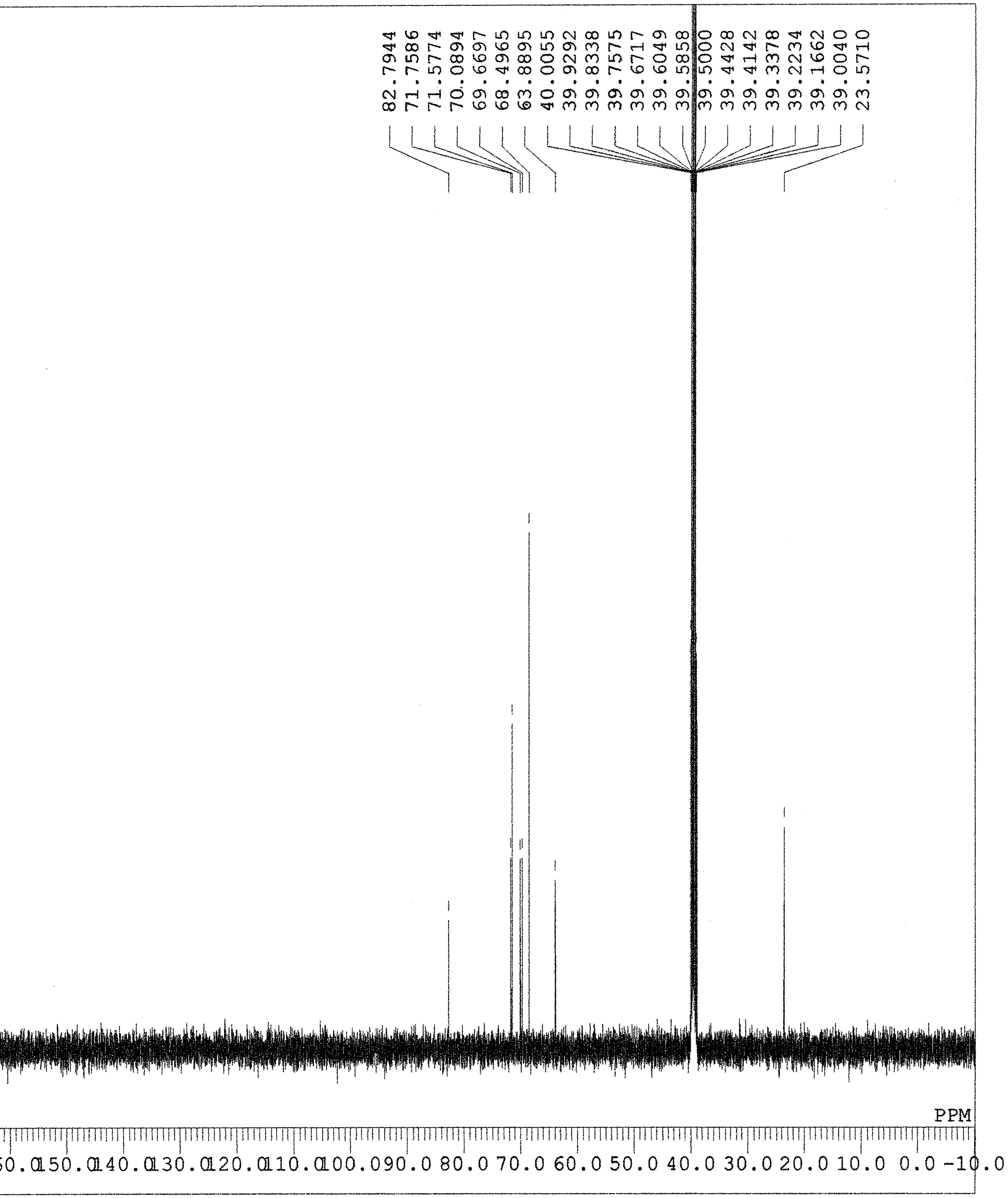


3a



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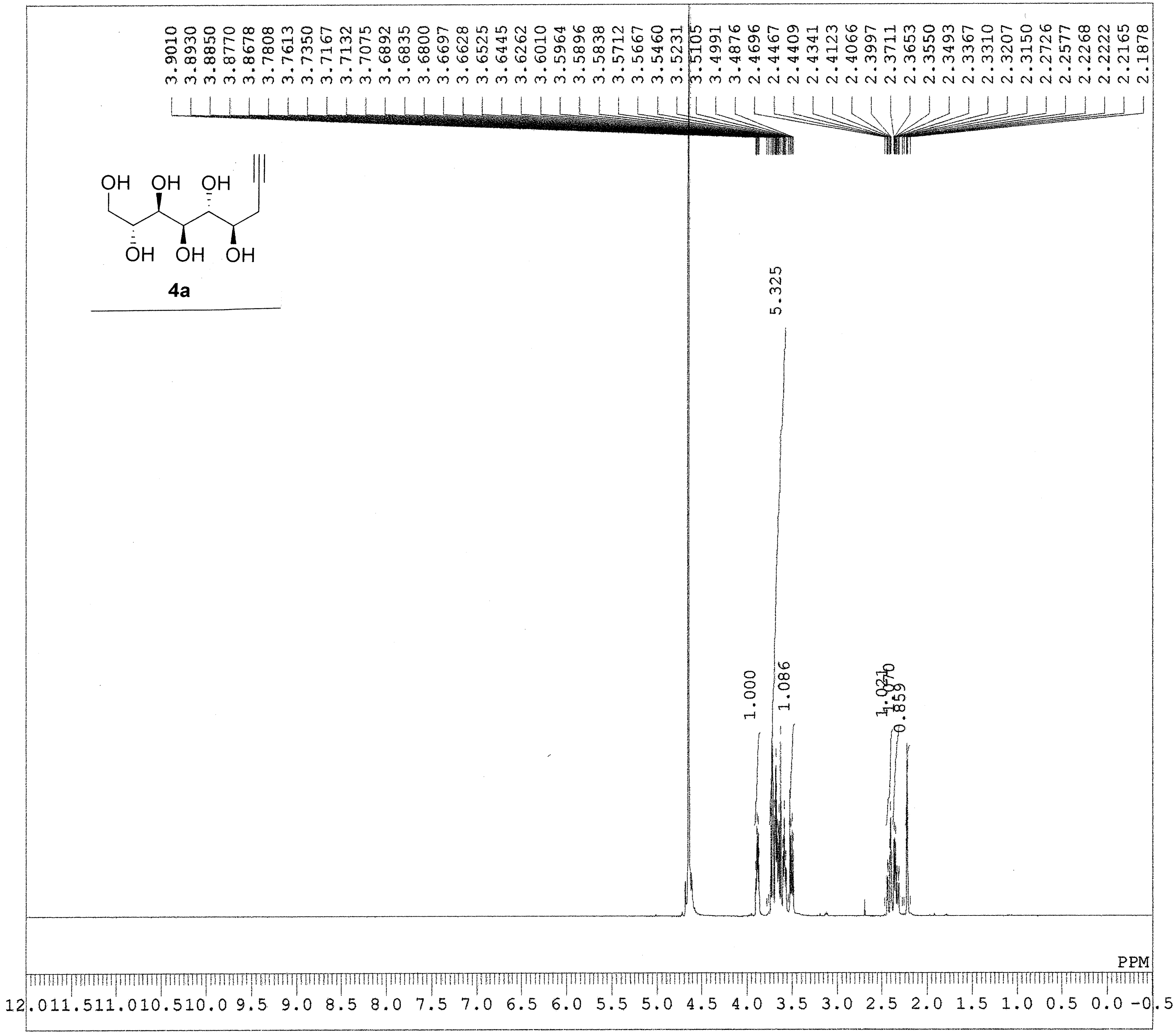

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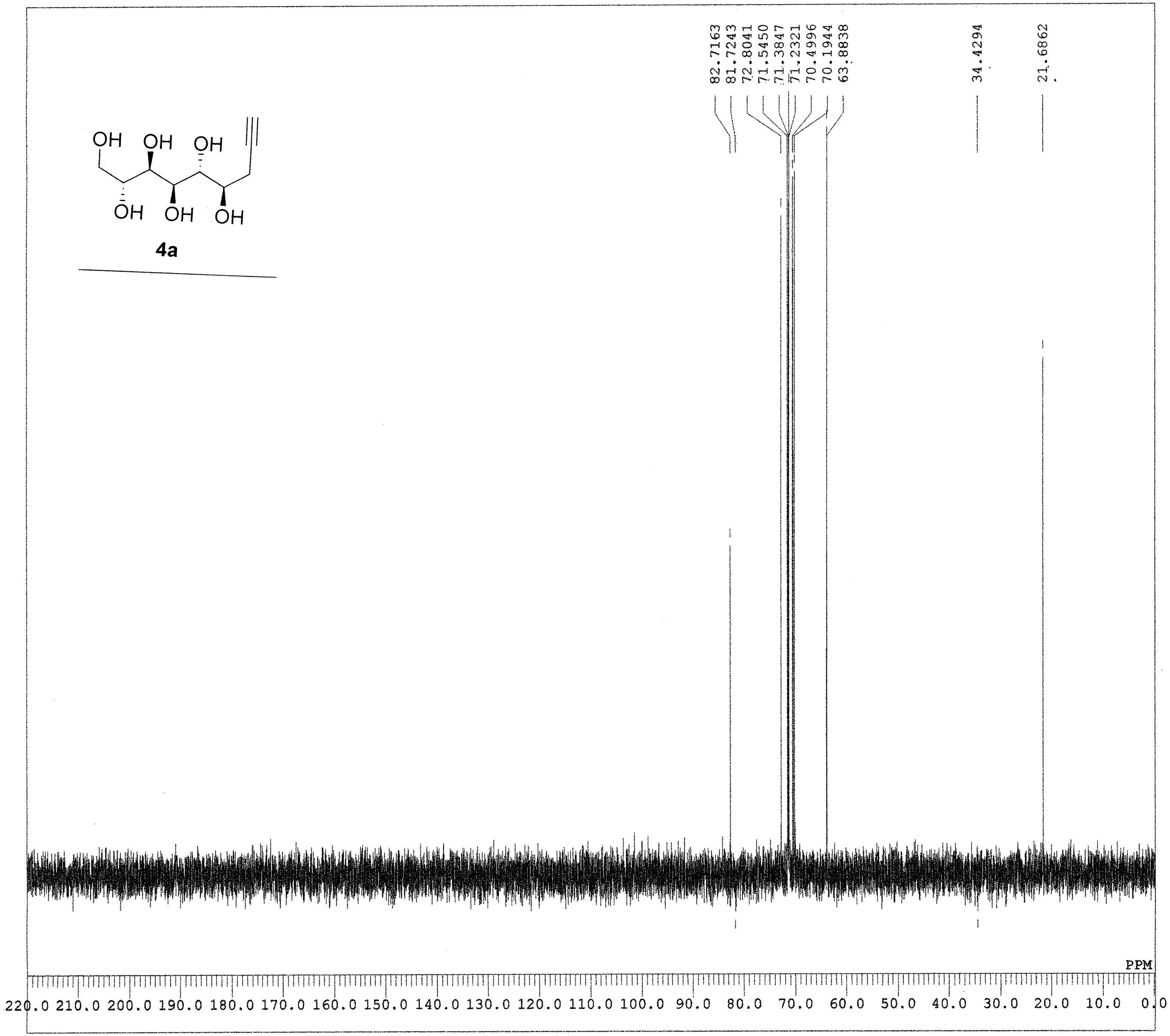

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CTEMP	20.9 c
SLVNT	D2O
EXREF	49.50 ppm
BF	1.02 Hz
RGAIN	76

3b

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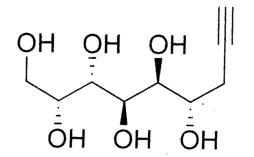
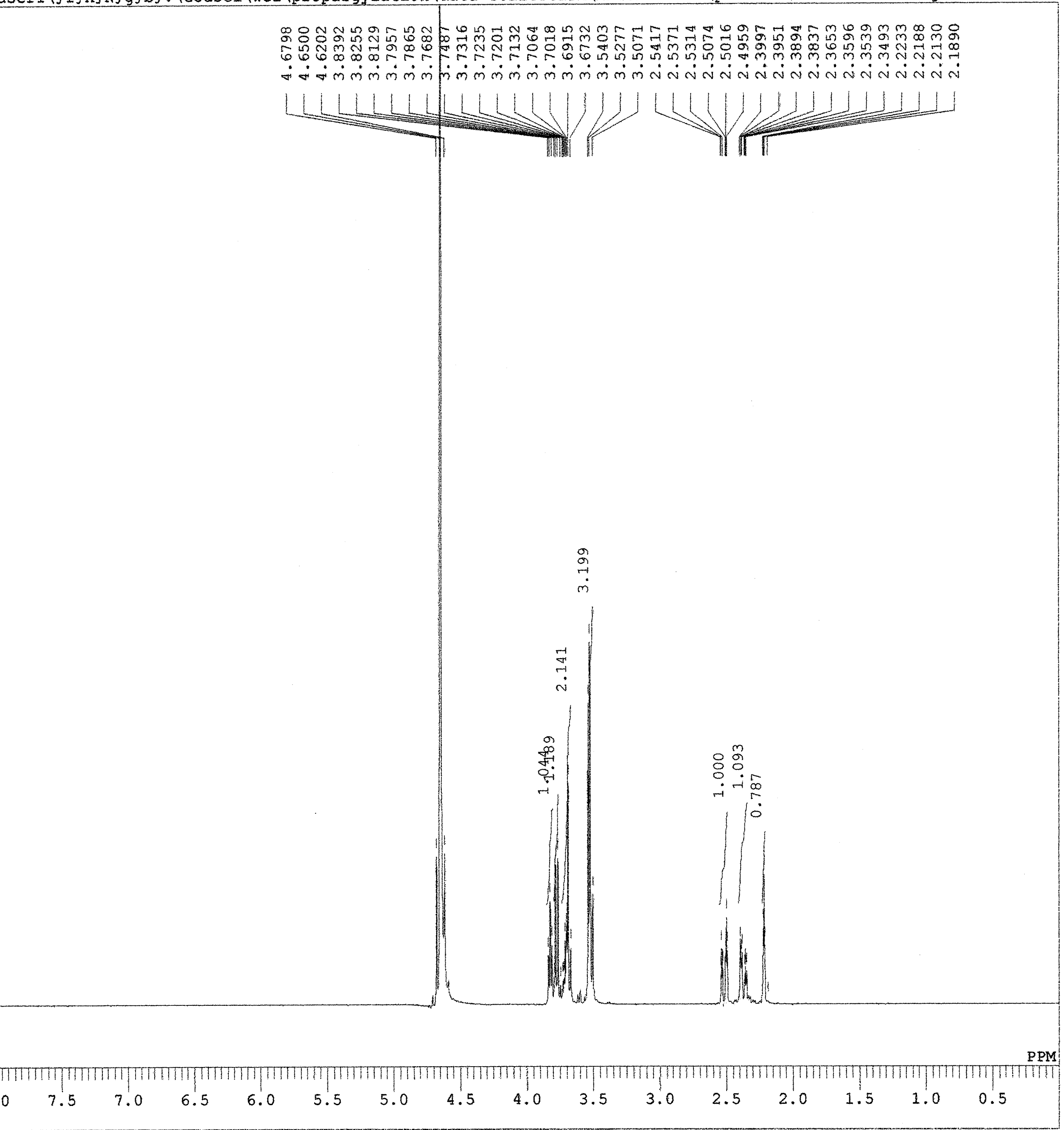


3b



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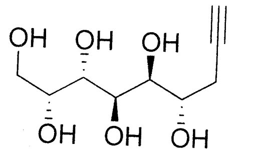
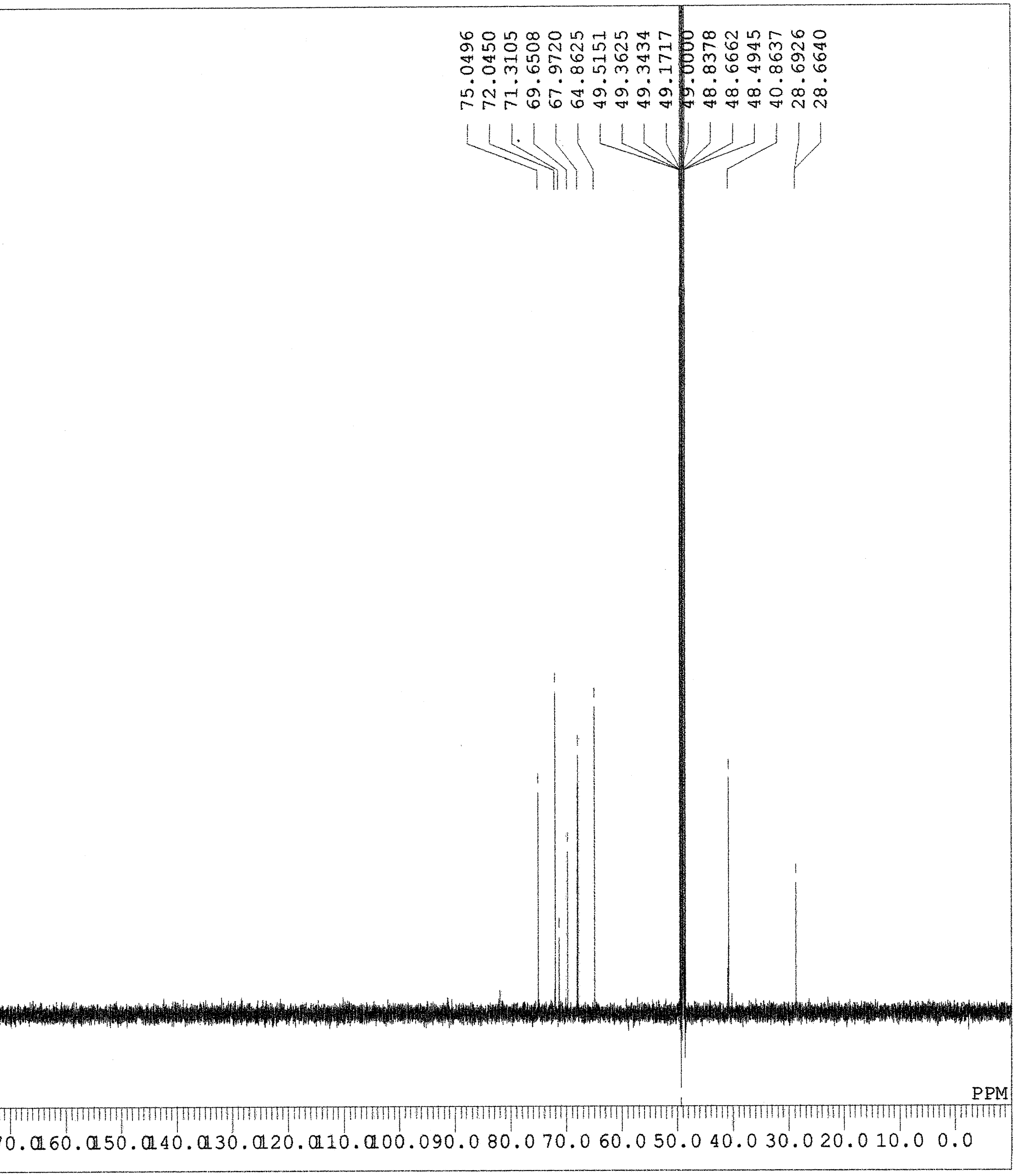
3b



3b



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CTEMP	21.9 c
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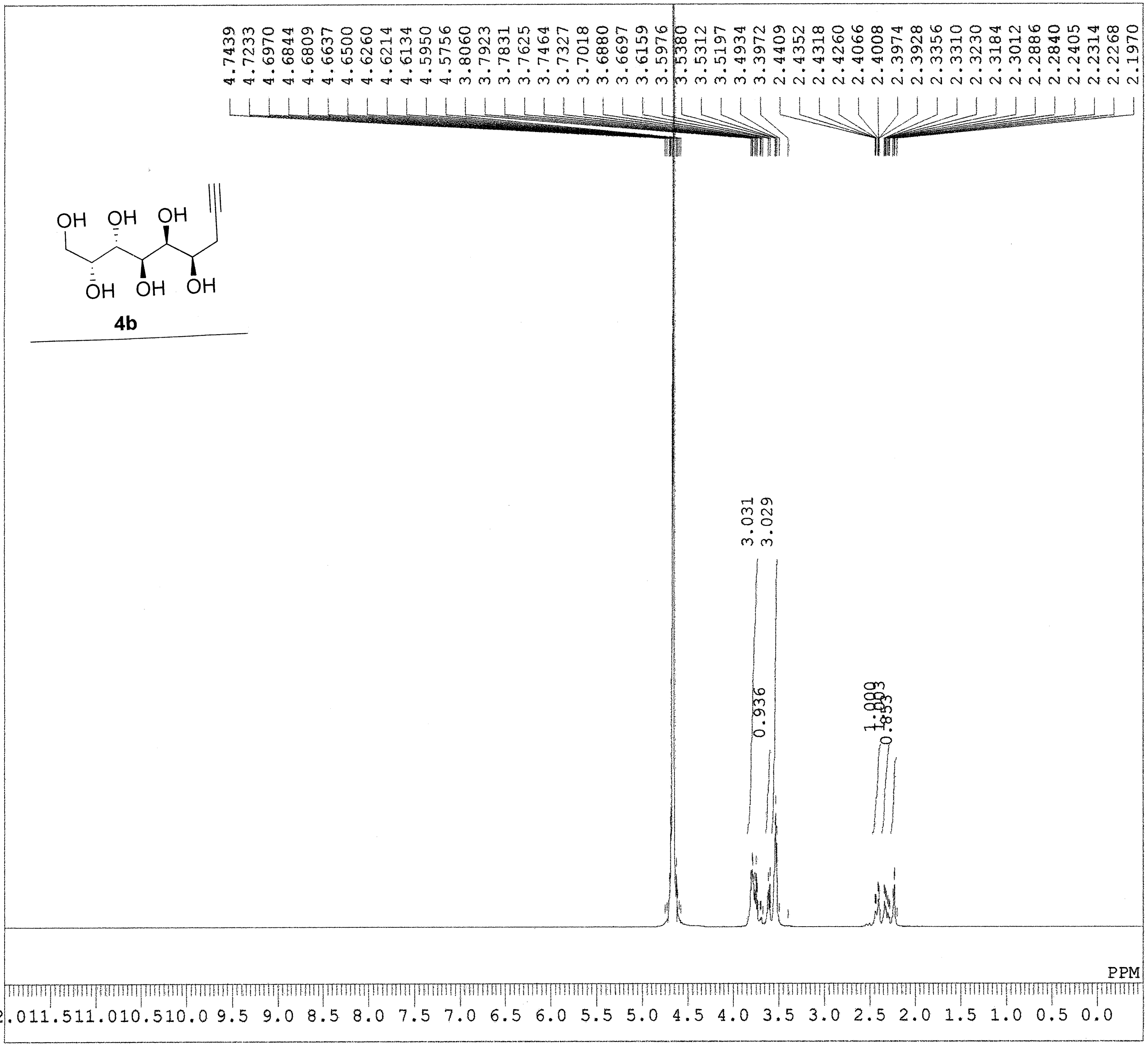
RGAIN	60

4b



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PD	5.0000 sec
PW1	5.55 usec
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CTEMP	21.3 c
SLVNT	D2O
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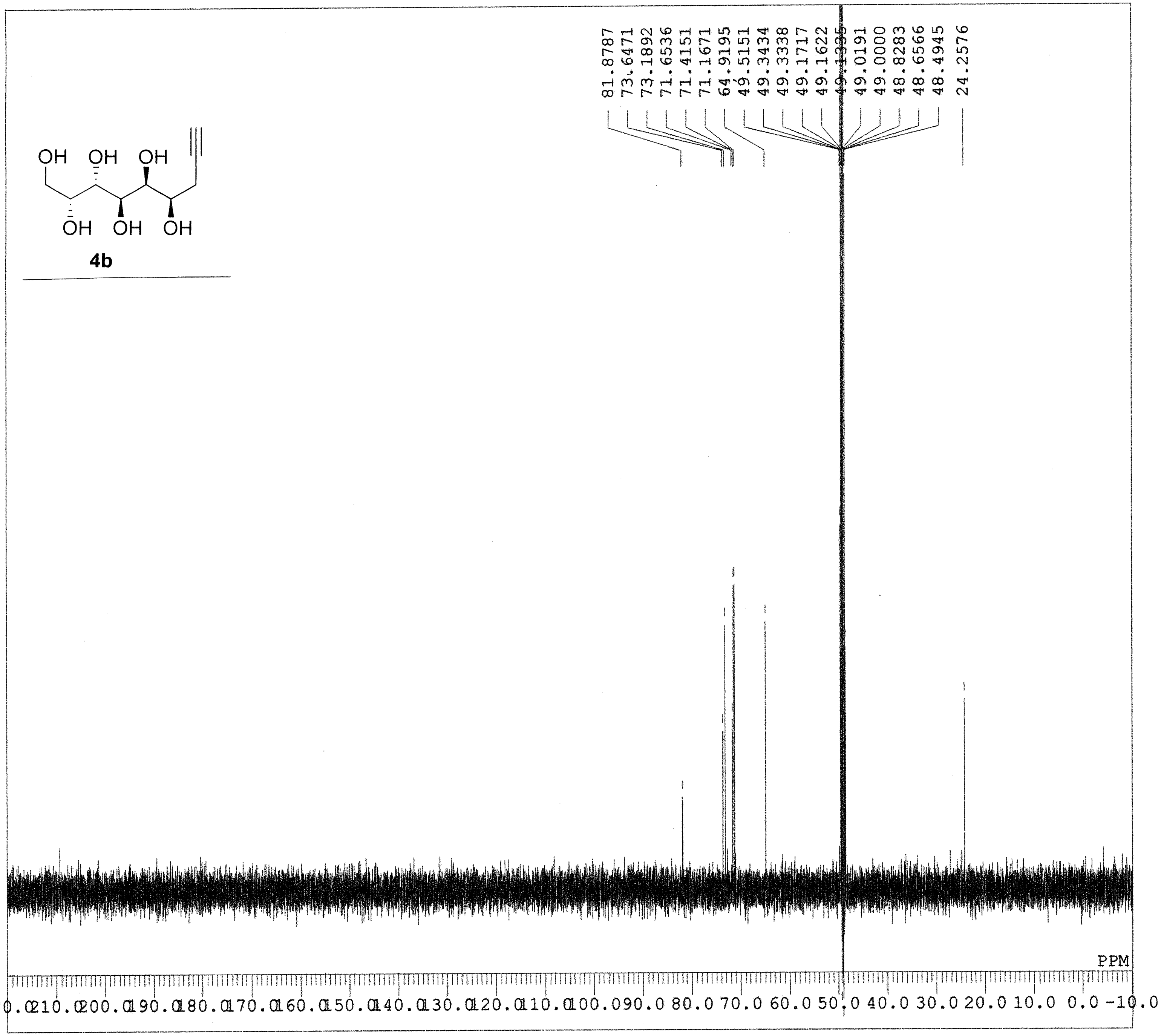
BF	0.12 Hz
RGAIN	30

4b



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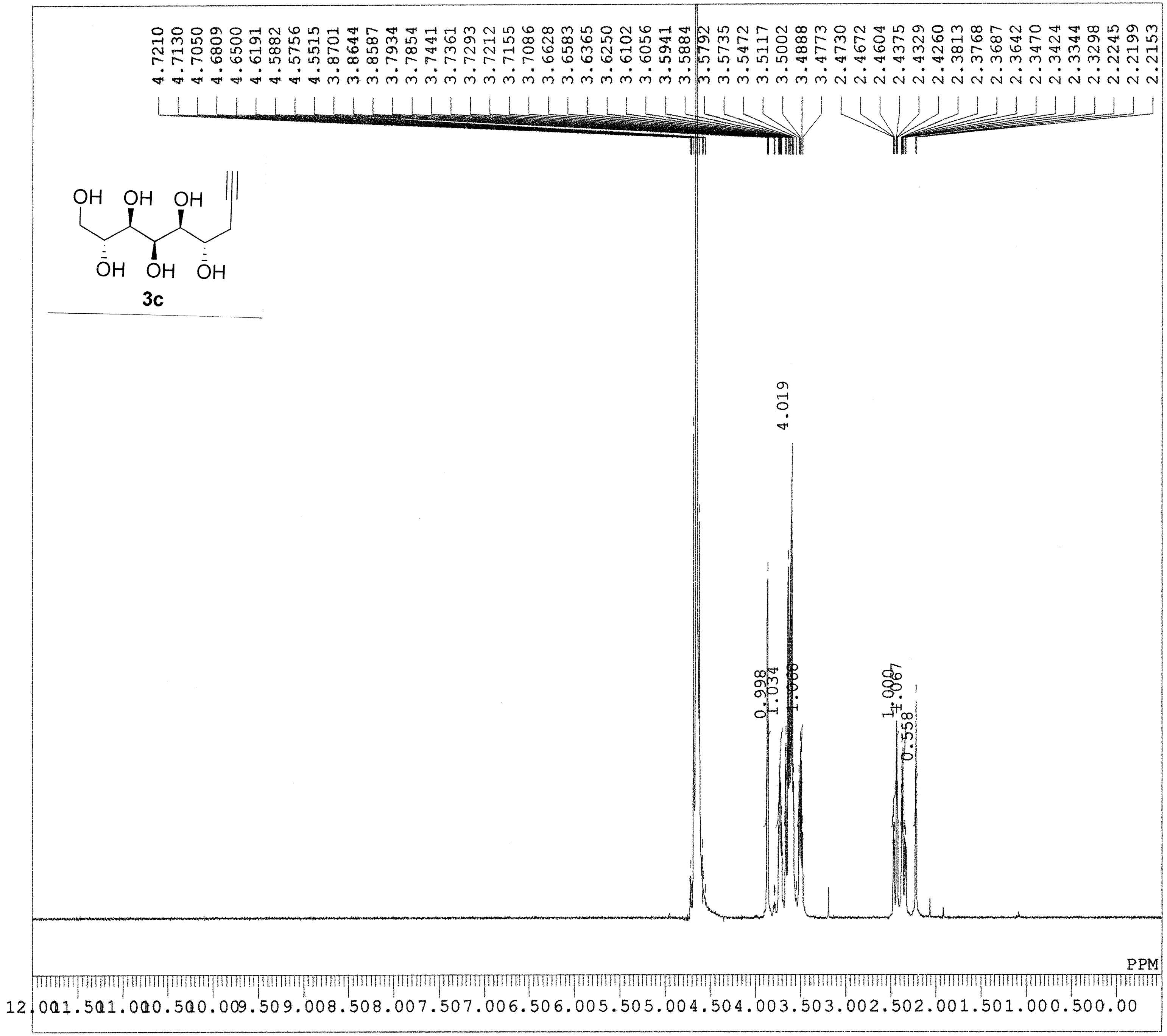
RGAIN	60

3C



3c

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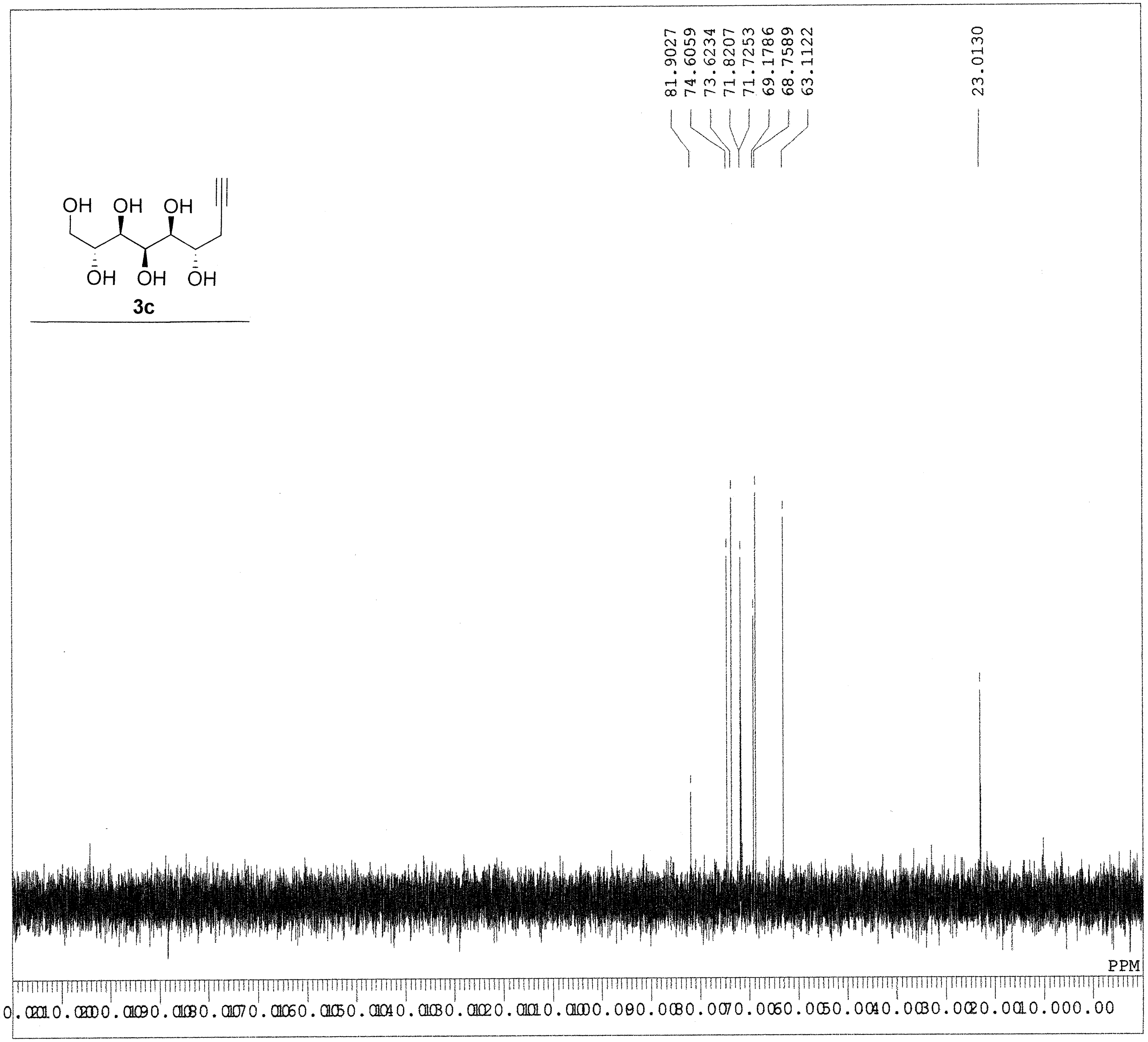
RGAIN	30



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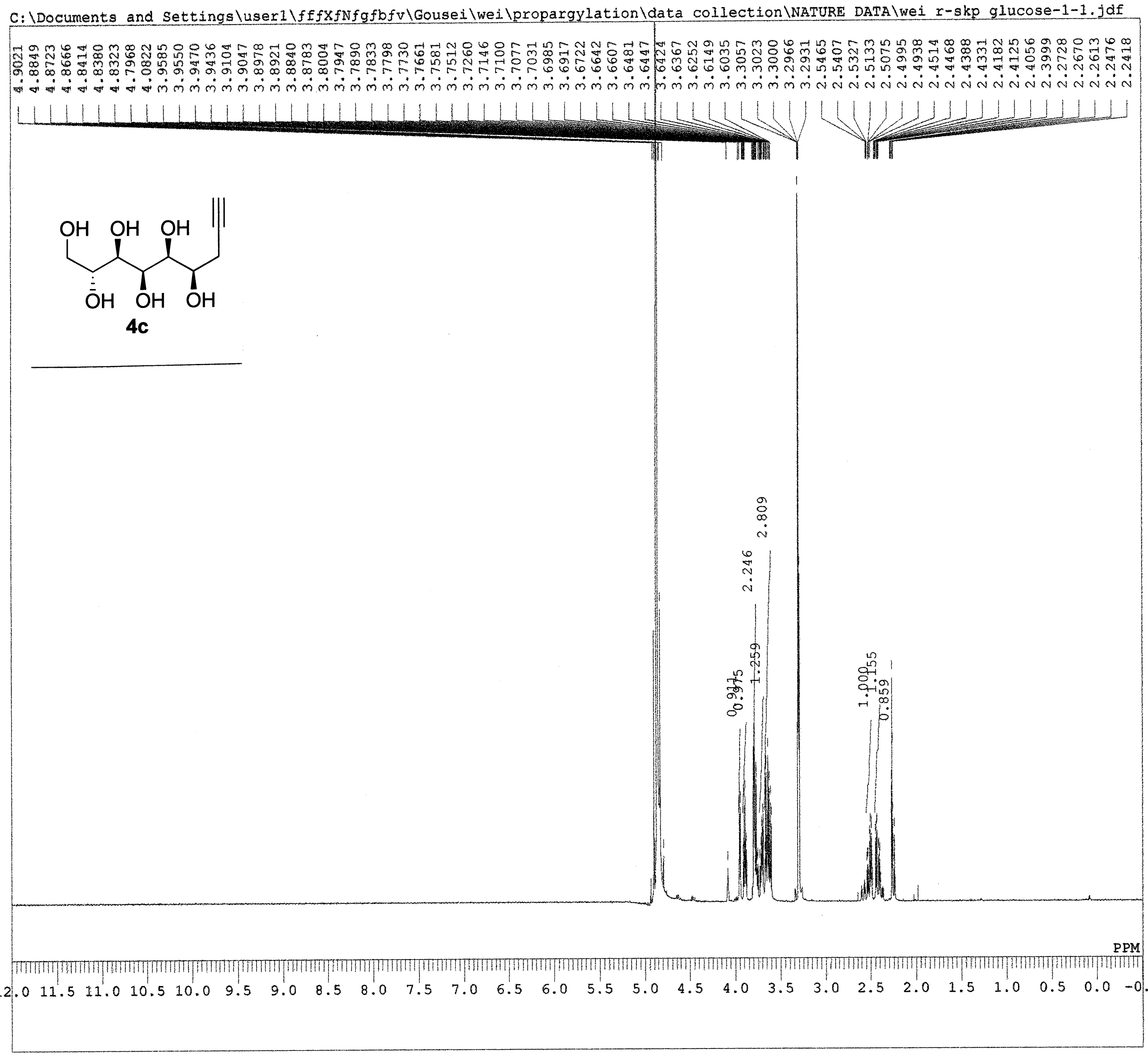

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PD 5.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 21.5 c
SLVNT CD3OD
EXREF 3.30 ppm
BF 0.12 Hz
RGAIN 30

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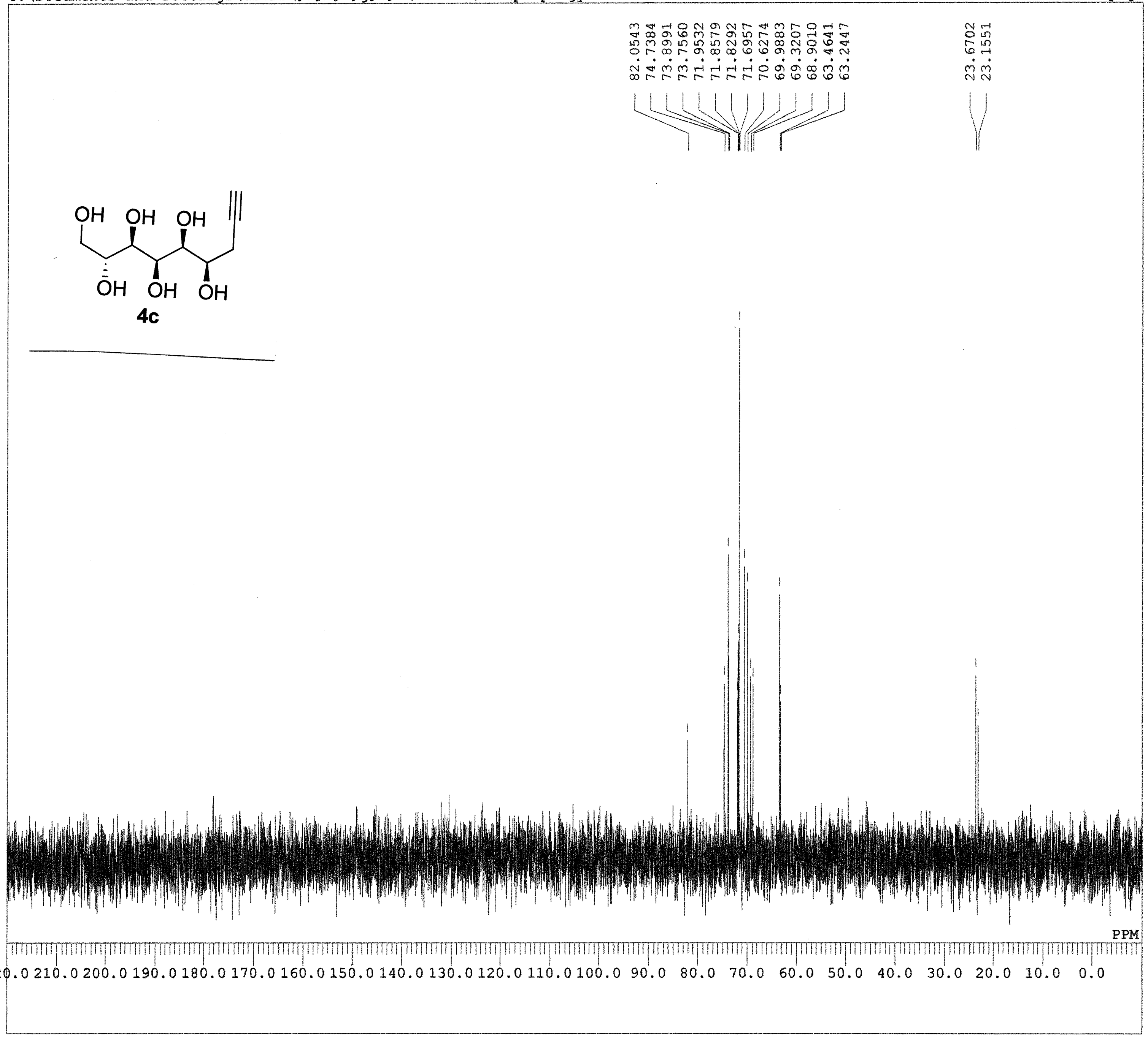

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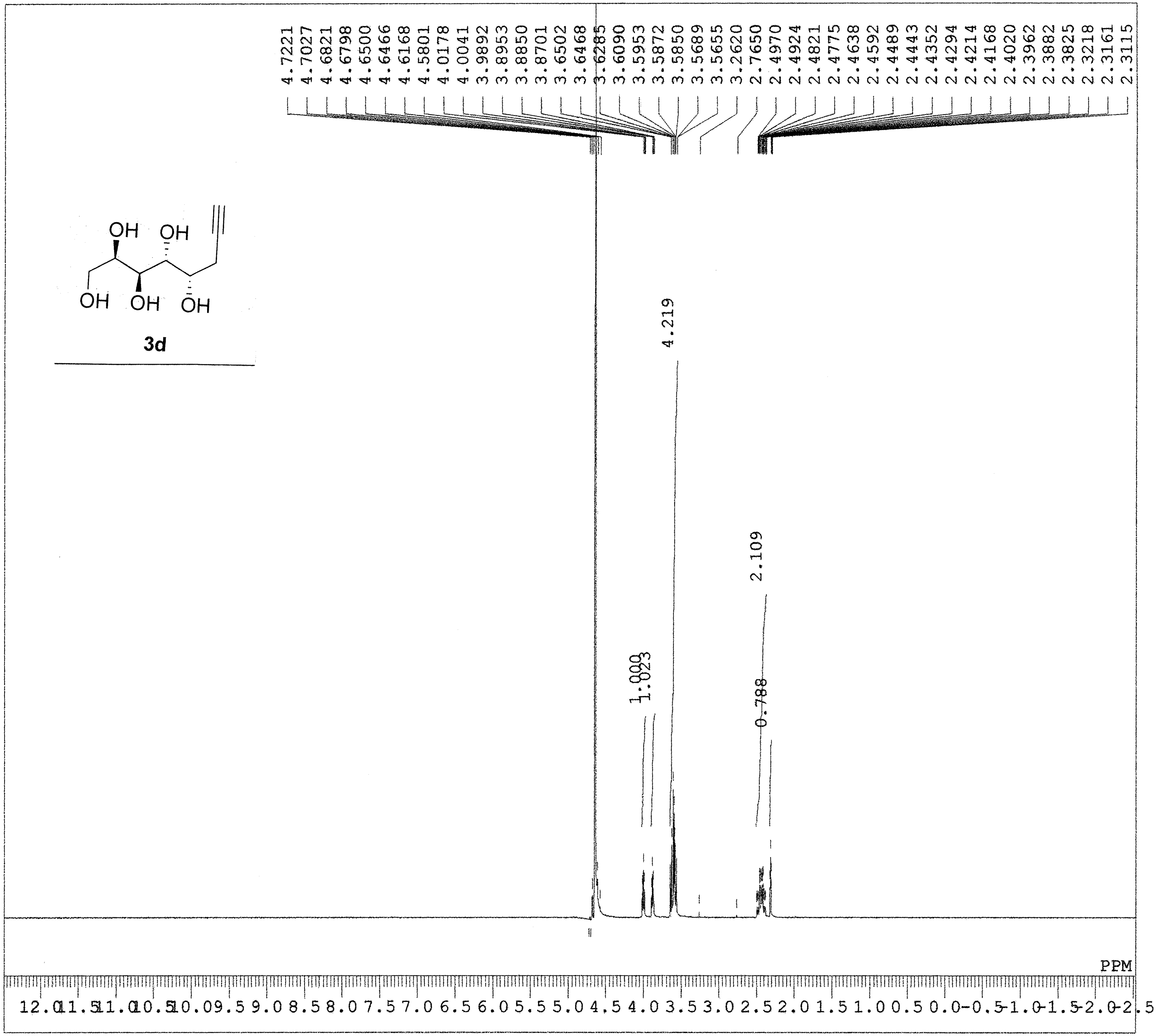
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32767
FREQU	39308.18 Hz
SCANS	2180
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	21.7 c
SLVNT	D2O
EXREF	49.50 ppm
BF	1.20 Hz
RGAIN	60



DFILE	3d S-SKP lyxose alkyne.als
COMNT	
DATIM	2015-04-27 16:50:14
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	12
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	28.0 c
SLVNT	D2O
EXREF	4.65 ppm
BF	0.12 Hz
RGAIN	34

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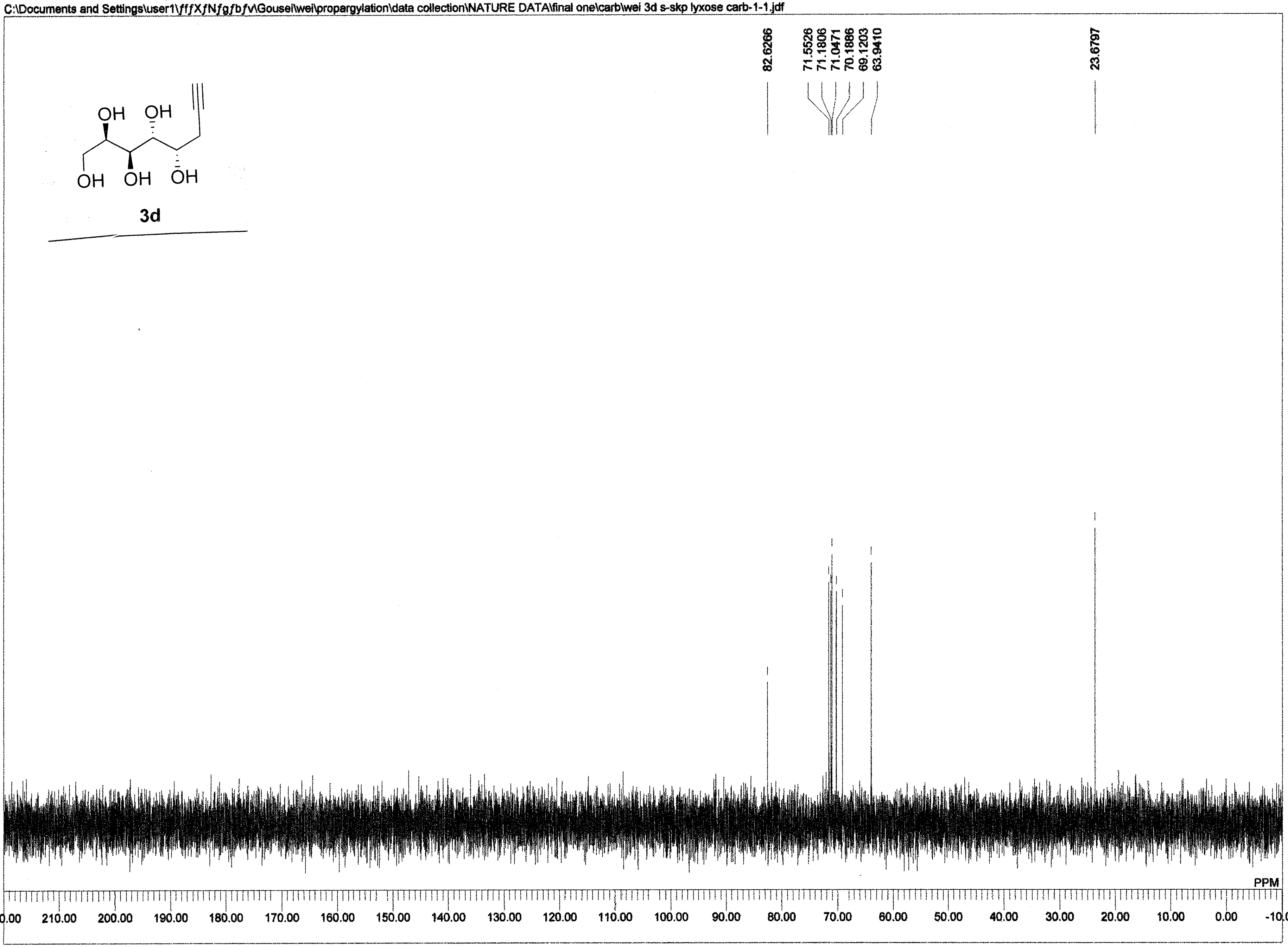
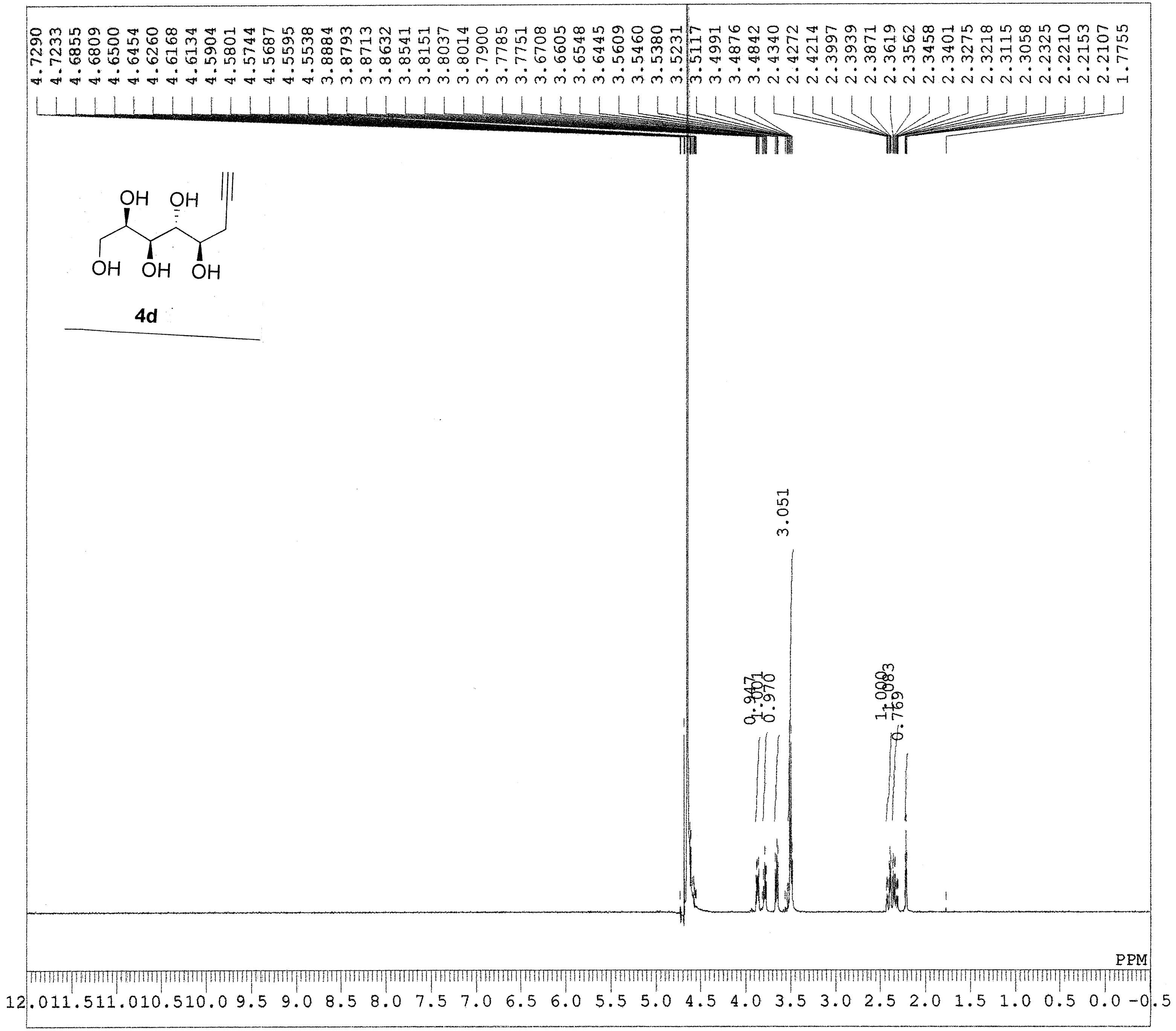
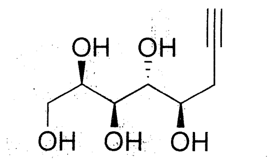


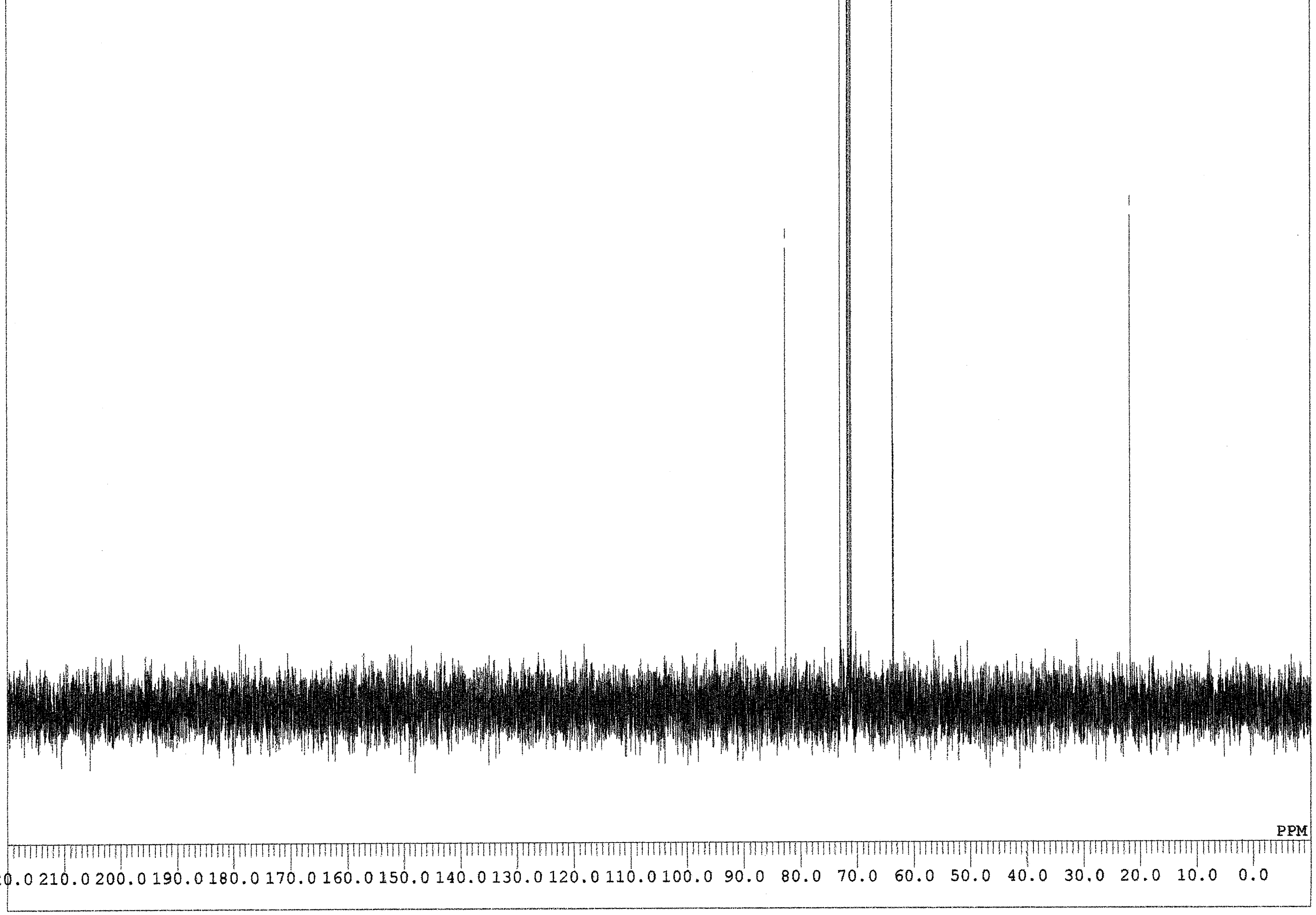
DFILE	wei 3d s-skp lyxose carb-1-1
COMNT	
DATIM	2015-09-03 22:02:21
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32767
FREQU	39308.18 Hz
SCANS	1344
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	21.6 c
SLVNT	D2O
EXREF	49.50 ppm
BF	0.12 Hz
RGAIN	60



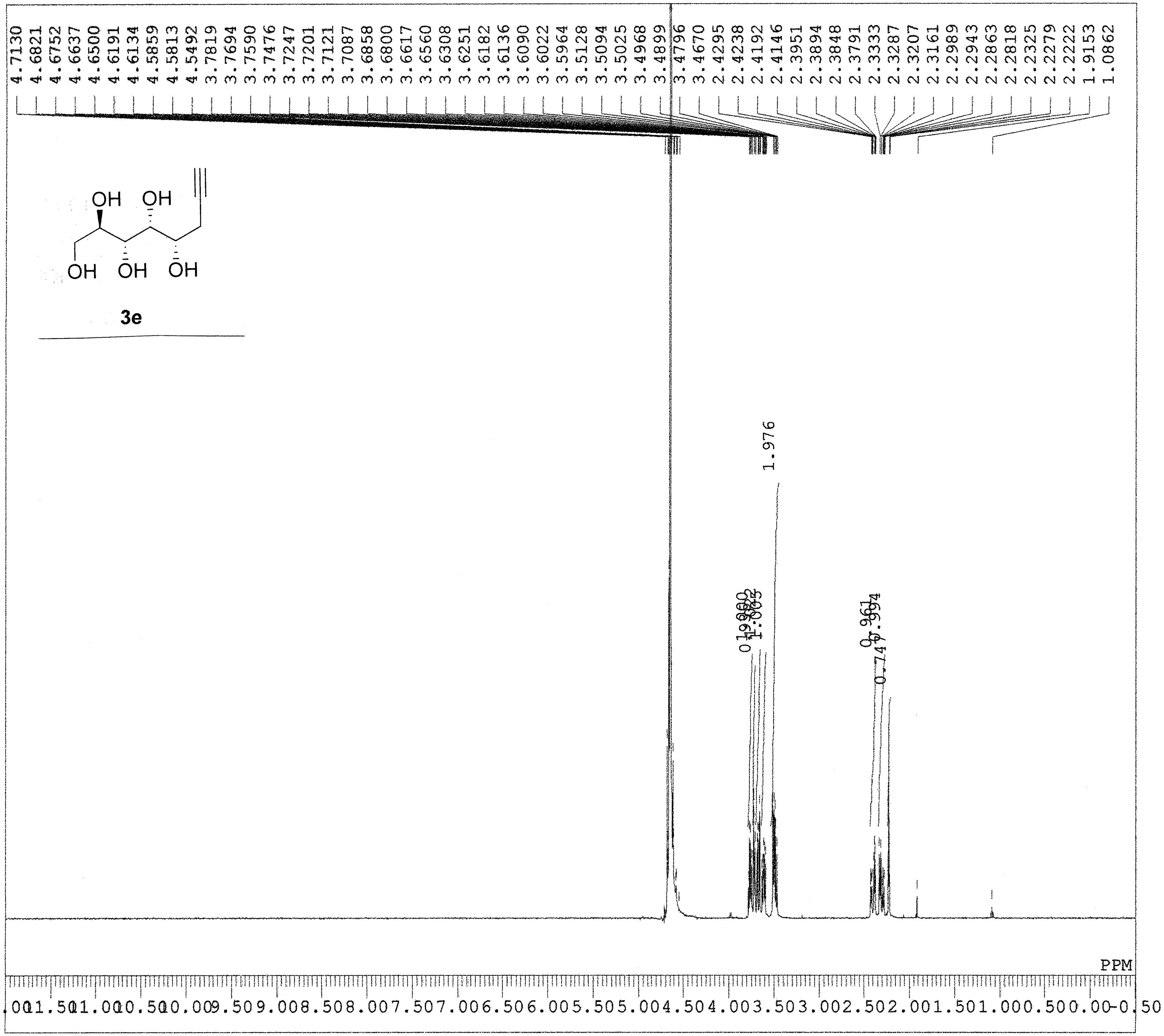
DFILE	4d R-skp lyxose alkyne.als
COMNT	
DATIM	2015-06-15 15:57:01
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	6
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.0 c
SLVNT	D2O
EXREF	4.65 ppm
BF	0.12 Hz
RGAIN	30



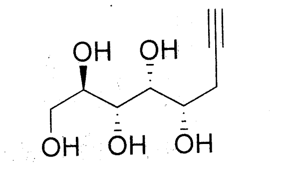
4d



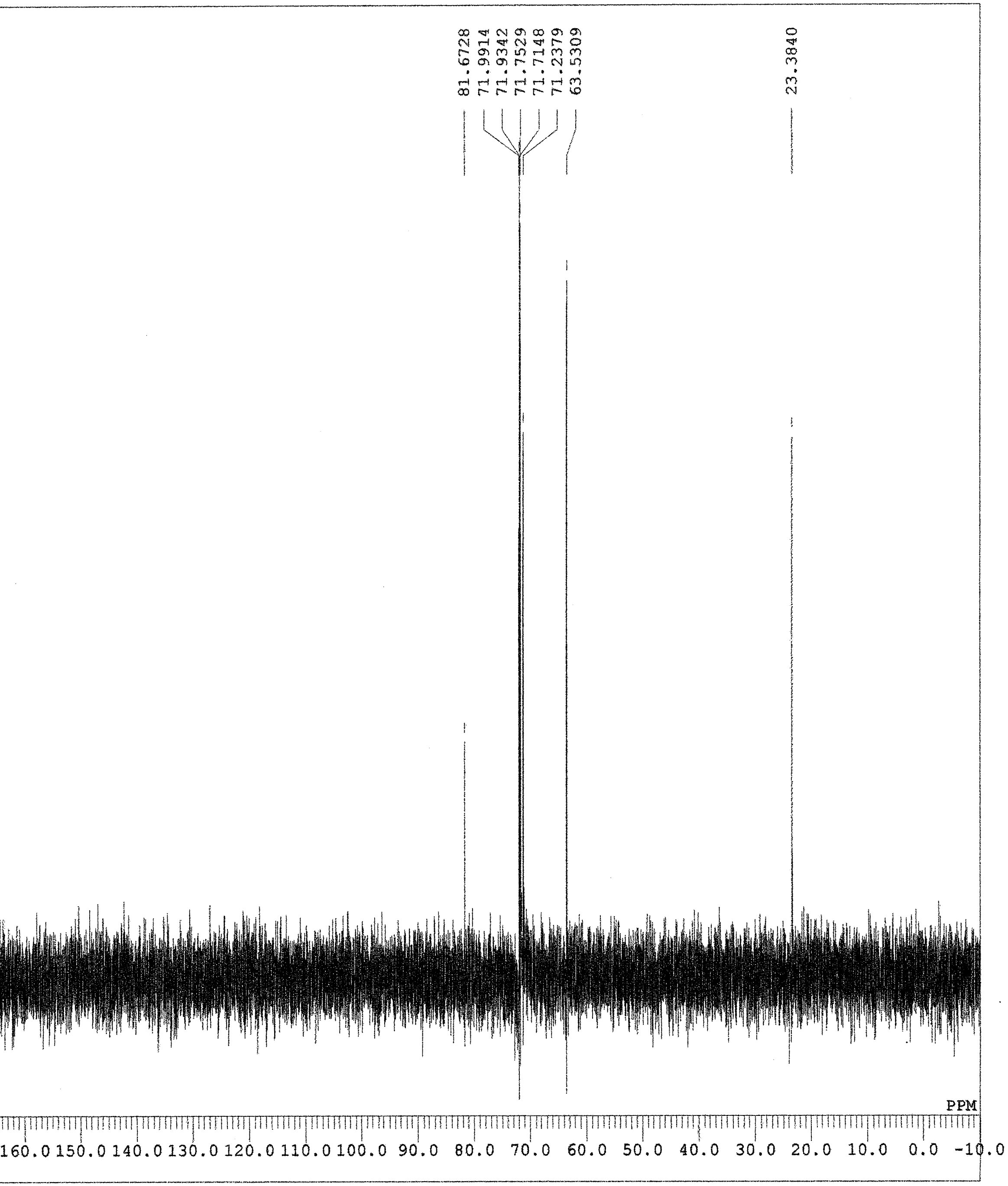
DFILE	4d R-SKP PGF lyxose carb-1-1.jdf
COMNT	
DATIM	2015-06-08 14:25:18
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32768
FREQU	31446.54 Hz
SCANS	117
ACQTM	1.0420 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	21.6 c
SLVNT	D2O
EXREF	49.50 ppm
BF	1.02 Hz
RGAIN	76



DFILE	3e s-skp arabinose-1-1.jdf
COMNT	
DATIM	2015-07-08 16:22:18
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16384
FREQU	9384.38 Hz
SCANS	6
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.2 c
SLVNT	D2O
EXREF	4.65 ppm
BF	0.12 Hz
RGAIN	30

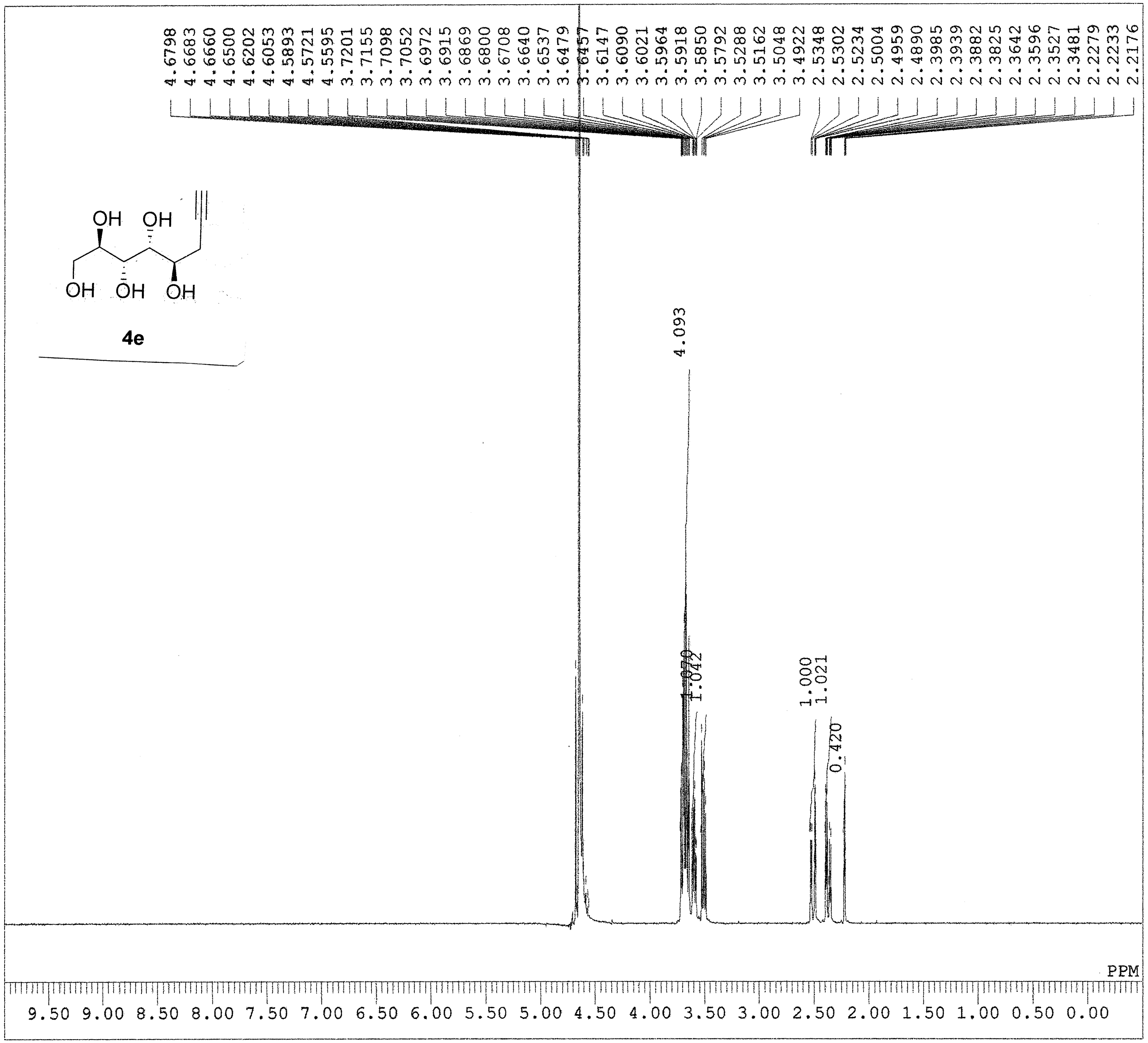


3e



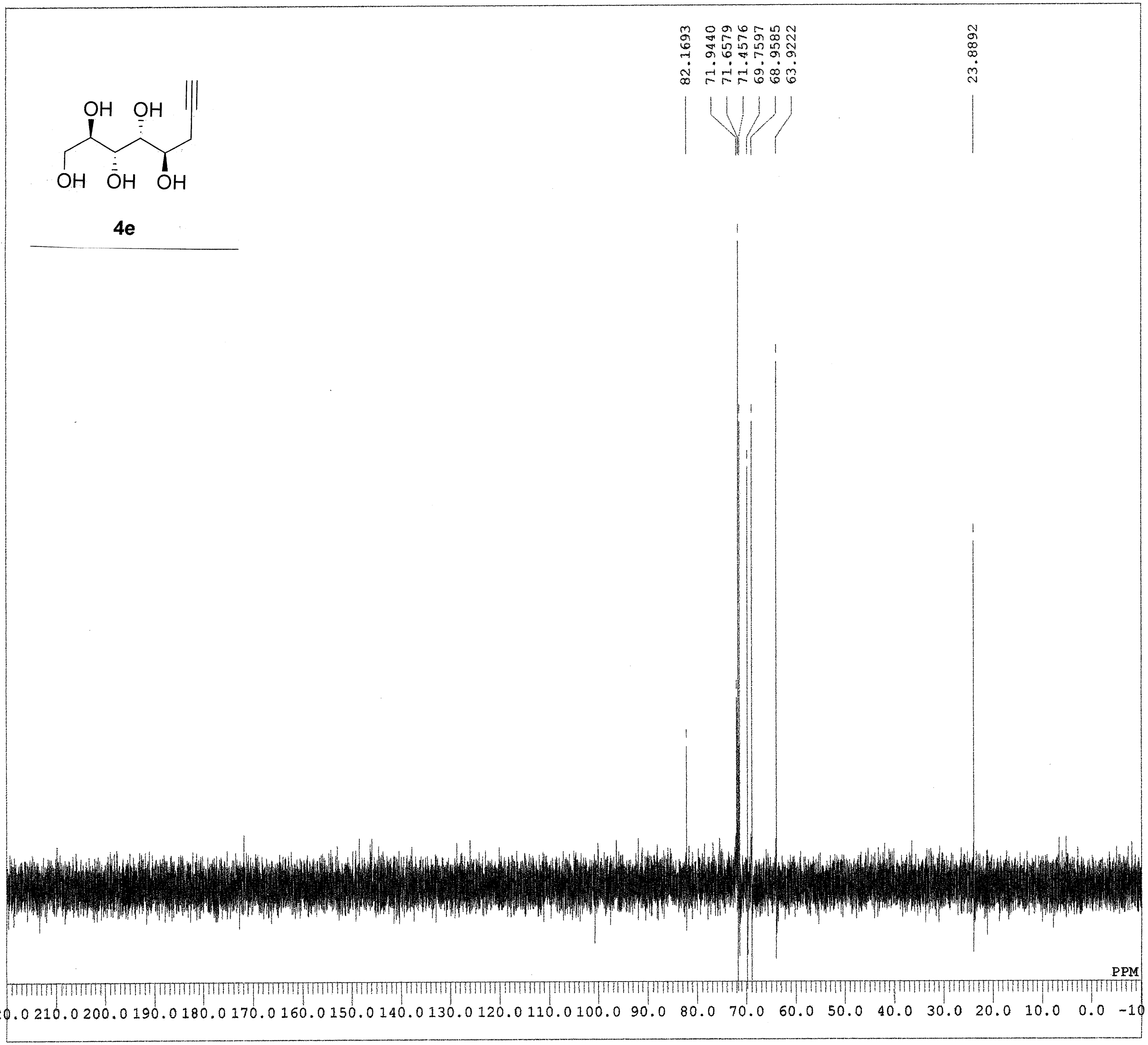
DFILE	3e s-skp arabinose.jdf
COMNT	
DATIM	2015-07-09 09:09:31
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32767
FREQU	39308.18 Hz
SCANS	1440
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	21.7 c
SLVNT	D2O
EXREF	49.50 ppm
BF	0.12 Hz
RGAIN	60

4e



DFILE wei r-skp arabinose-1-1.als
COMNT
DATIM 2015-07-10 09:26:50
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 21.4 c
SLVNT D2O
EXREF 4.65 ppm
BF 0.12 Hz
RGAIN 30

4e



4e

DFILE	wei r-skp arabinose carb-1-1.als
COMNT	
DATIM	2015-07-10 09:28:20
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.54 Hz
SCANS	1323
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	21.7 c
SLVNT	D2O
EXREF	49.50 ppm
BF	0.12 Hz
RGAIN	60

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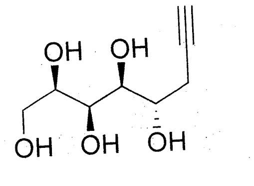

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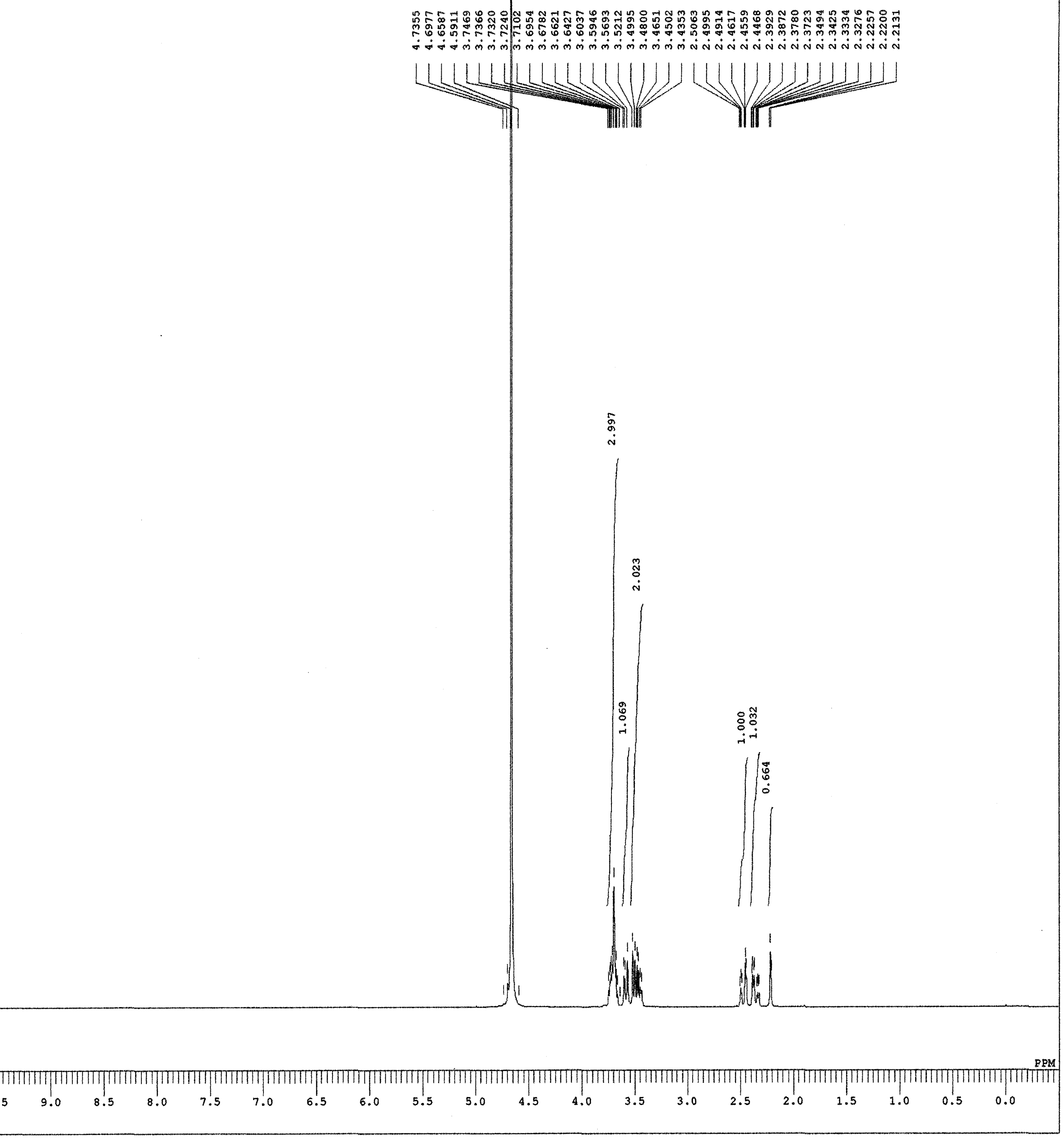

DFILE	wei 928-6-1-1.jdf
COMNT	
DATIM	07-07-2015 18:52:06
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	16384
FREQU	7352.94 Hz
SCANS	14
ACQTM	2.2282 sec
PD	5.0000 sec
PW1	4.99 usec
IRNUC	1H
CTEMP	21.5 c
SLVNT	D2O
EXREF	0.00 ppm
BF	1.20 Hz
RGAIN	38

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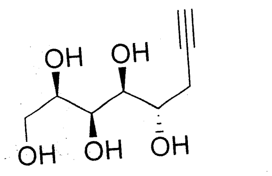

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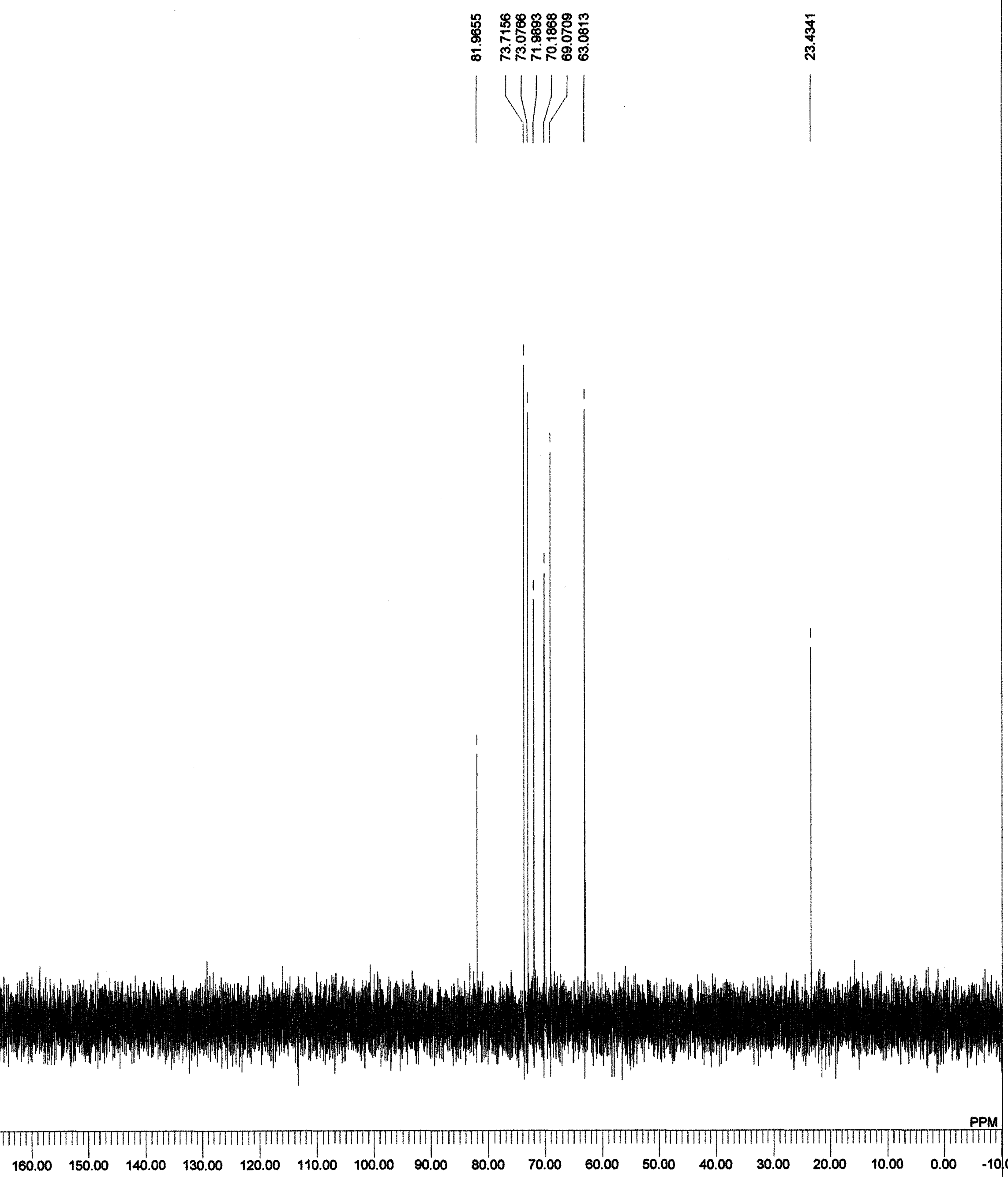
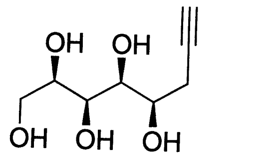


DFILE	wei 928-6 carb-1-1.jdf
COMNT	
DATIM	07-07-2015 18:54:28
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	32767
FREQU	30788.18 Hz
SCANS	1700
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	3.16 usec
IRNUC	1H
CTEMP	21.7 c
SLVNT	D2O
EXREF	49.50 ppm
BF	0.12 Hz
RGAIN	60



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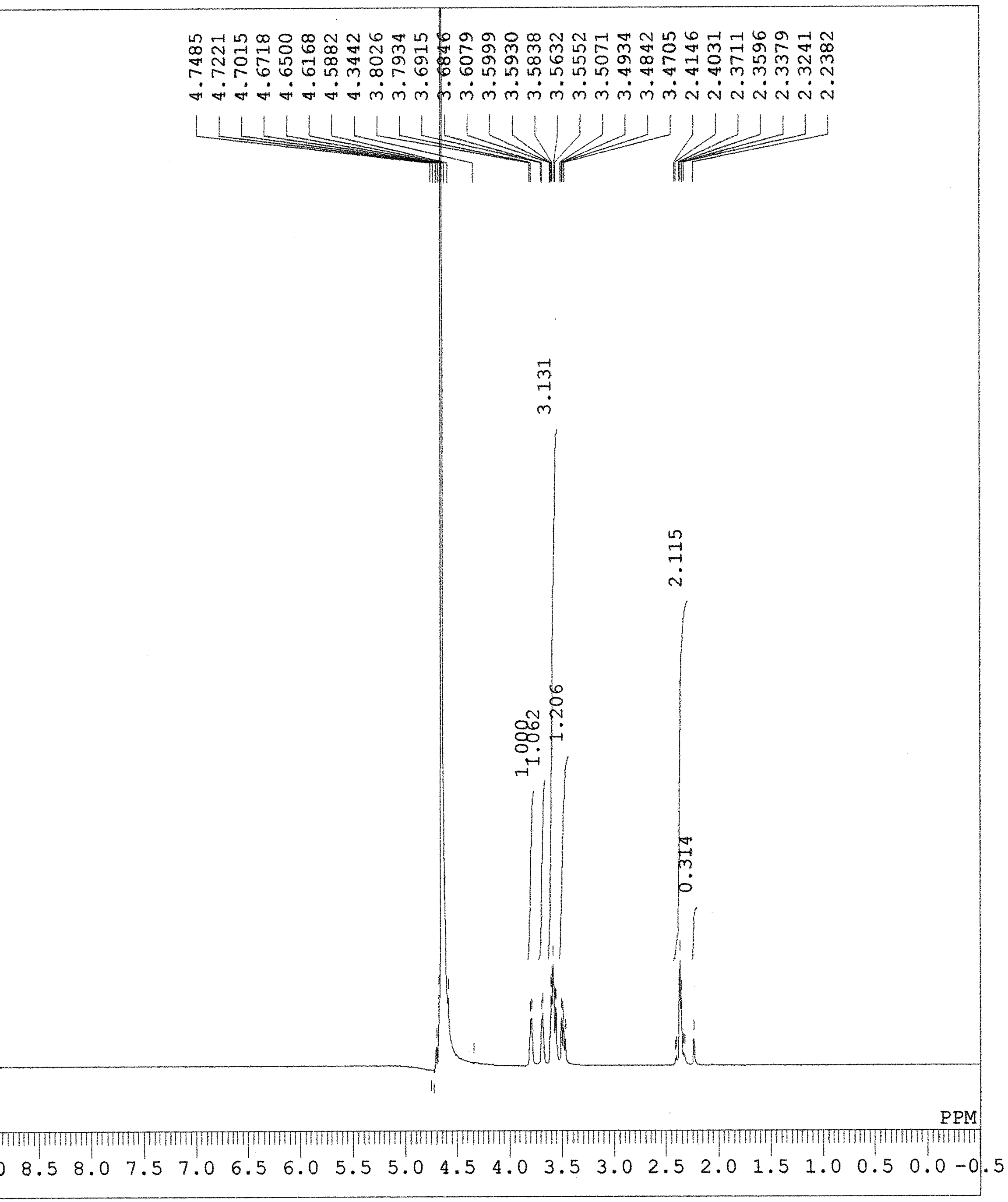
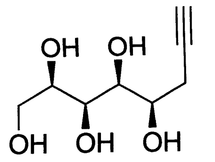


DFILE	wei r-skp xylose new-1-1.jdf
COMNT	
DATIM	2015-09-08 23:09:05
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16384
FREQU	9384.38 Hz
SCANS	10
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.3 c
SLVNT	D2O
EXREF	4.65 ppm
BF	1.20 Hz
RGAIN	34

\\172.20.1.201\users\delta\Documents\JEOL\data\wei r-skp xylose new carb-1-1.jdf



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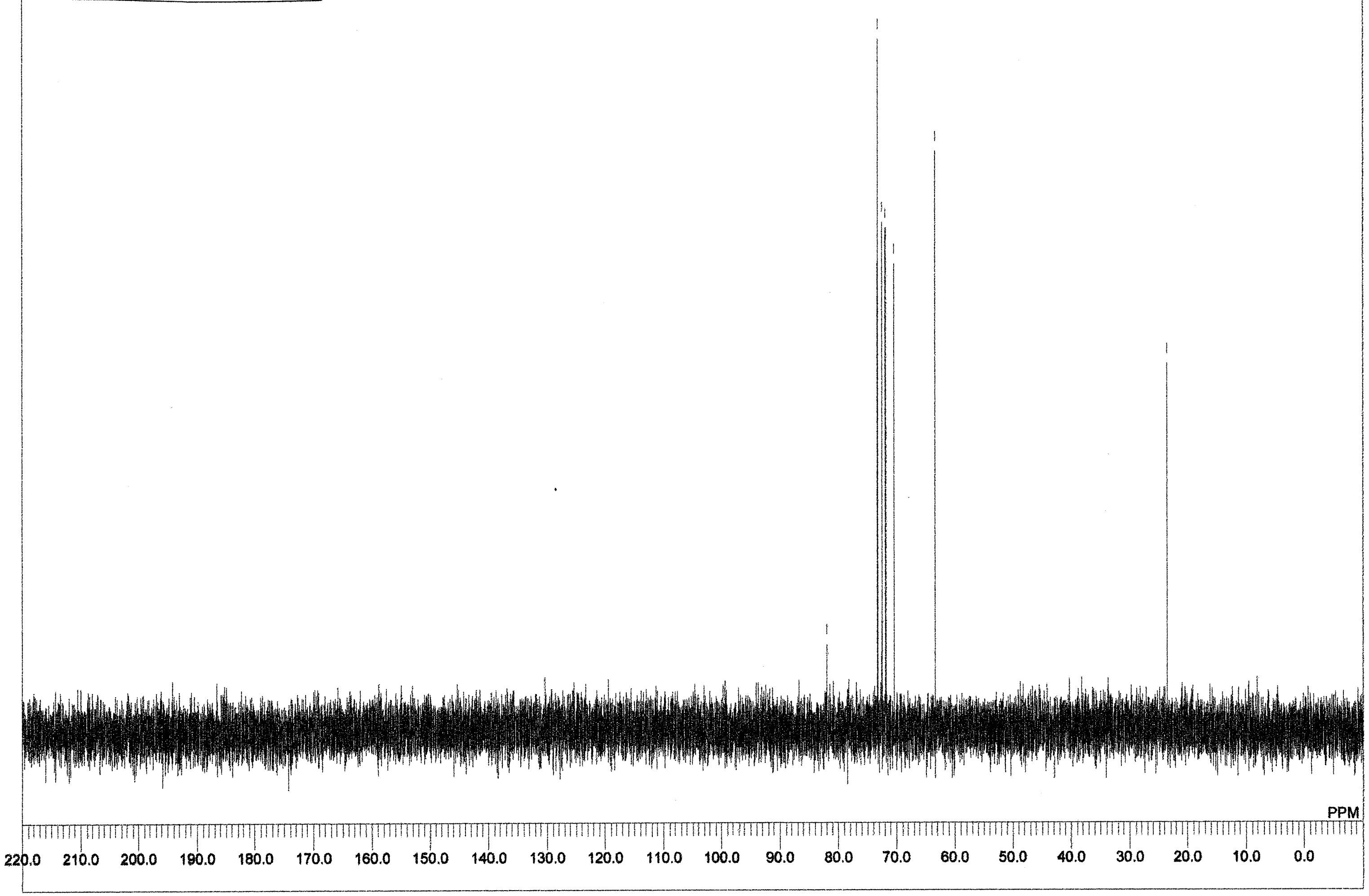
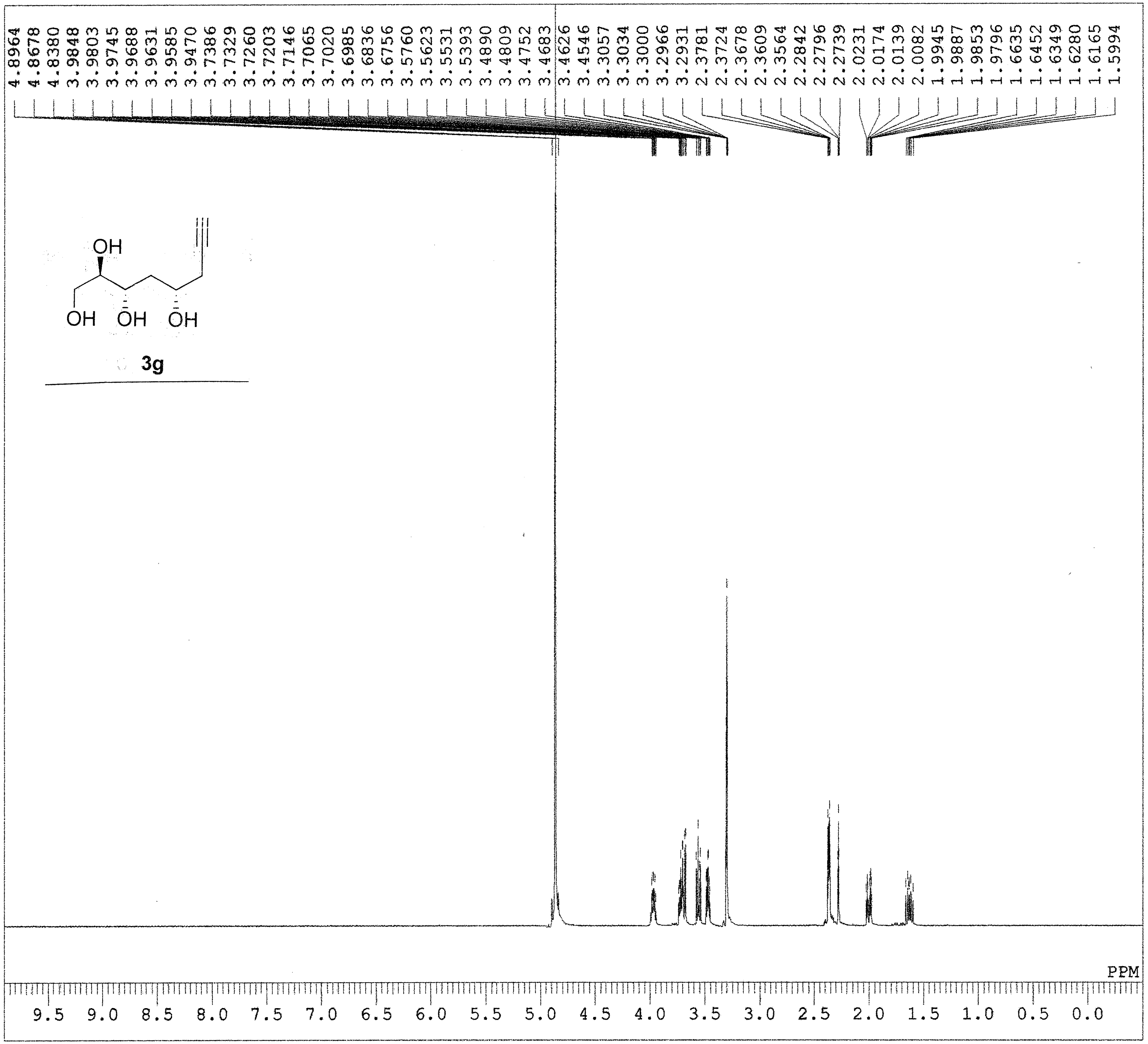
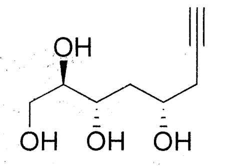


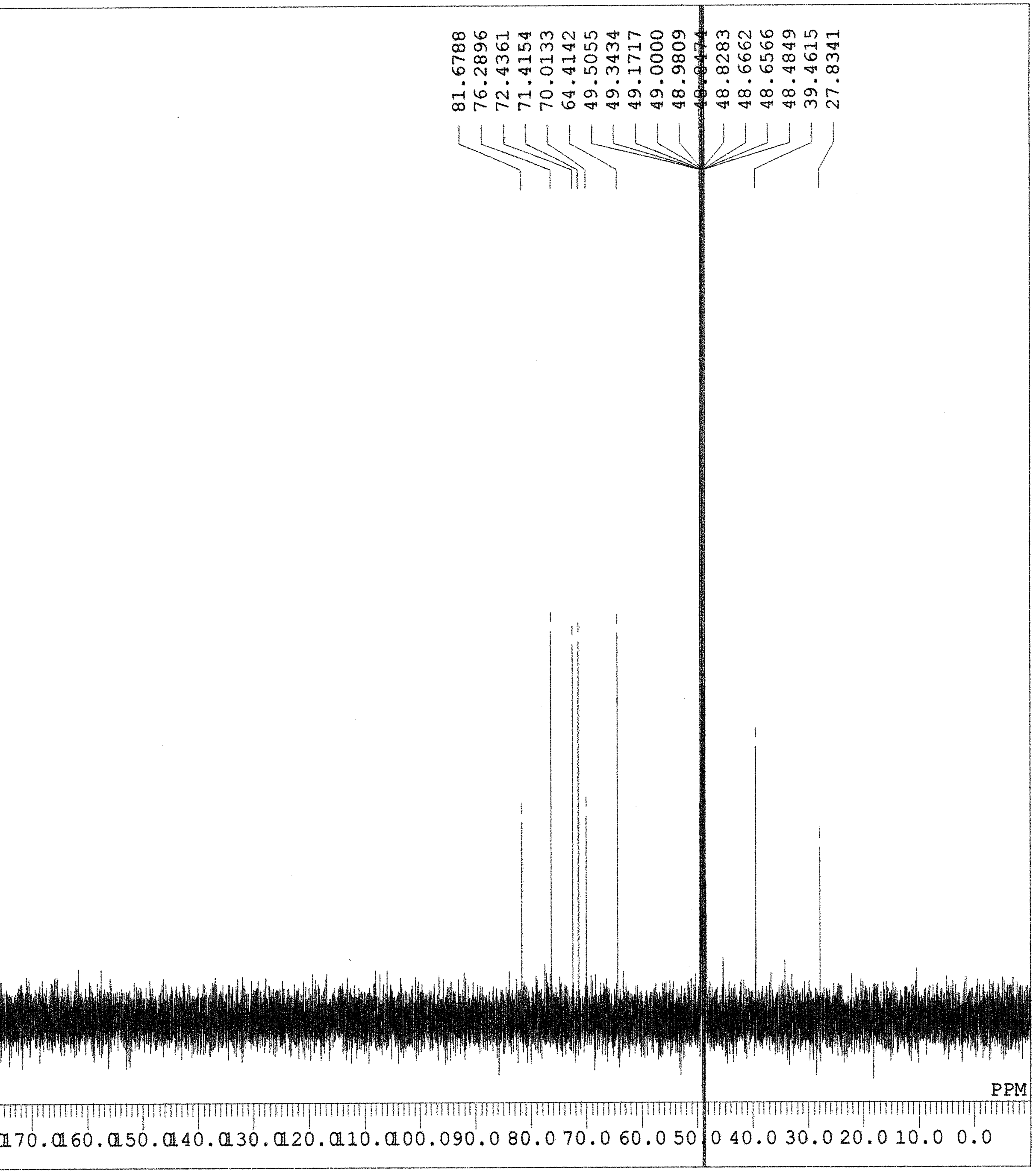
DFILE	wei r-skp xylose new carb-1-1.jdf
COMNT	
DATIM	2015-09-08 23:11:04
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32767
FREQU	39308.18 Hz
SCANS	12000
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	21.3 c
SLVNT	D2O
EXREF	49.50 ppm
BF	0.12 Hz
RGAIN	60



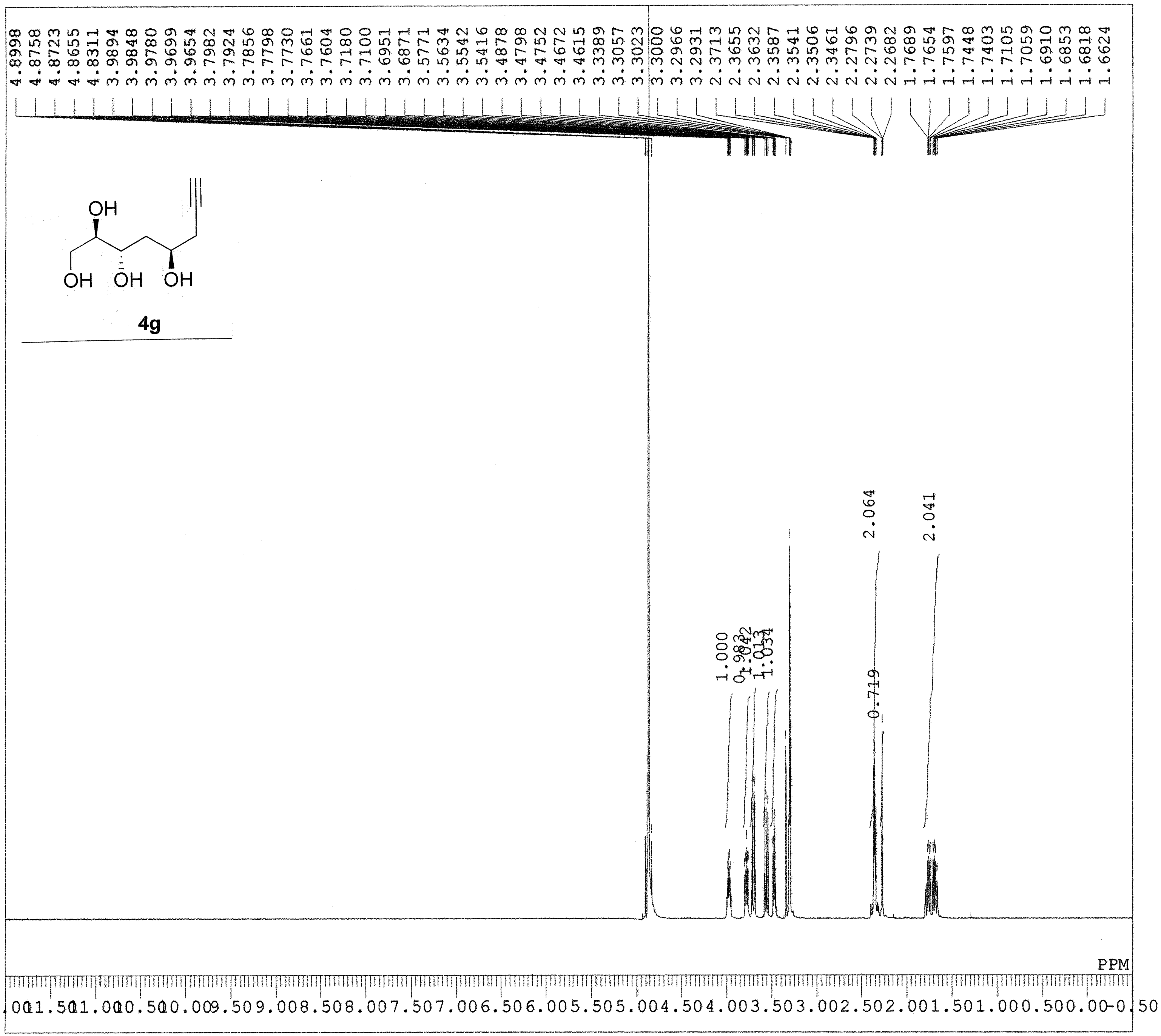
DFILE	3g s-skp 2-d-ribose alkyne-1-
COMNT	
DATIM	2015-07-18 14:40:56
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	10
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.2 c
SLVNT	CD3OD
EXREF	3.30 ppm
BF	0.12 Hz
RGAIN	32



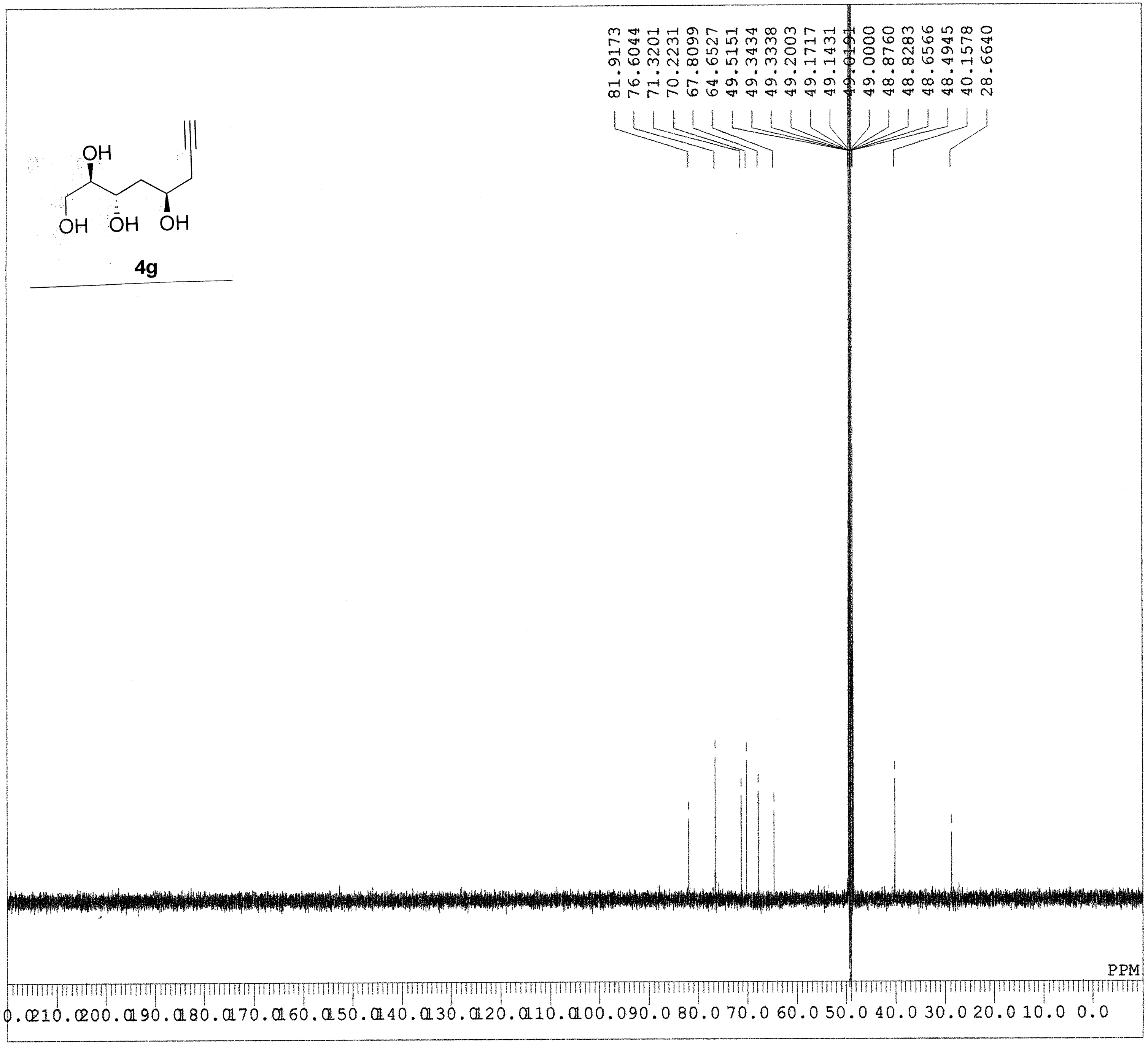
3g



DFILE	3g s-skp 2-d-ribose alkyne ca
COMNT	
DATIM	2015-07-18 14:42:39
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.54 Hz
SCANS	424
ACQTM	0.0000 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	21.7 c
SLVNT	CD3OD
EXREF	49.00 ppm
BF	0.12 Hz
RGAIN	60



DFILE	4g r-skp 2-d-ribose-1-1.jdf
COMNT	
DATIM	2015-07-17 18:10:32
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16384
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.3 c
SLVNT	CD3OD
EXREF	3.30 ppm
BF	0.12 Hz
RGAIN	30



DFILE	4g r-skp 2-d-ribose carb-1-1.a
COMNT	
DATIM	2015-07-17 18:12:02
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.54 Hz
SCANS	668
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	21.7 c
SLVNT	CD3OD
EXREF	49.00 ppm
BF	0.12 Hz
RGAIN	60

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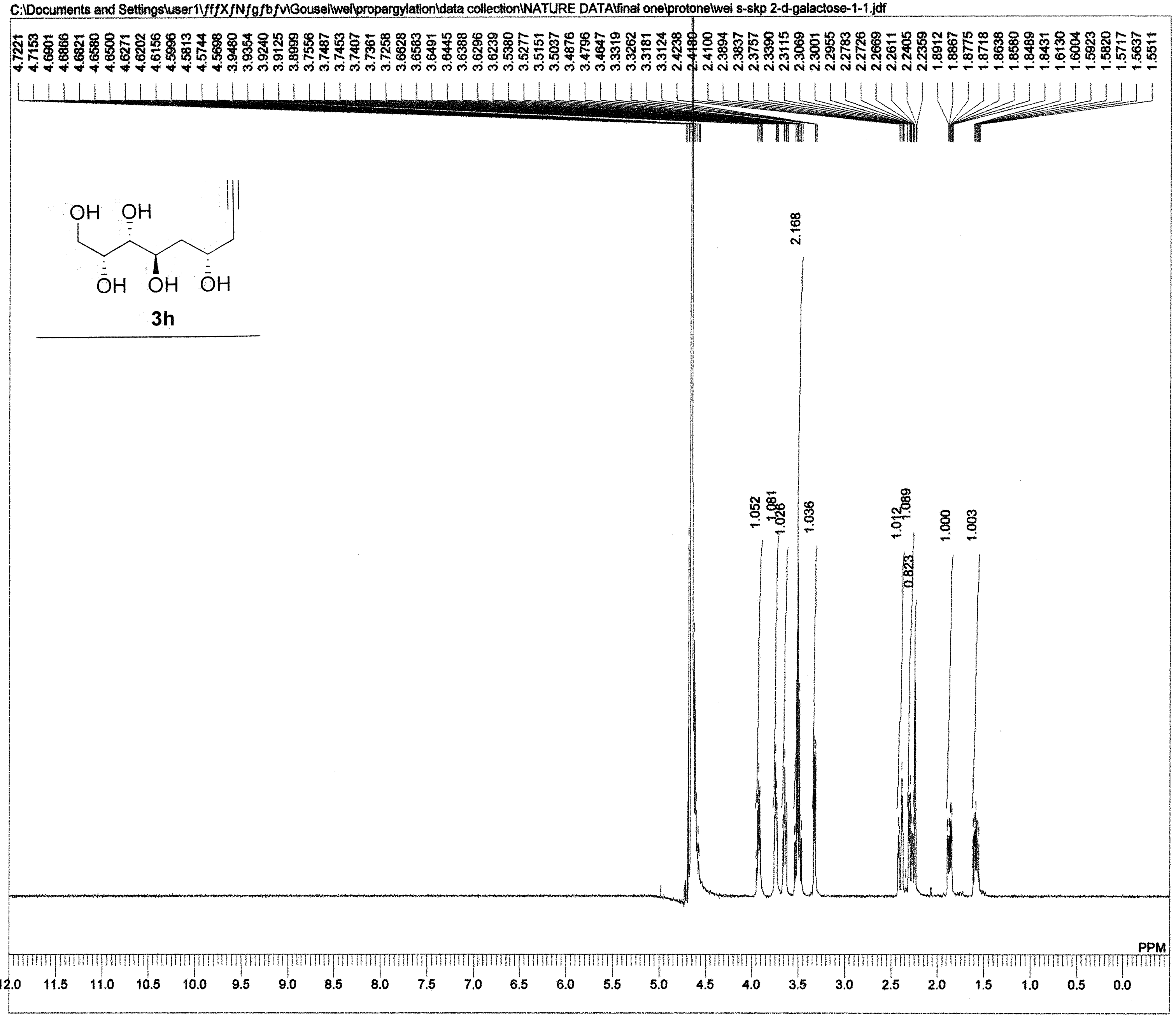
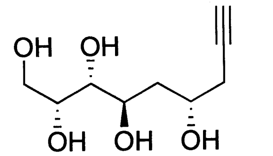
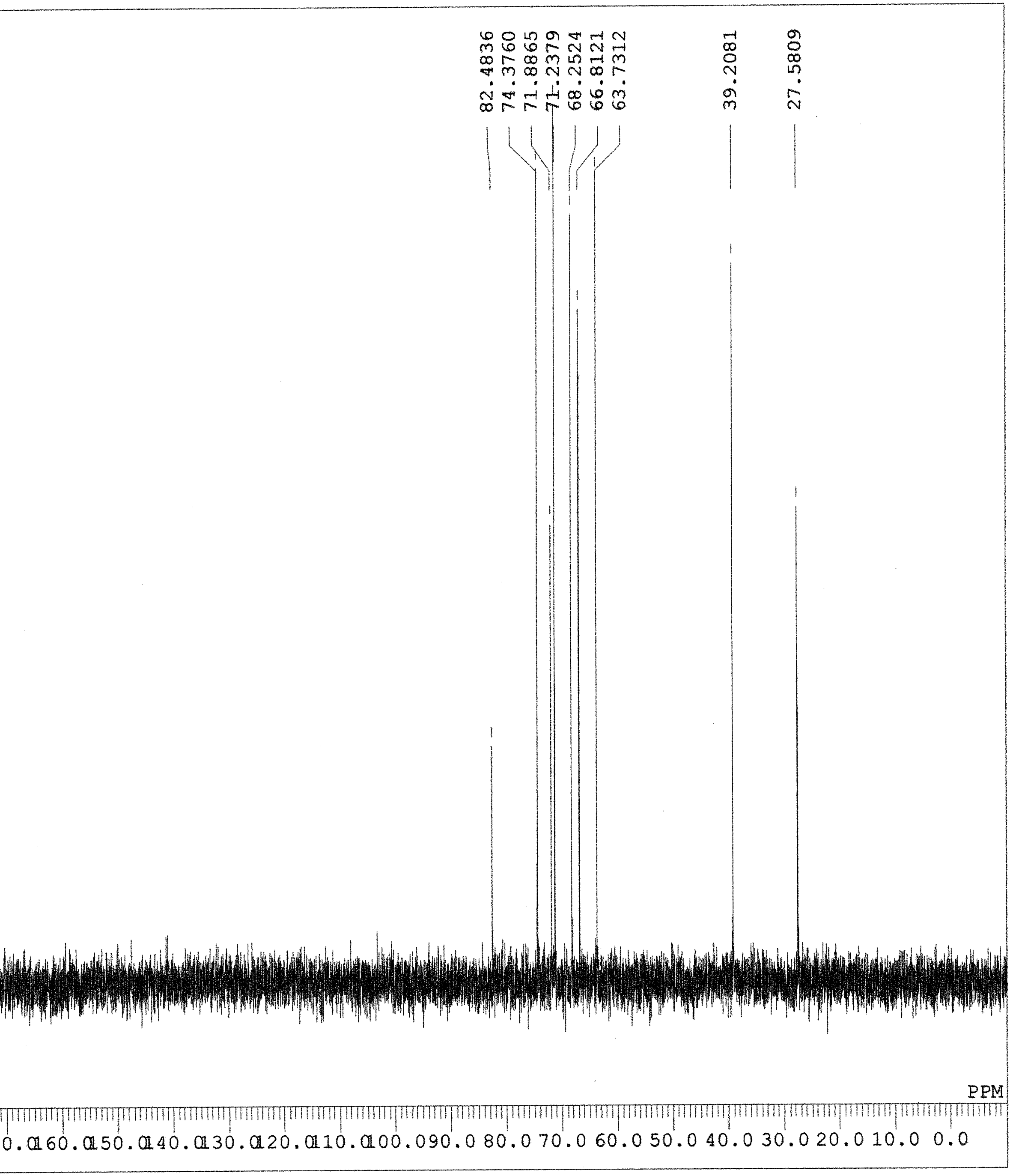


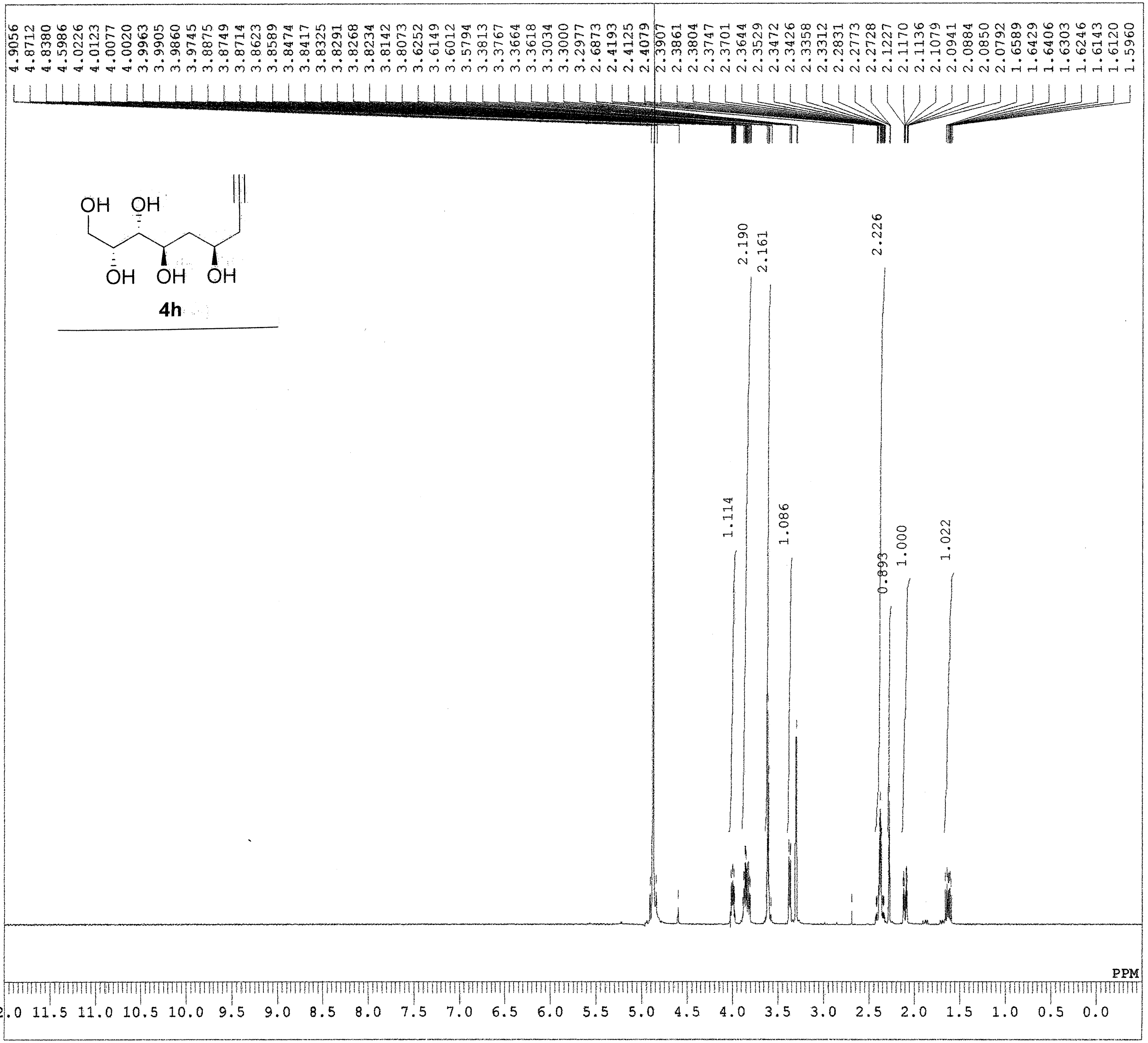
DFILE	wei s-skp 2-d-galactose-1-1.jdf
COMNT	
DATIM	2015-07-28 08:57:21
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16384
FREQU	9384.38 Hz
SCANS	6
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.2 c
SLVNT	D2O
EXREF	4.65 ppm
BF	0.12 Hz
RGAIN	30



3h



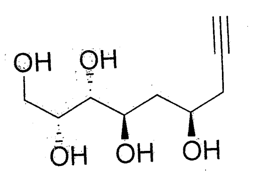
DFILE	wei s-skp 2-d-galactose carb-
COMNT	
DATIM	2015-09-09 21:18:11
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32767
FREQU	39308.18 Hz
SCANS	1533
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	20.7 c
SLVNT	D2O
EXREF	49.50 ppm
BF	1.20 Hz
RGAIN	60



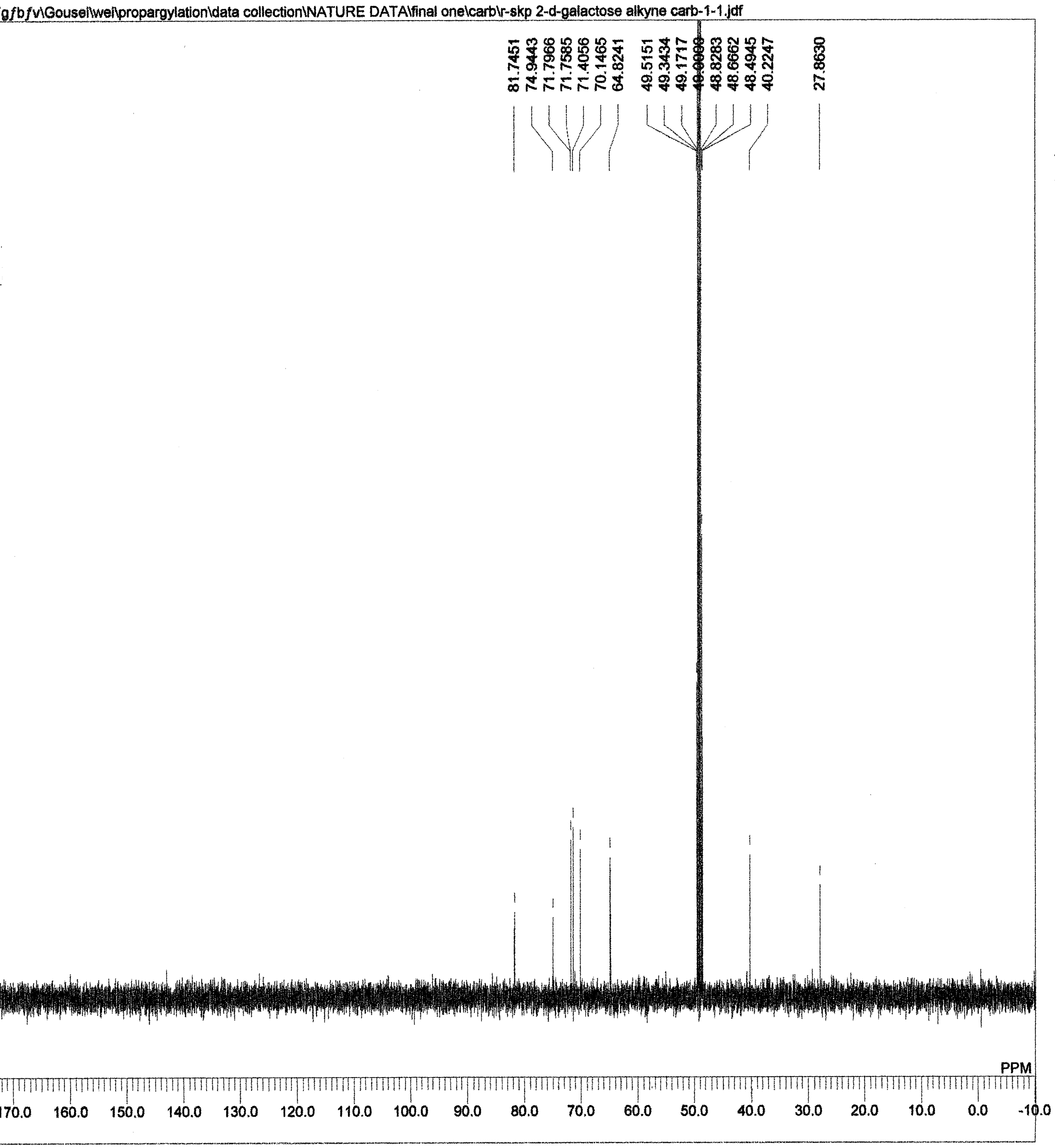
DFILE	r-skp 2-d-galactose alkyne-1-1.jdf
COMNT	
DATIM	2015-07-27 10:20:13
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16384
FREQU	9384.38 Hz
SCANS	11
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.0 c
SLVNT	CD3OD
EXREF	3.30 ppm
BF	0.12 Hz
RGAIN	30

42

DFILE	r-skp 2-d-galactose alkyne carb-1-1.jdf
COMNT	
DATIM	2015-07-27 10:22:03
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32767
FREQU	39308.18 Hz
SCANS	164
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	21.6 c
SLVNT	CD3OD
EXREF	49.00 ppm
BF	0.12 Hz
RGAIN	60



4h



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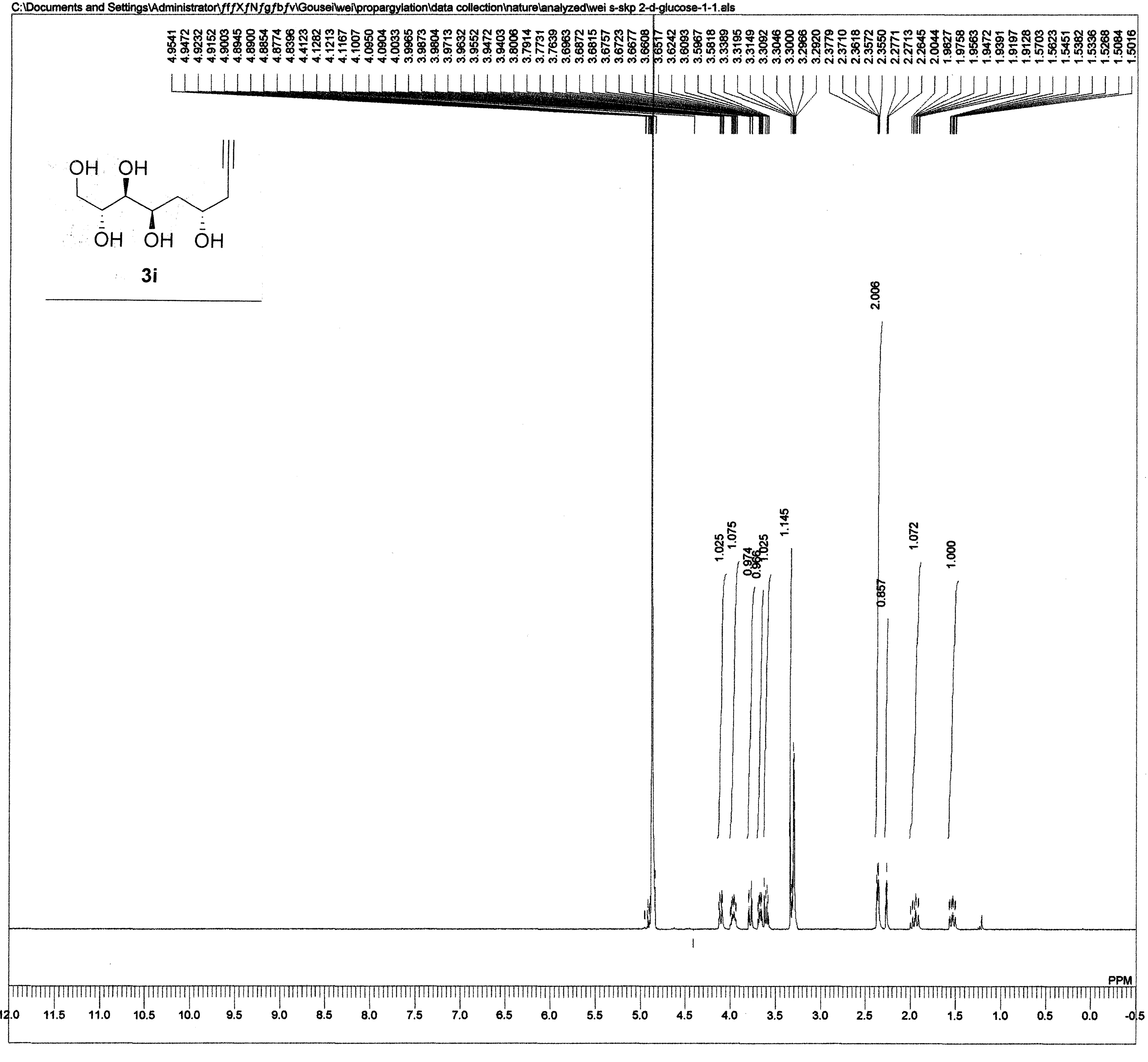

DFILE	wei s-skp 2-d-glucose-1-1.als
COMNT	
DATIM	21-08-2015 20:28:40
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5882.35 Hz
SCANS	5
ACQTM	2.2282 sec
PD	5.0000 sec
PW1	4.99 usec
IRNUC	1H
CTEMP	22.4 c
SLVNT	CD3OD
EXREF	3.30 ppm
BF	0.12 Hz
RGAIN	38

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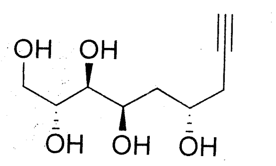

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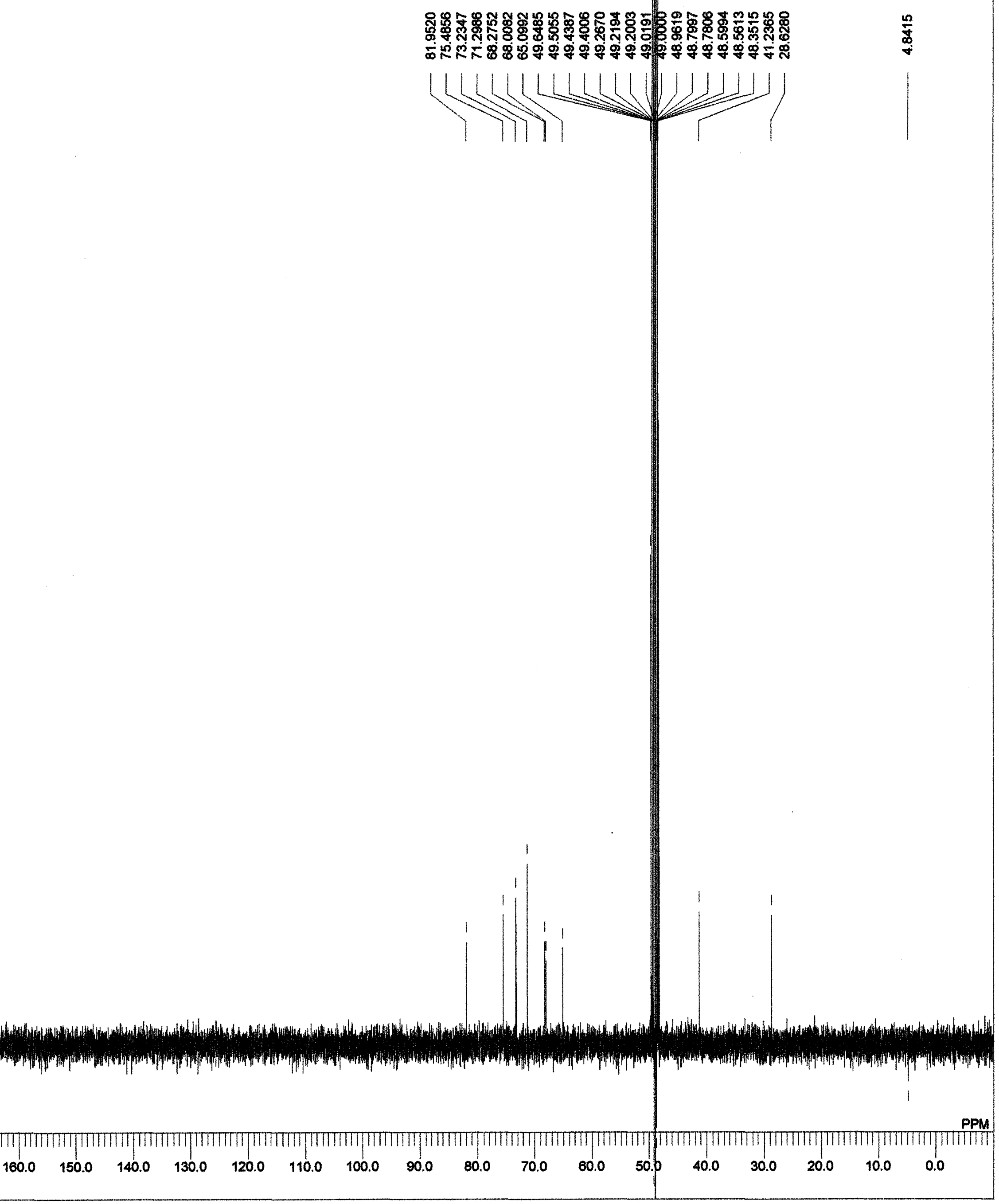
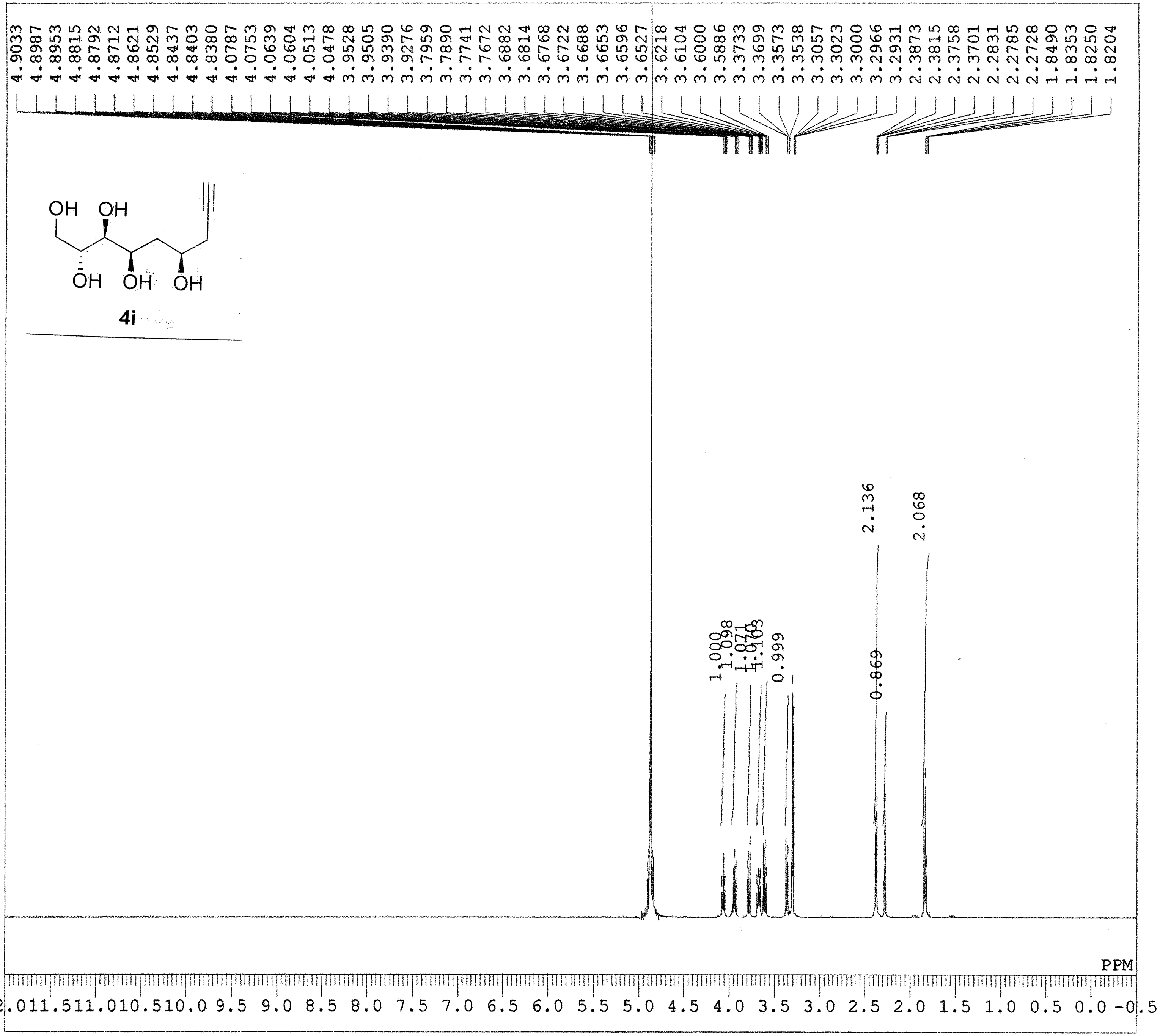
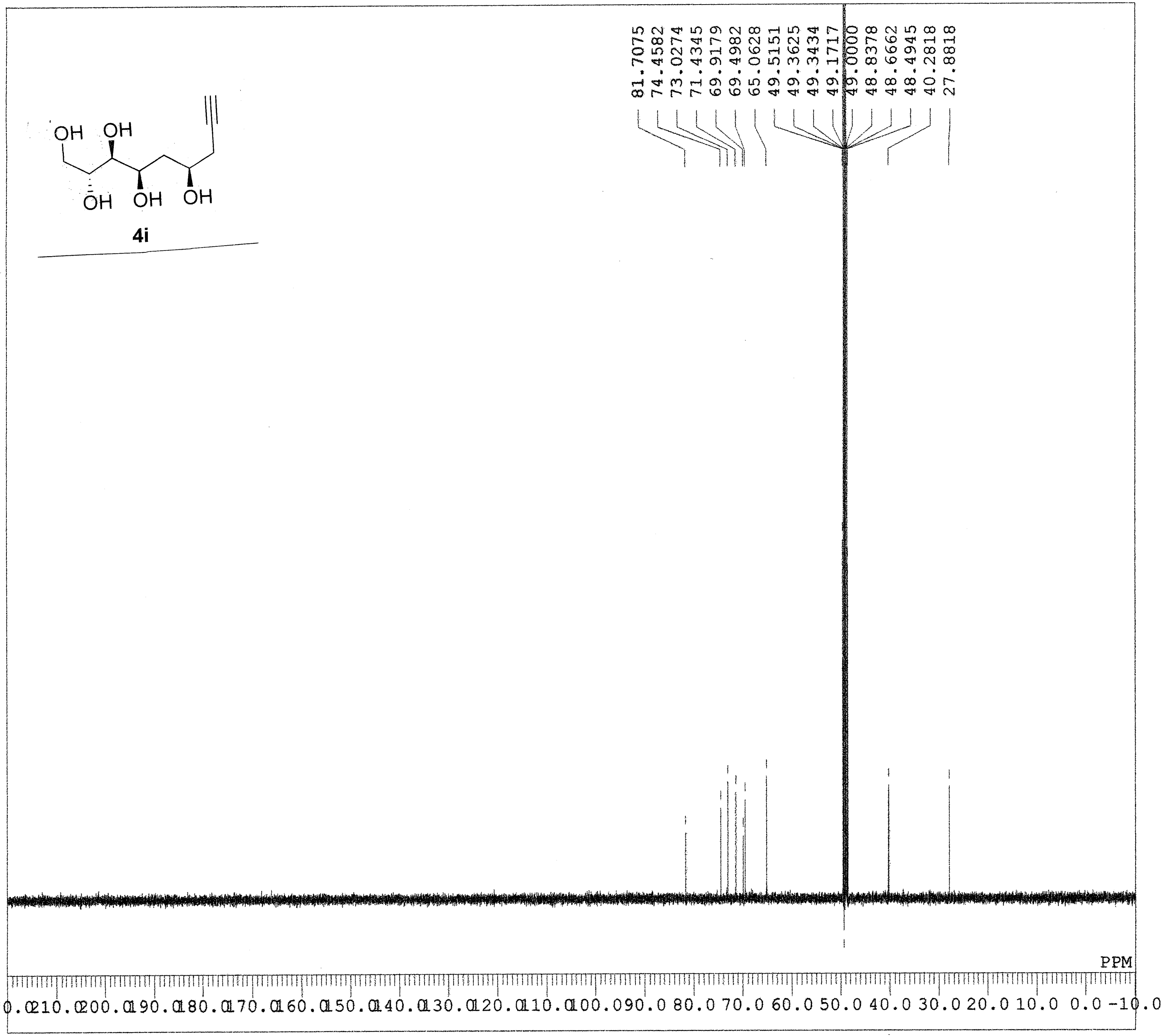


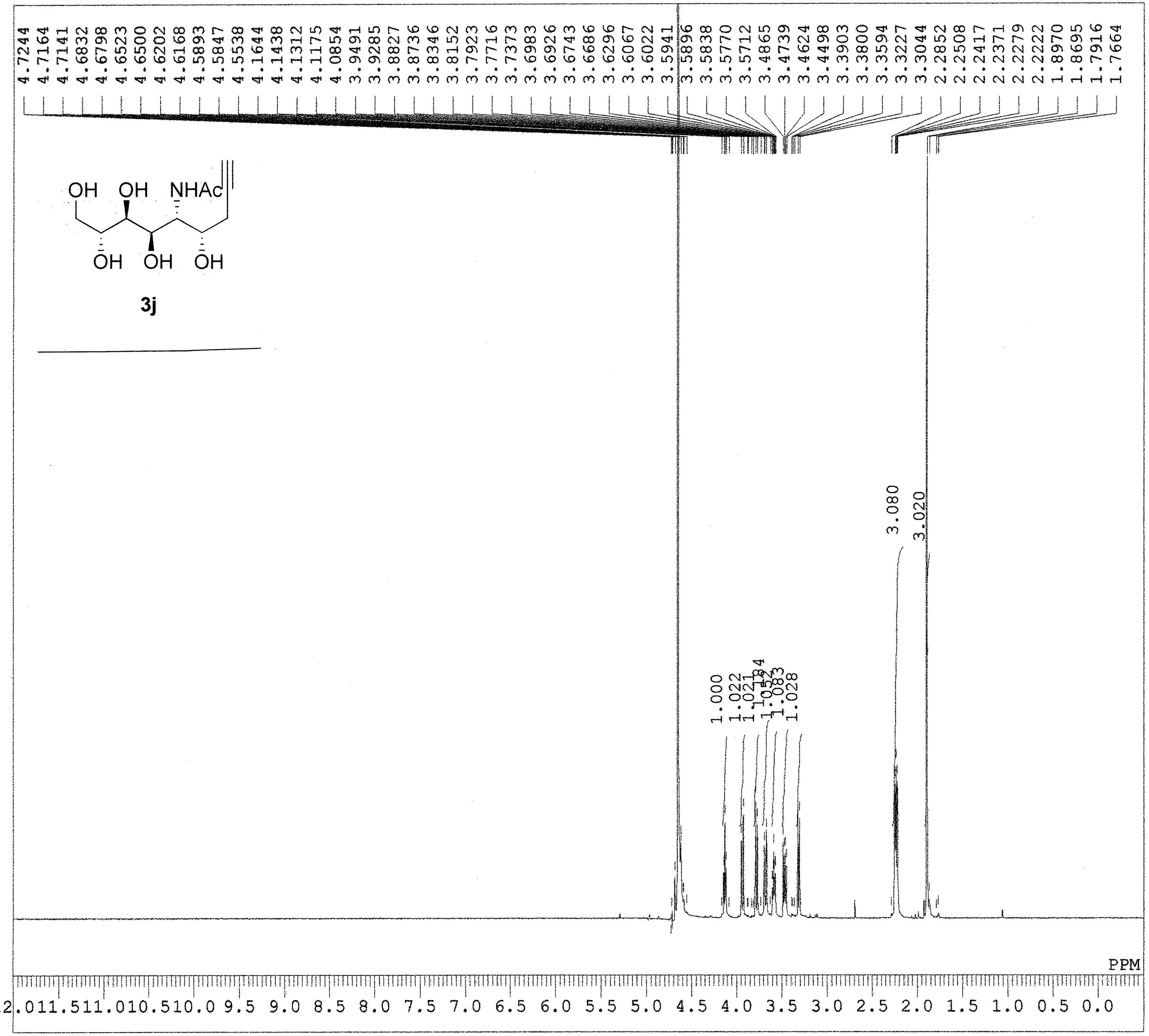
DFILE	wei s-2-d-glucose carb-1-1.jdf
COMNT	
DATIM	21-08-2015 20:37:44
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	32767
FREQU	30788.18 Hz
SCANS	1056
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	3.16 usec
IRNUC	1H
CTEMP	22.6 c
SLVNT	CD3OD
EXREF	49.00 ppm
BF	0.12 Hz
RGAIN	60



DFILE	4i r-skp 2-d-glu alkyne.als
COMNT	
DATIM	2015-07-17 21:39:37
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	13107
FREQU	7507.51 Hz
SCANS	4
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.2 c
SLVNT	CD3OD
EXREF	3.30 ppm
BF	0.12 Hz
RGAIN	30

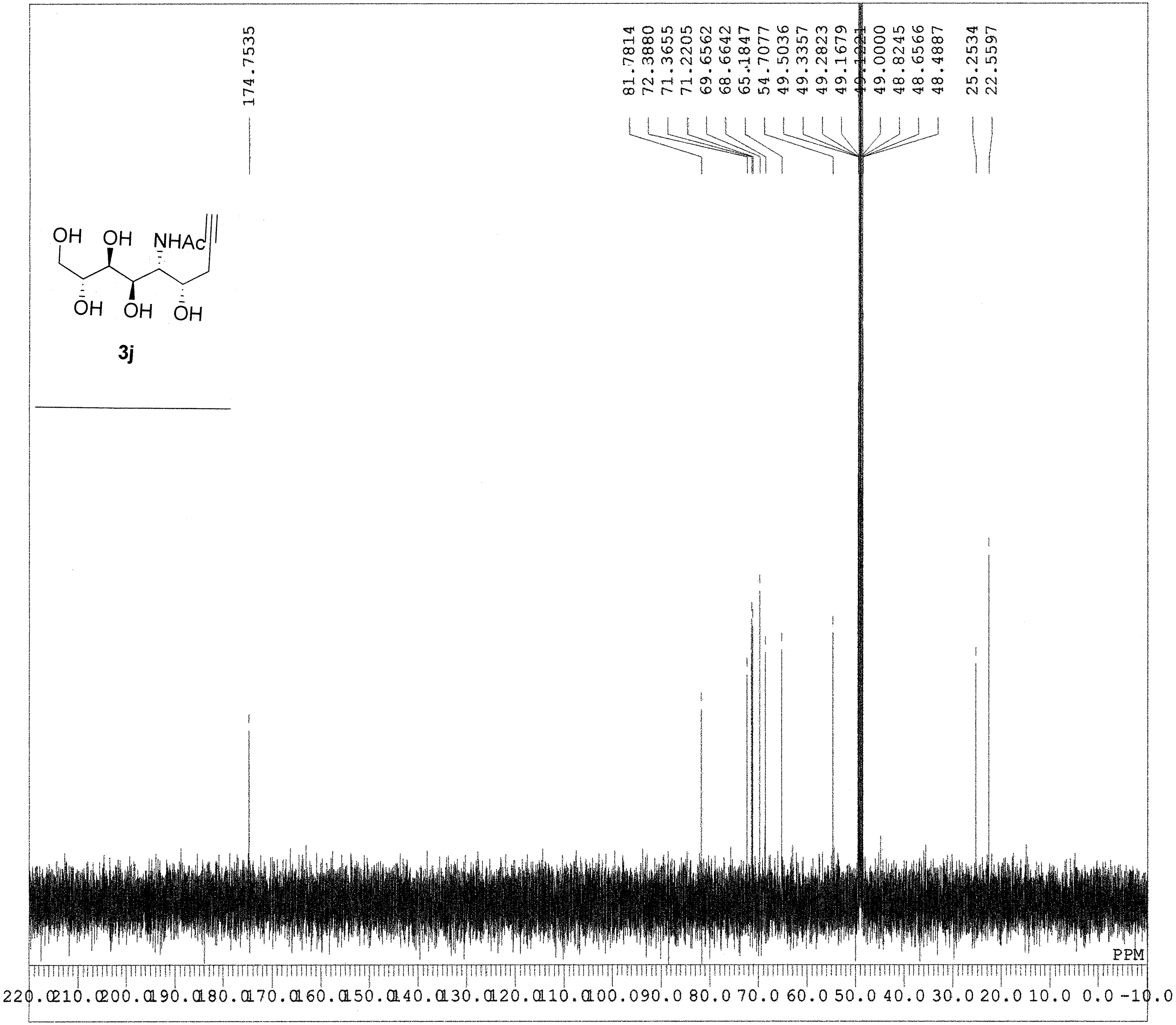


DFILE	4i r-skp 2-d-glu carb-1-1.als
COMNT	
DATIM	2015-07-17 21:40:57
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.54 Hz
SCANS	841
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	21.9 c
SLVNT	CD3OD
EXREF	49.00 ppm
BF	0.12 Hz
RGAIN	60

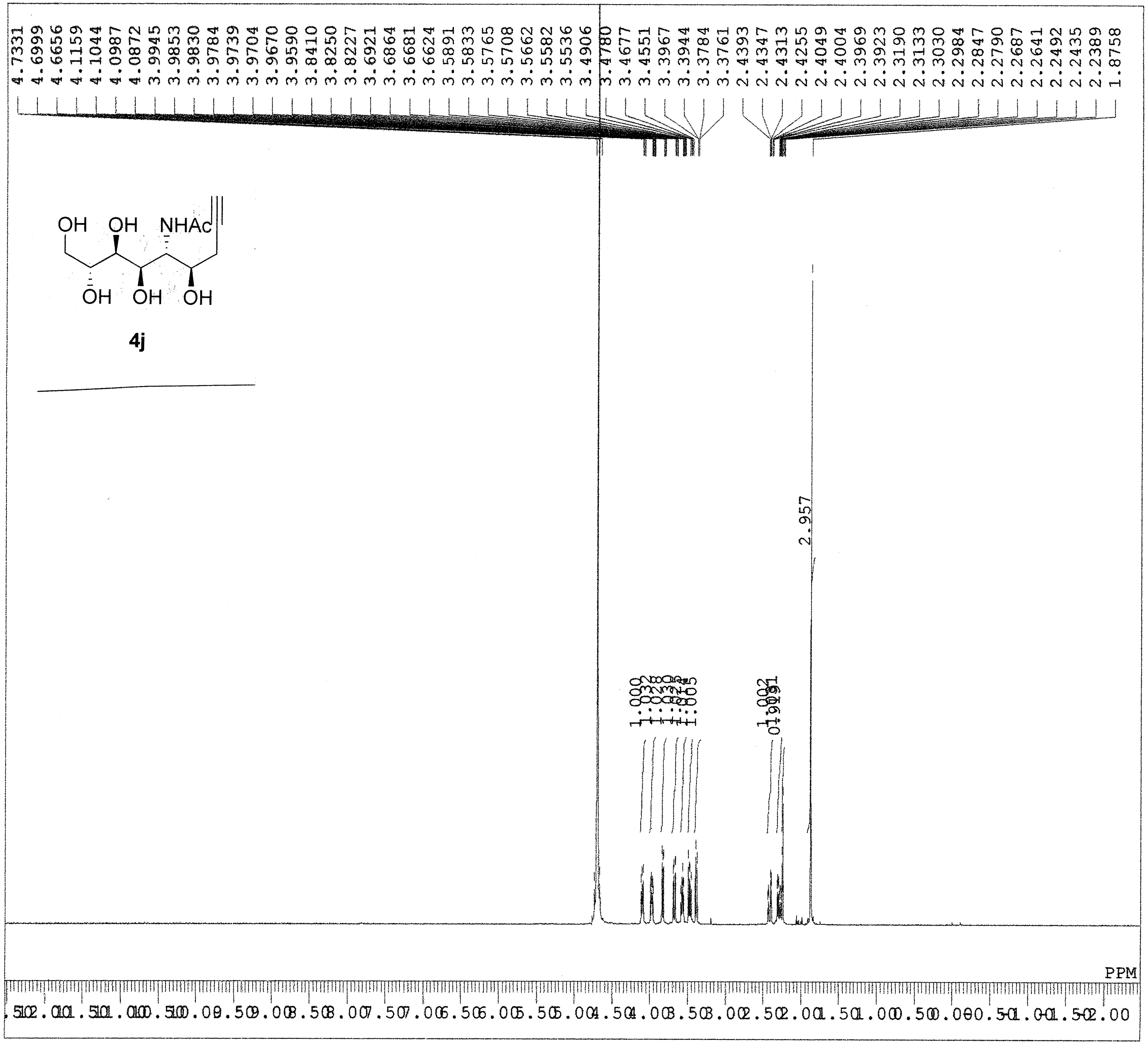


DFILE	PGF s-skp mannosamin-1-1.jdf
COMNT	
DATIM	2015-05-23 14:18:51
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16384
FREQU	9384.38 Hz
SCANS	7
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	20.7 c
SLVNT	D2O
EXREF	4.65 ppm
BF	0.12 Hz
RGAIN	30

3J



DFILE	PGF s-skp mannosamin carb-1-1
COMNT	
DATIM	2015-05-23 15:24:60
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32768
FREQU	31446.54 Hz
SCANS	310
ACQTM	1.0420 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	21.6 c
SLVNT	CD3OD
EXREF	49.00 ppm
BF	0.12 Hz
RGAIN	74



DFILE wei R-SKP mannosamin alkyne-1
COMNT
DATIM 2015-06-22 20:12:07
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 4
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 21.2 c
SLVNT D2O
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 28

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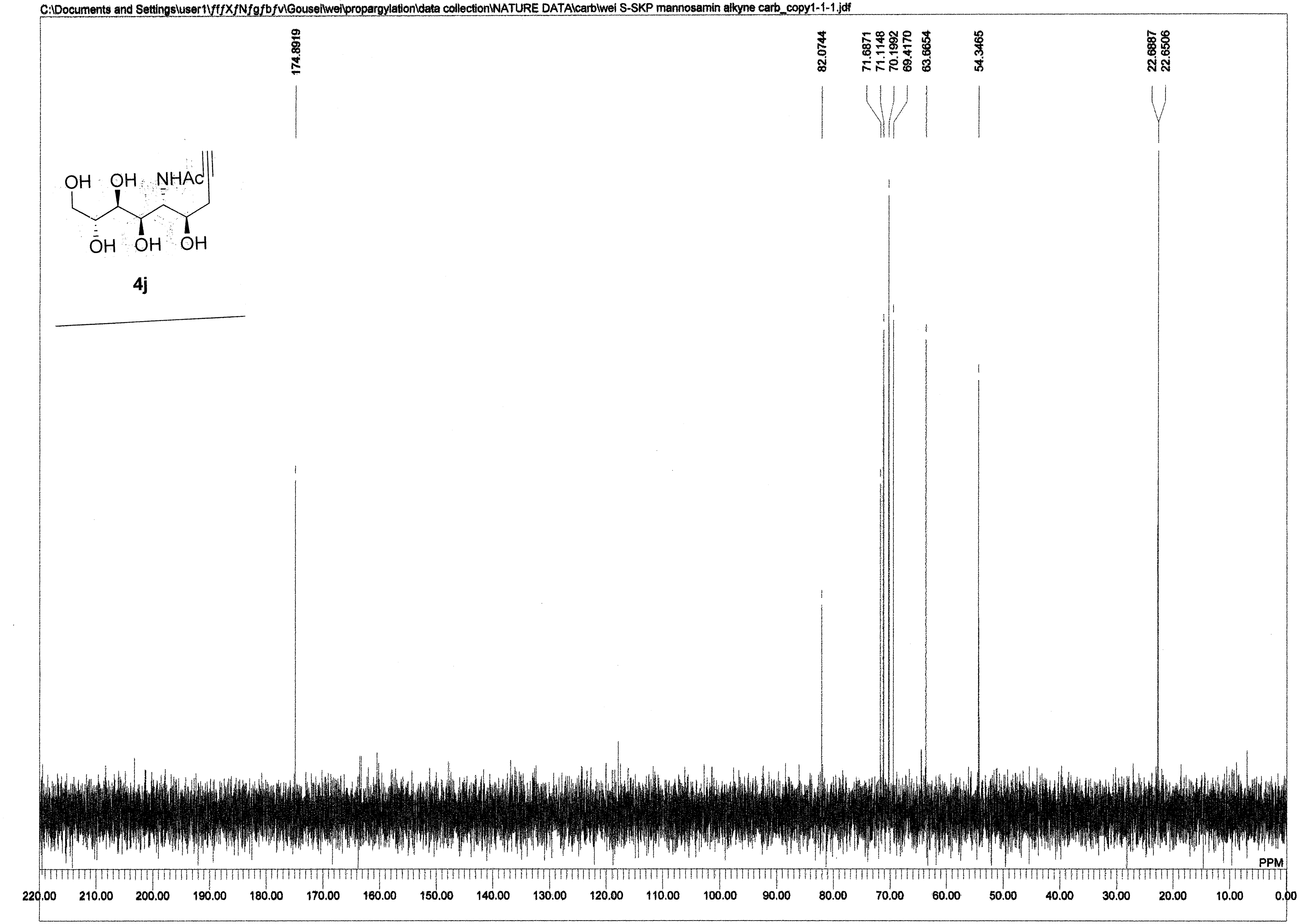
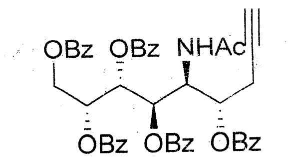
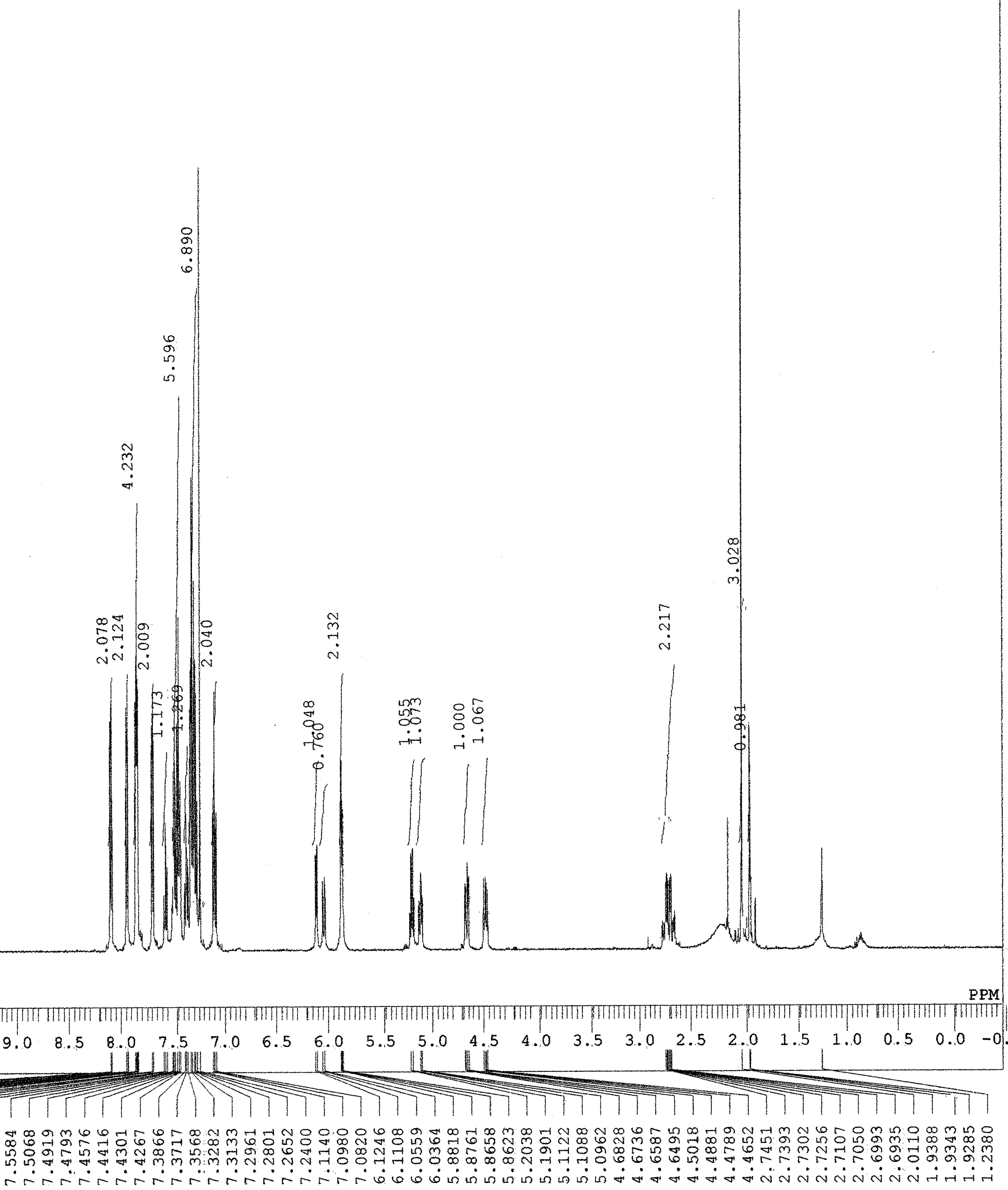


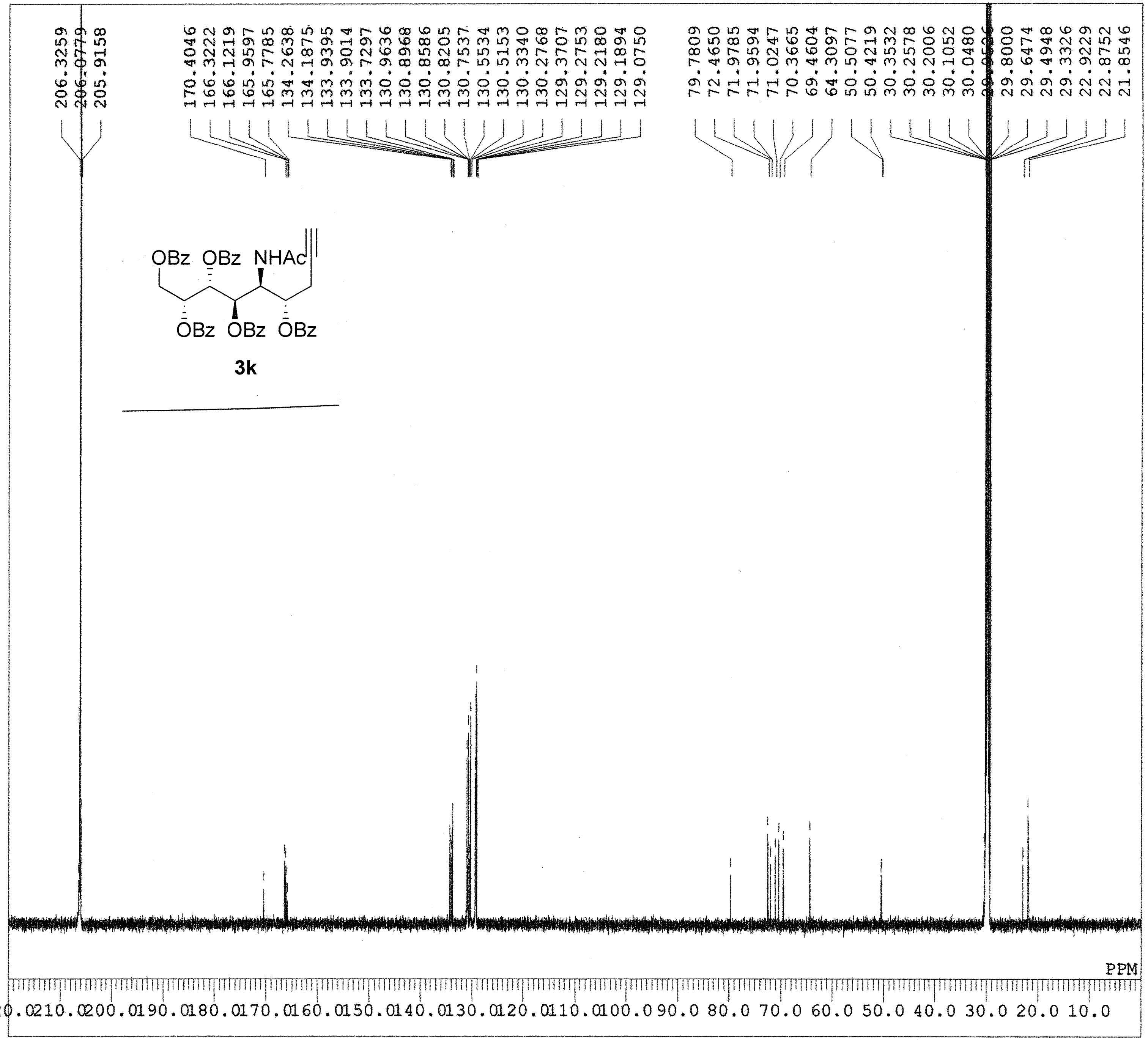
DFILE	wei S-SKP mannosamin alk
COMNT	
DATIM	2015-06-22 20:13:27
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32767
FREQU	39308.18 Hz
SCANS	170
ACQTM	0.0000 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	21.5 c
SLVNT	D2O
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60



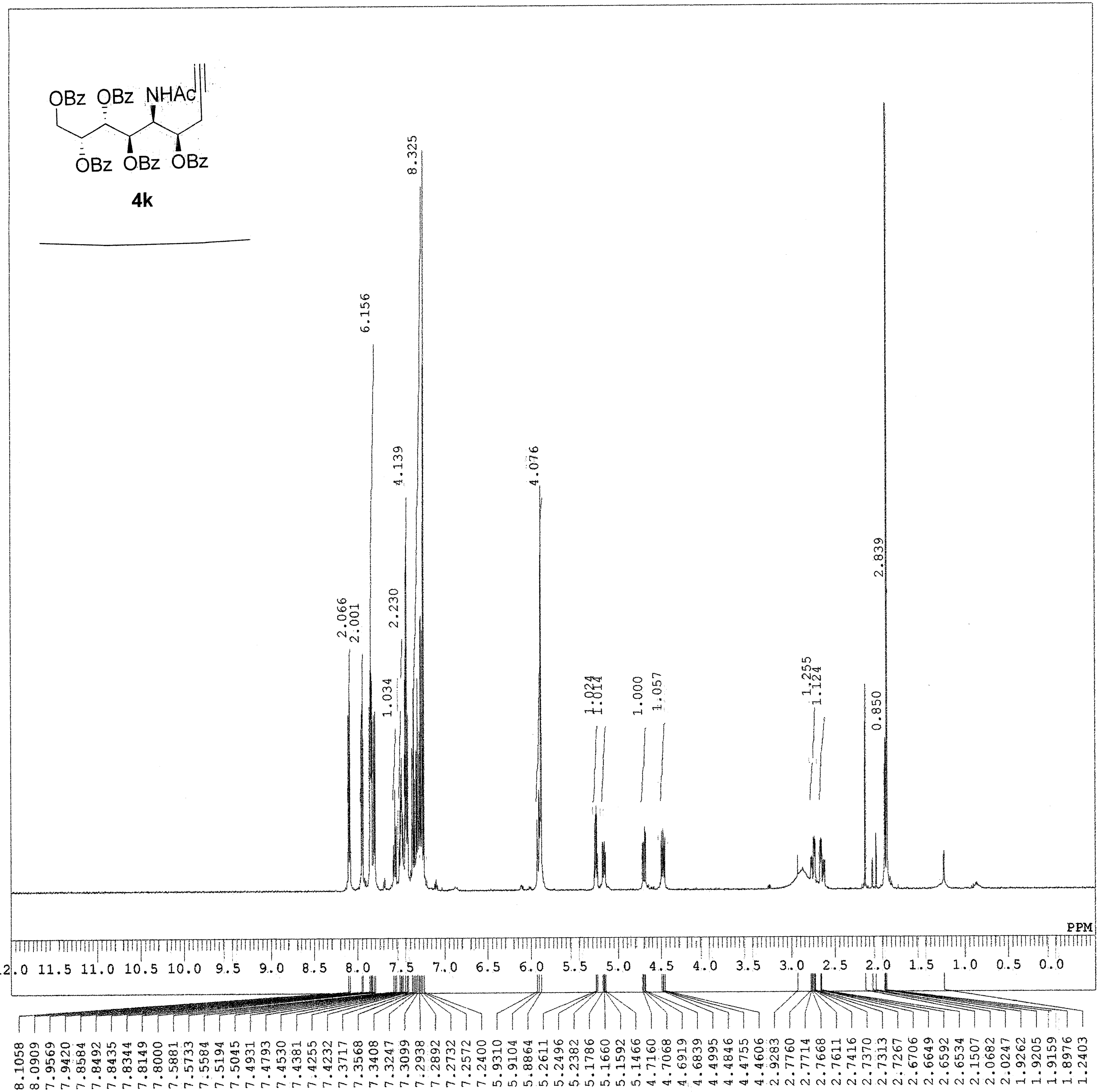
3k



DFILE wei S-SKP galactosamin-2-1.als
COMNT
DATIM 2015-04-24 08:41:38
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 28.5 c
SLVNT CDCL3
EXREF 7.24 ppm
BF 0.12 Hz
RGAIN 30



DFILE	wei S-SKP galactosamin carb-1
COMNT	
DATIM	2015-04-09 22:55:17
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32767
FREQU	39308.18 Hz
SCANS	12500
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	27.4 c
SLVNT	ACETN
EXREF	29.80 ppm
BF	0.12 Hz
RGAIN	60



DFILE wei R-SKP galactosamin-2-1.als
COMNT
DATIM 2015-04-24 08:44:49
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 12
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 28.4 c
SLVNT CDCL3
EXREF 7.24 ppm
BF 0.12 Hz
RGAIN 30

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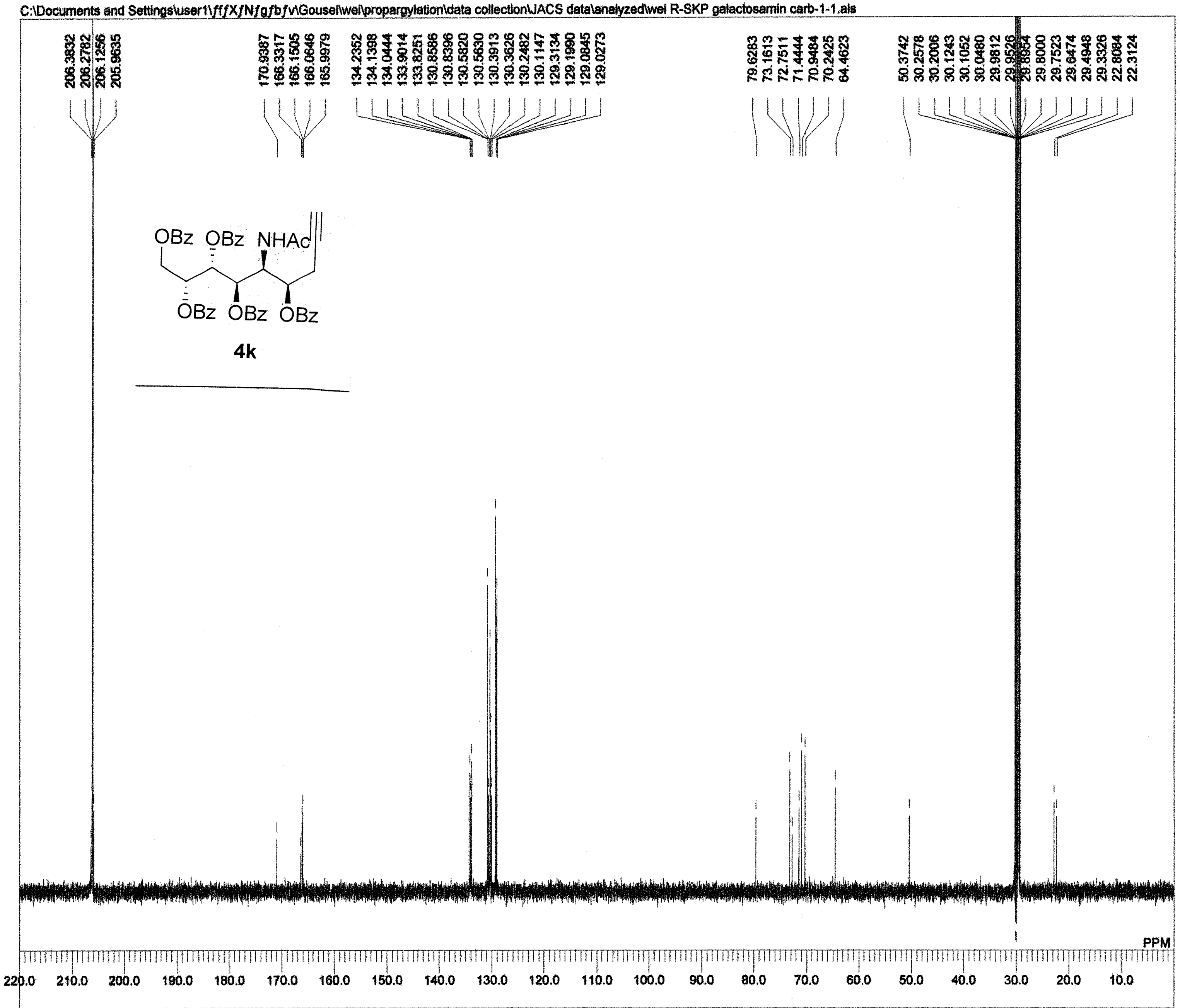
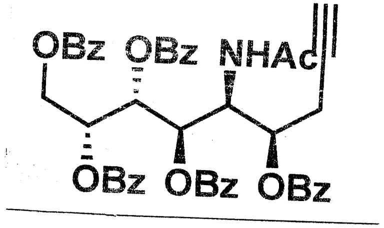
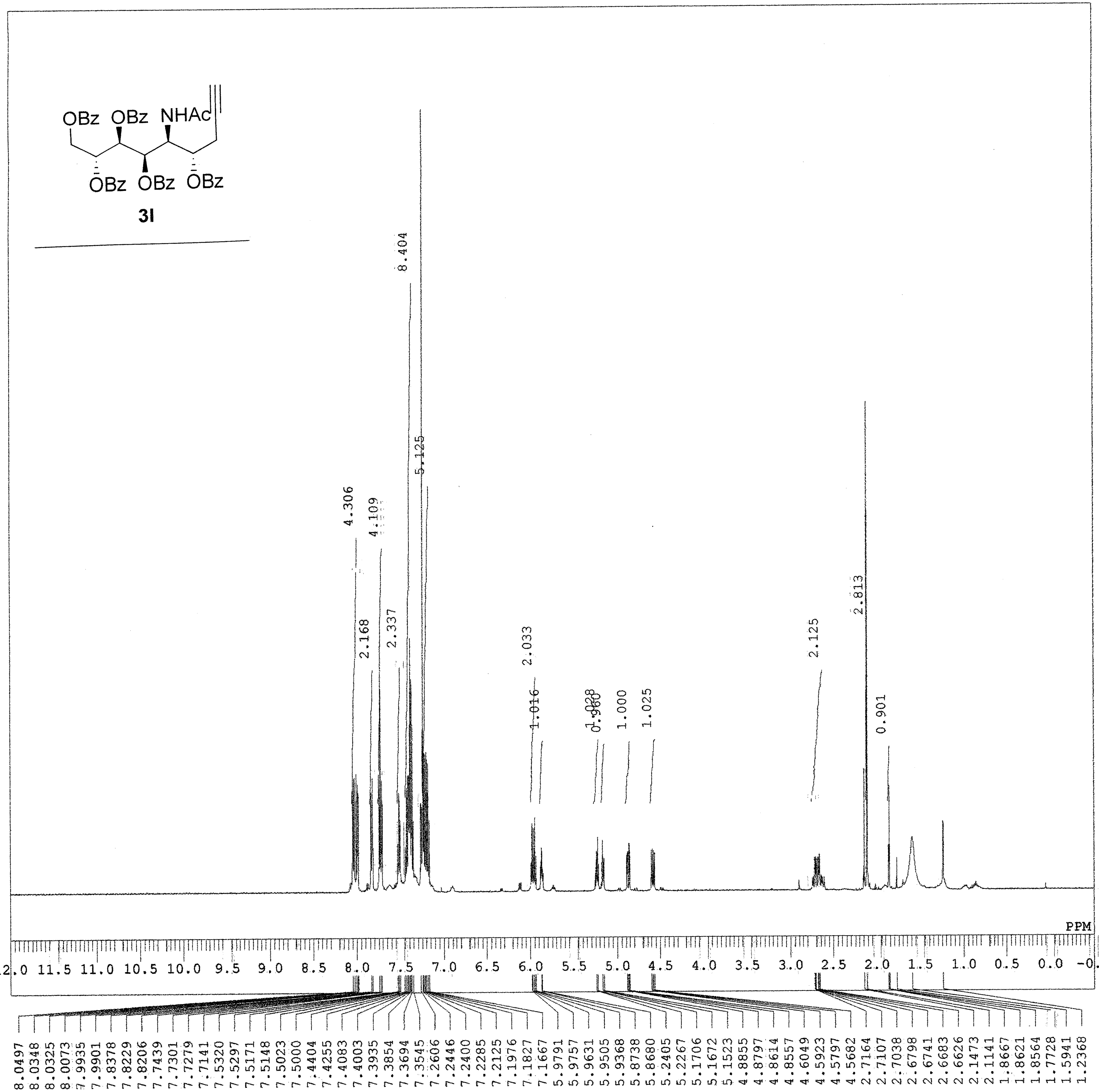


DFILE	wei R-SKP galactosamin carb-1-1.als
COMNT	
DATIM	2015-04-09 16:12:37
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32767
FREQU	39308.18 Hz
SCANS	2181
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	28.1 c
SLVNT	ACETN
EXREF	29.80 ppm
BF	0.12 Hz
RGAIN	60





DFILE wei S-SKP glucosamin-2-1.als
COMNT
DATIM 2015-04-24 08:54:44
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 32
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 28.3 c
SLVNT CDCL3
EXREF 7.24 ppm
BF 0.12 Hz
RGAIN 30

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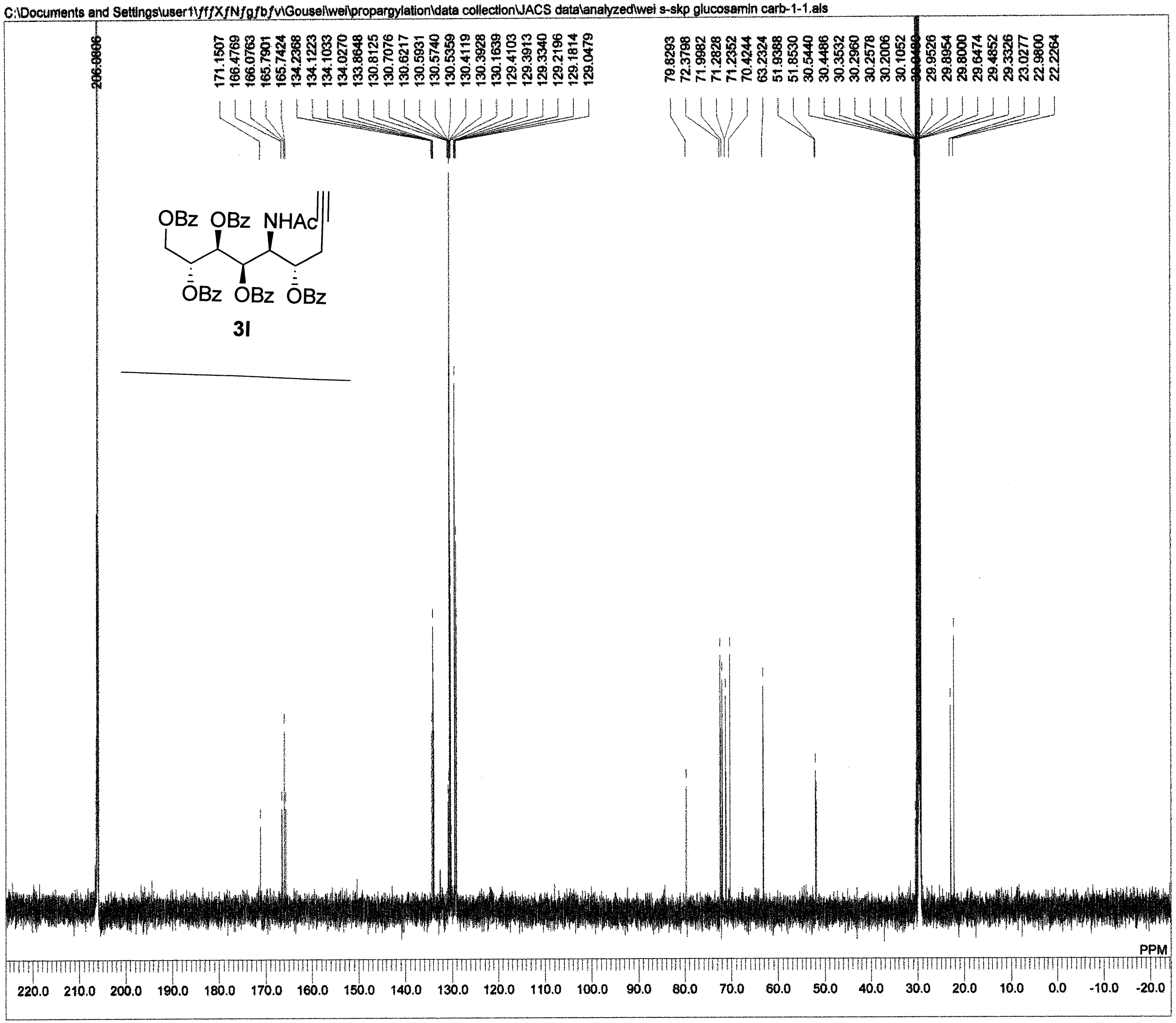
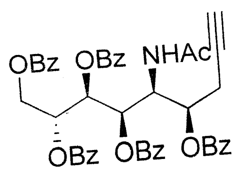
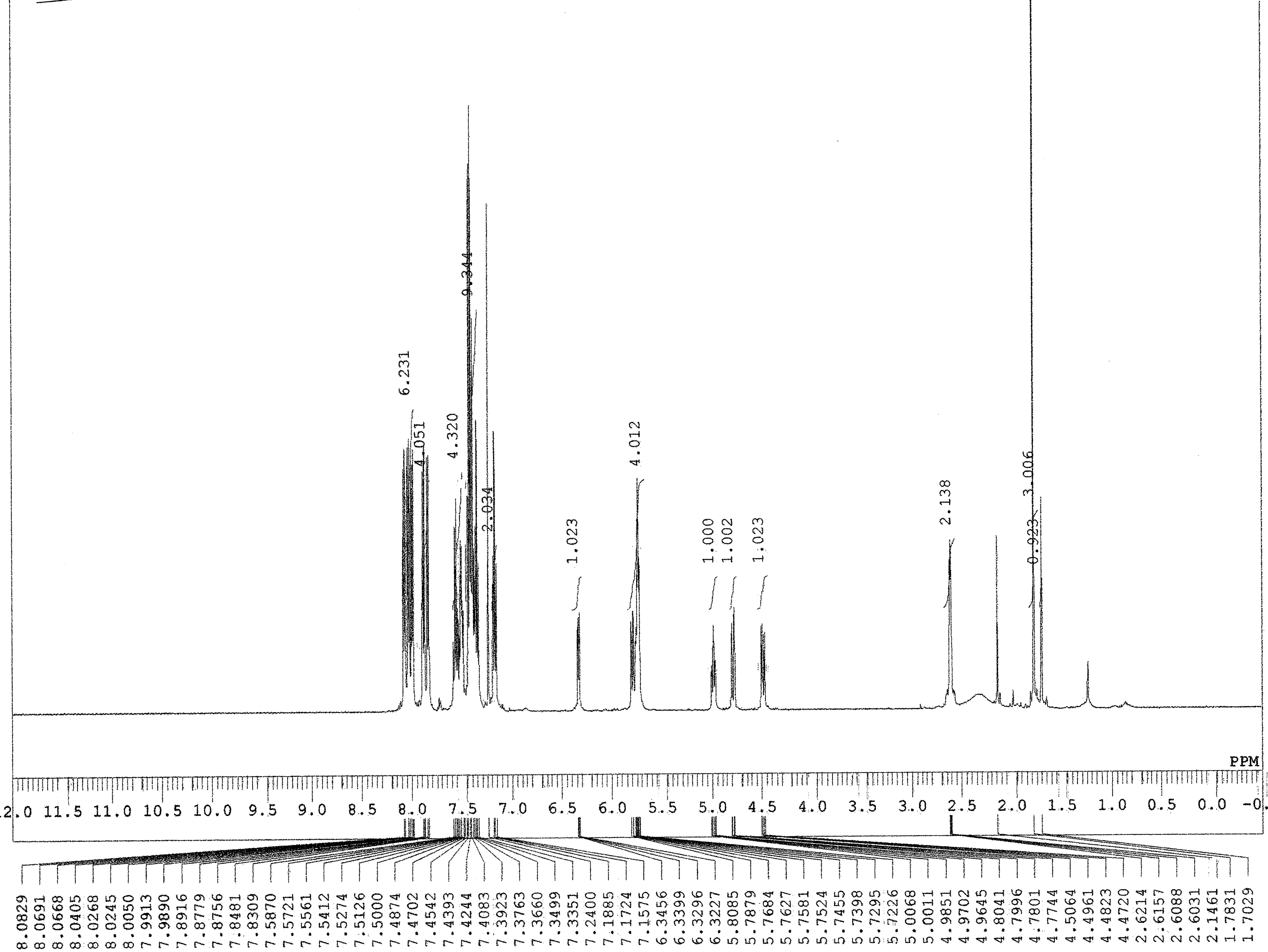


DFILE	wei s-skp glucosamin carb-1-1.als
COMNT	
DATIM	2015-04-10 23:11:28
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.54 Hz
SCANS	12500
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	27.7 c
SLVNT	ACETN
EXREF	29.80 ppm
BF	0.12 Hz
RGAIN	60



4l



DFILE wei R-SKP glucosamin-2-1.als
COMNT
DATIM 2015-04-24 09:14:10
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 17
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 28.2 c
SLVNT CDCL3
EXREF 7.24 ppm
BF 0.12 Hz
RGAIN 30

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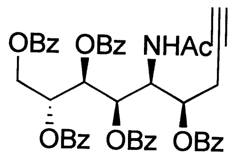
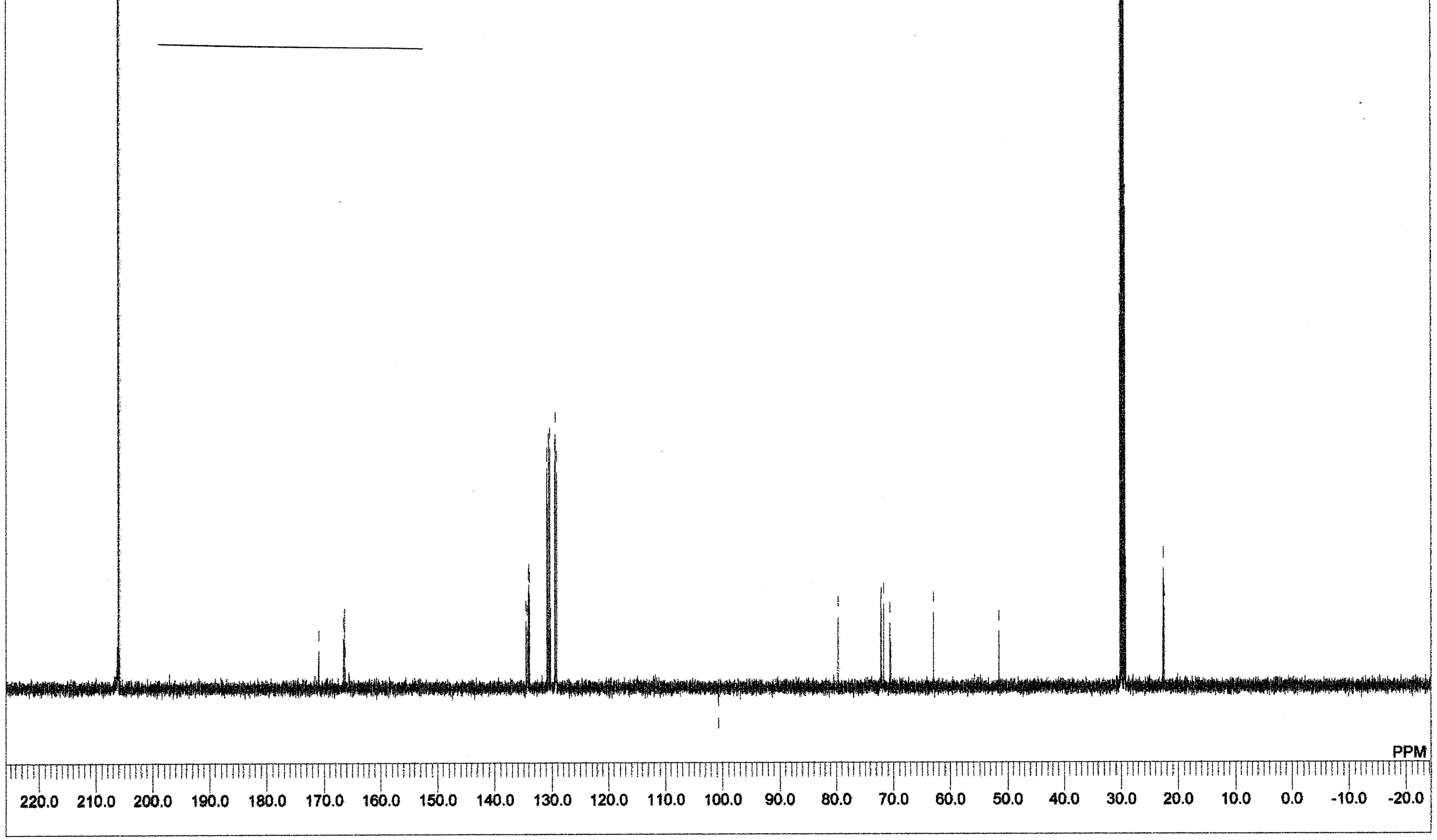
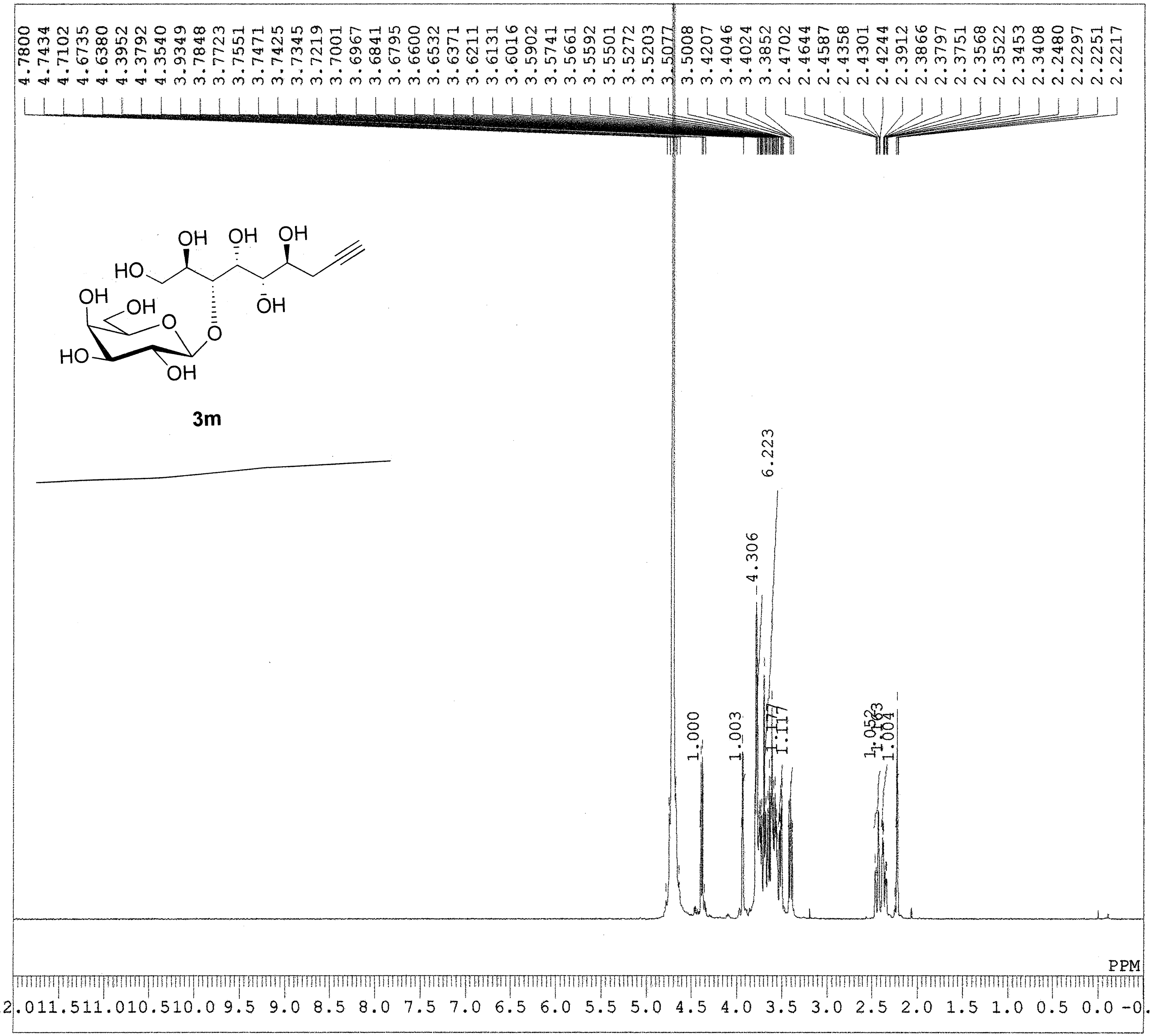


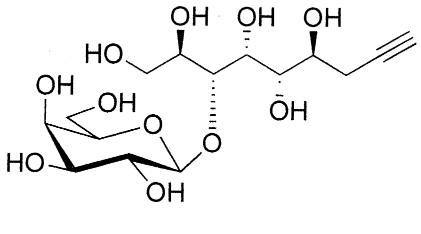
4l



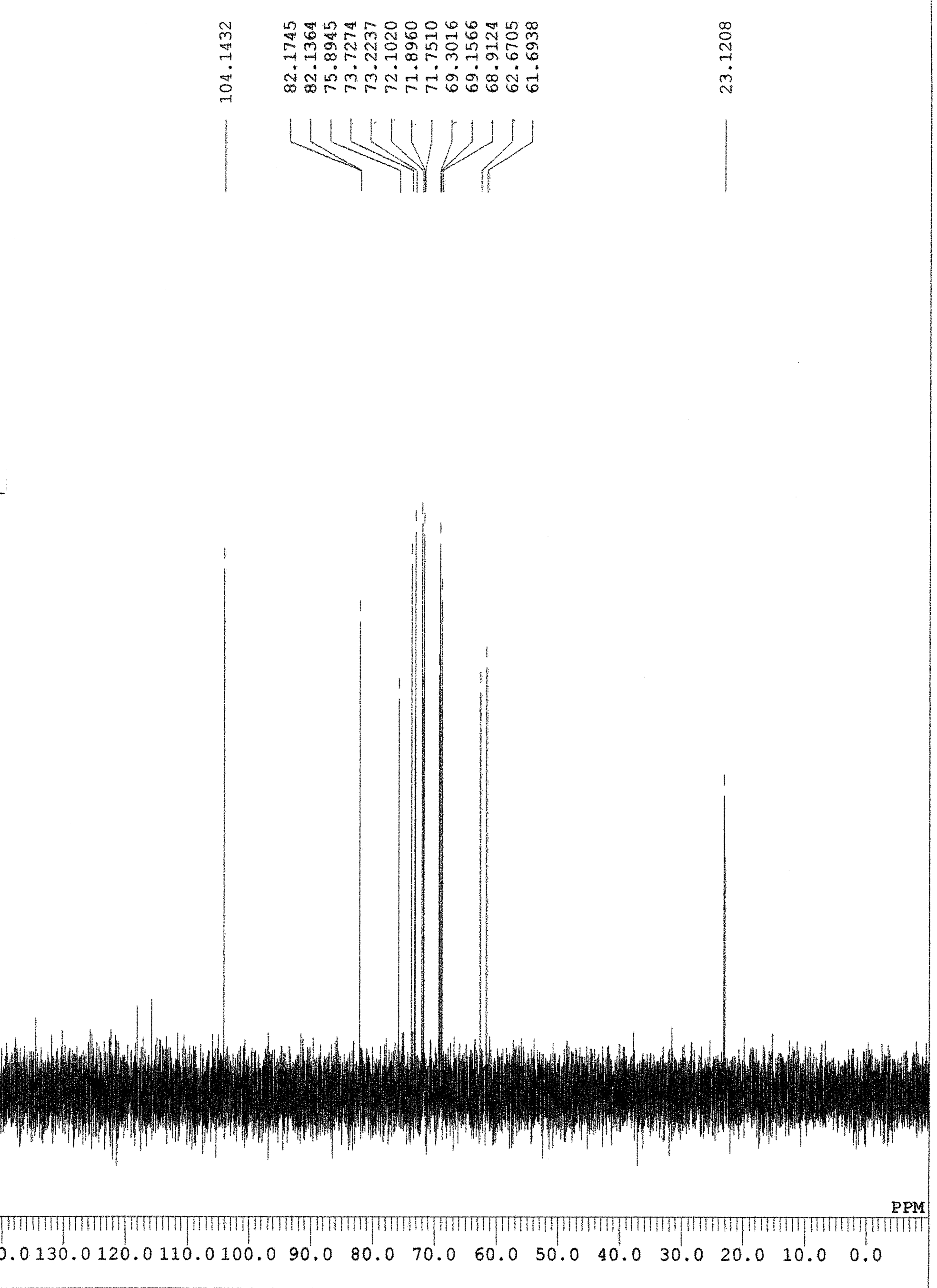
DFILE	wei R-SKP Glucosamin carb_copy1-1-1.als
COMNT	
DATIM	2015-04-08 15:08:38
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	26214
FREQU	31446.54 Hz
SCANS	665
ACQTM	0.0000 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	29.6 c
SLVNT	ACETN
EXREF	29.80 ppm
BF	0.12 Hz
RGAIN	60



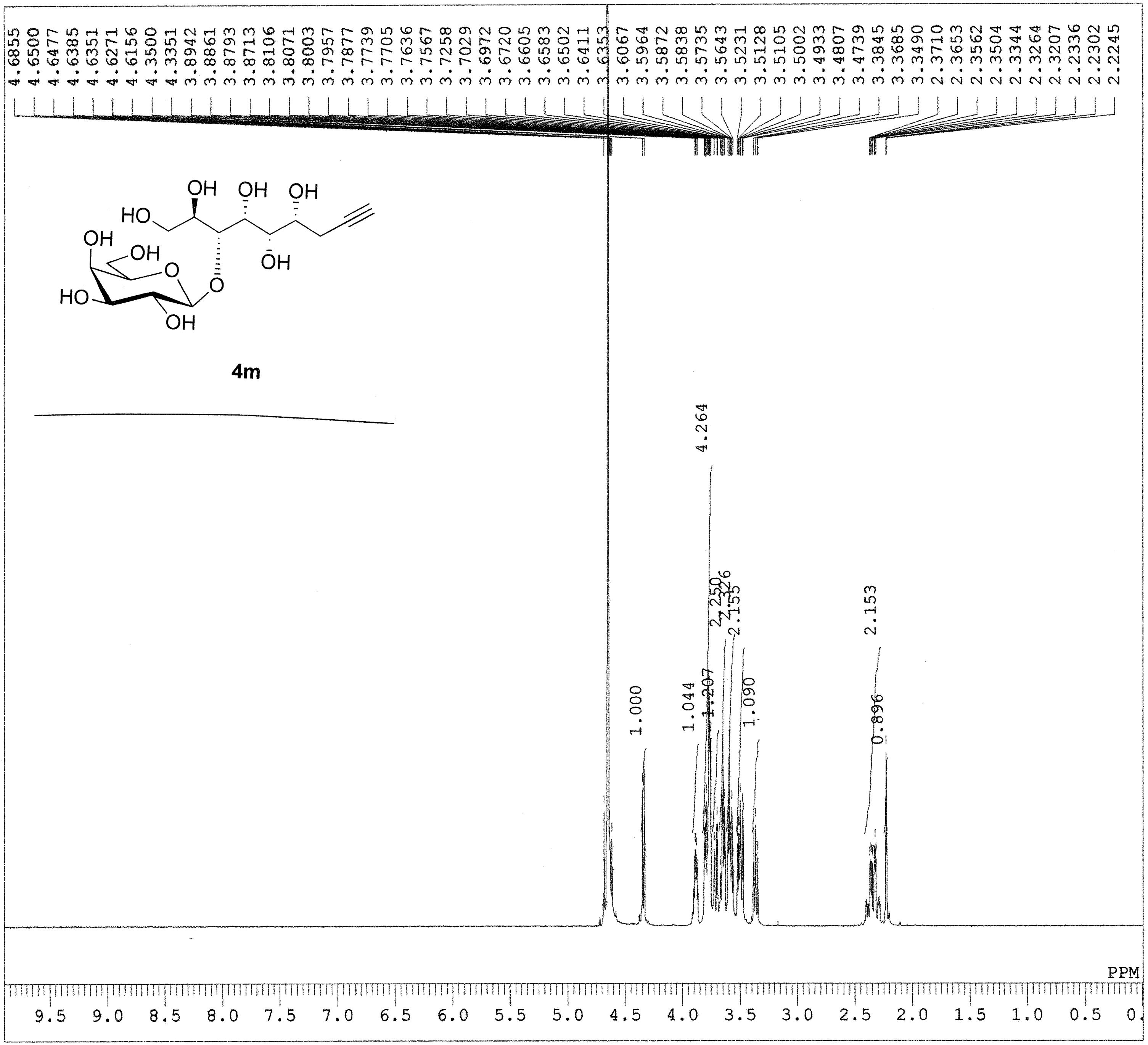
DFILE	wei PGF-lactose S-SKP-1-1.jdf
COMNT	
DATIM	2015-05-22 20:38:01
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16384
FREQU	9384.38 Hz
SCANS	12
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.5 c
SLVNT	D2O
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	30



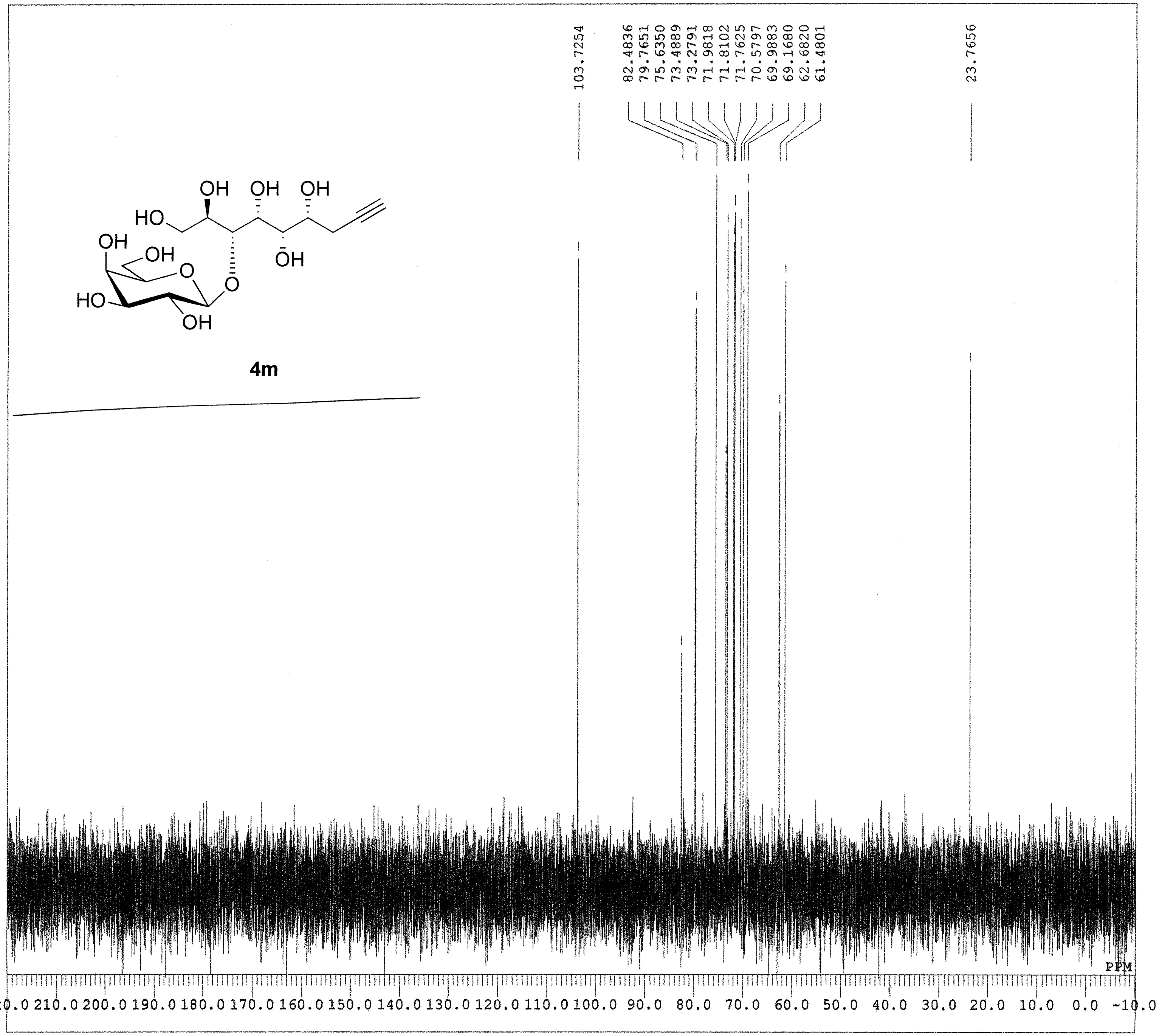
3m



DFILE	3m s-skp lactose alkyne carb-1-1.jdf
COMNT	
DATIM	2015-06-02 12:53:23
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32768
FREQU	31446.54 Hz
SCANS	431
ACQTM	1.0420 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	21.6 c
SLVNT	D2O
EXREF	49.50 ppm
BF	0.12 Hz
RGAIN	76



DFILE 4m R-skp lactose alkyne.als
COMNT
DATIM 2015-06-15 16:00:01
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.55 usec
IRNUC 1H
CTEMP 21.2 c
SLVNT D2O
EXREF 4.65 ppm
BF 0.12 Hz
RGAIN 30



DFILE	4m R-skp lactose carb-1-1.jdf	
COMNT		
DATIM	2015-06-15 16:01:31	
OBNUC	13C	
EXMOD	carbon.jxp	
OBFRQ	125.77	MHz
OBSET	7.87	KHz
OBFIN	4.21	Hz
POINT	32767	
FREQU	39308.18	Hz
SCANS	242	
ACQTM	0.8336	sec
PD	2.0000	sec
PW1	3.40	usec
IRNUC	1H	
CTEMP	21.5	c
SLVNT	D2O	
EXREF	49.50	ppm
BF	0.12	Hz
RGAIN	60	

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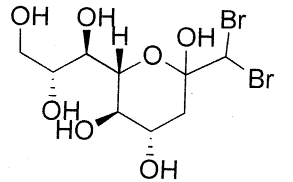
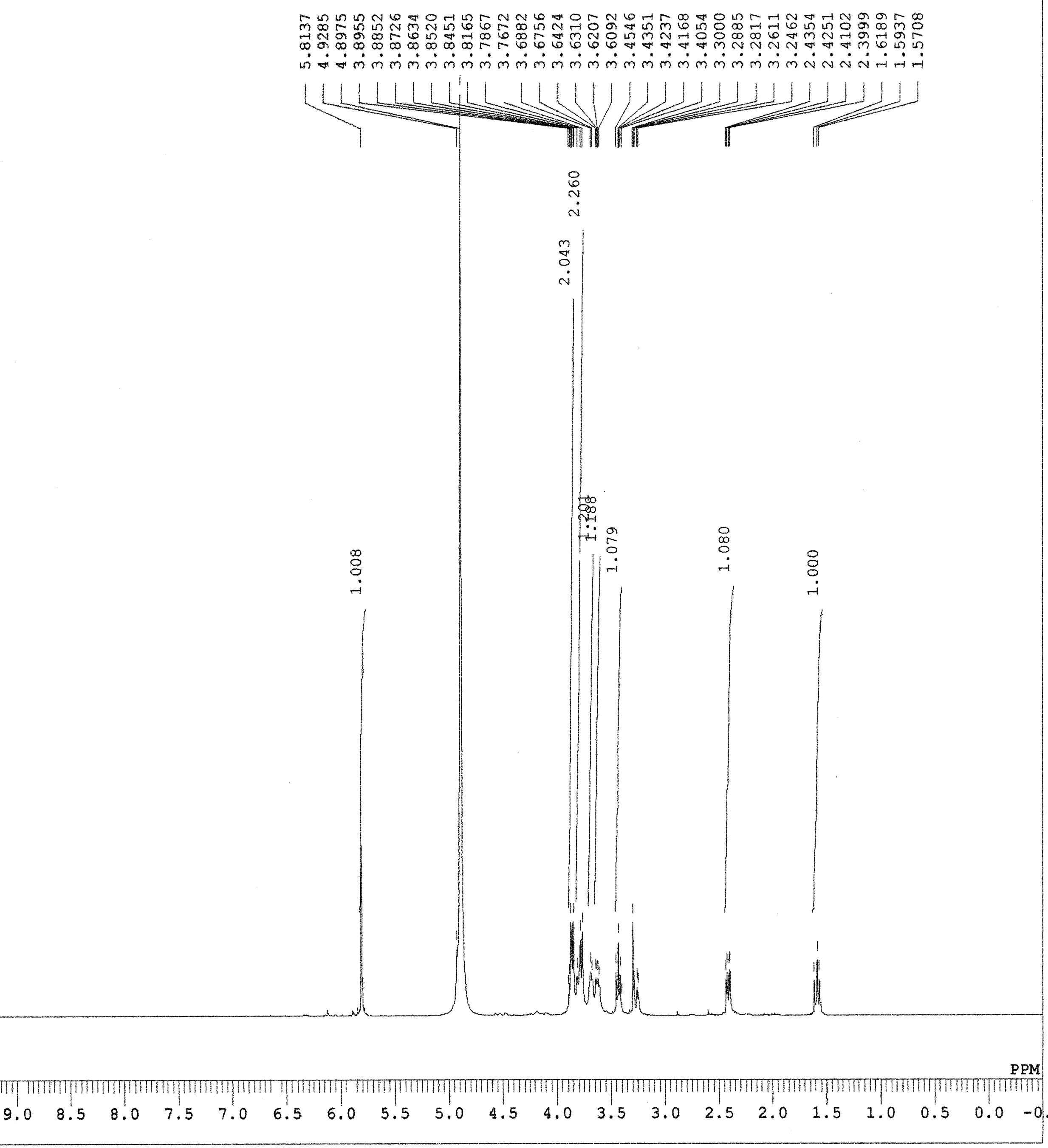
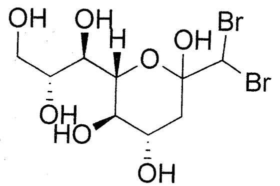


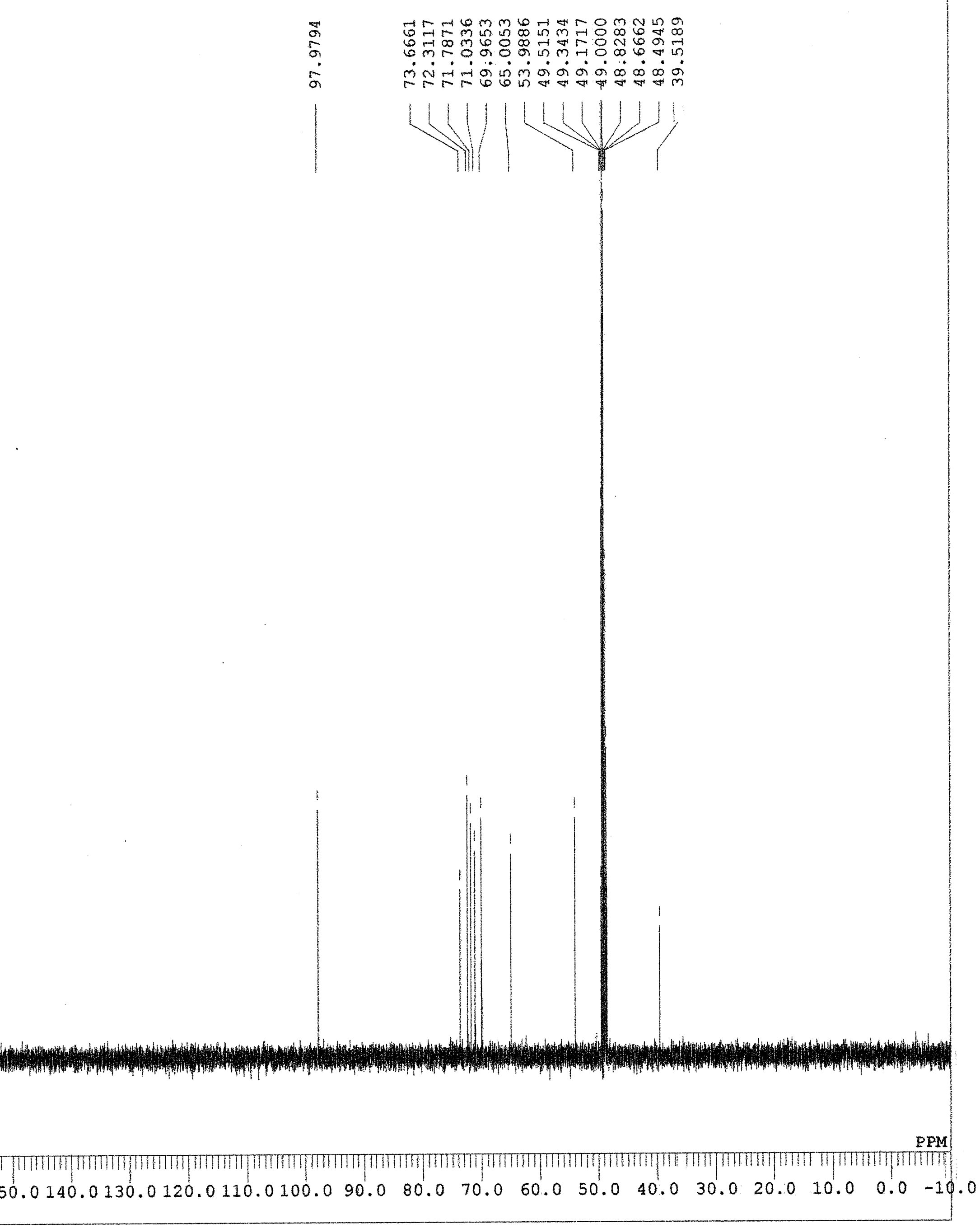
5a



DFILE	wei dibromo determin-1-1.jdf
COMNT	
DATIM	2014-11-08 13:38:16
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16384
FREQU	9384.38 Hz
SCANS	6
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	23.0 c
SLVNT	CD3OD
EXREF	3.30 ppm
BF	0.12 Hz
RGAIN	32



5a



DFILE	wei man dibromo carb-1-1.jdf
COMNT	
DATIM	2015-08-20 16:06:46
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32767
FREQU	39308.18 Hz
SCANS	68
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	21.5 c
SLVNT	CD3OD
EXREF	49.00 ppm
BF	0.12 Hz
RGAIN	60

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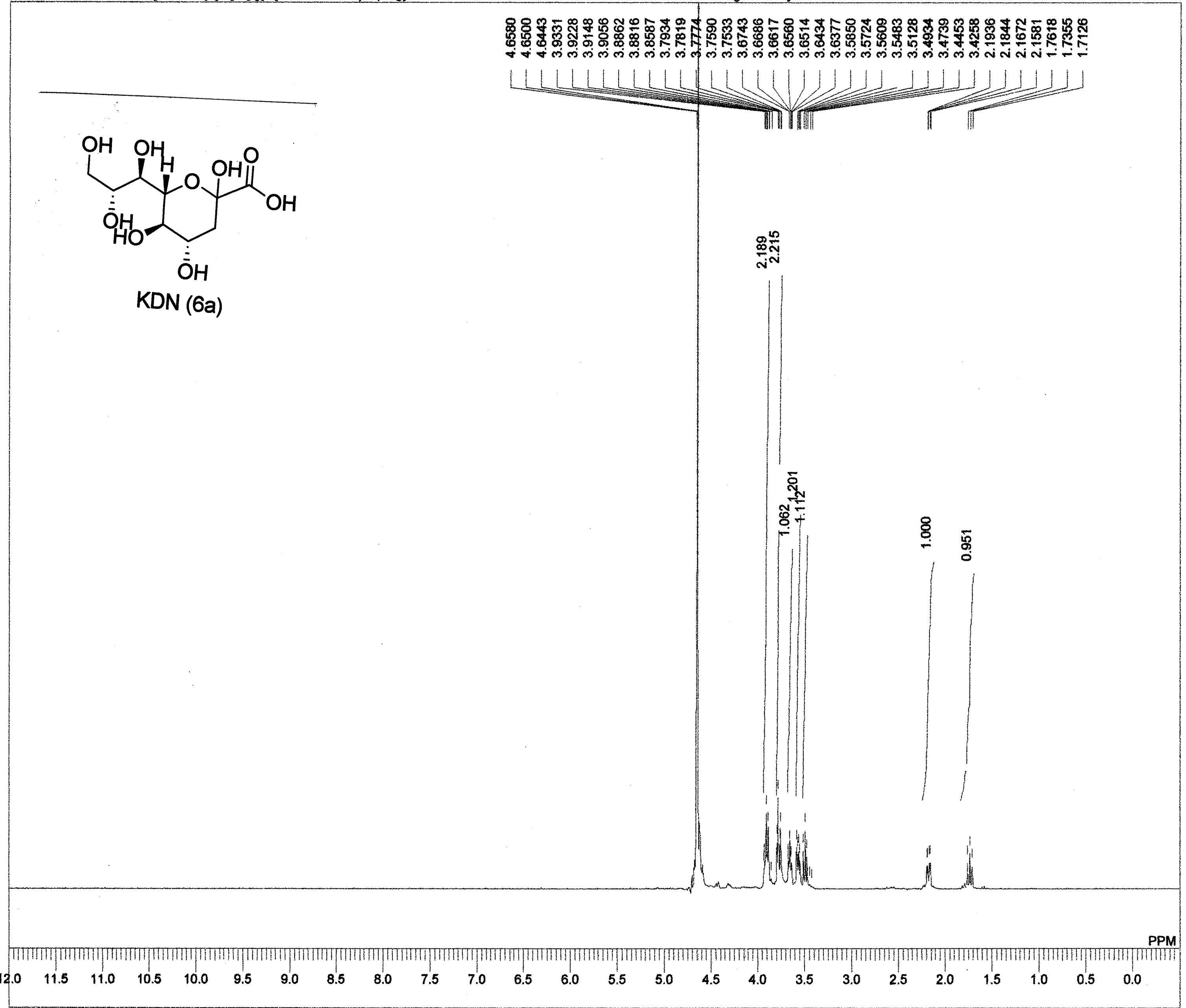

DFILE	wei kdn large scale .jdf
COMNT	
DATIM	2015-08-14 13:33:28
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16384
FREQU	9384.38 Hz
SCANS	16
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.4 c
SLVNT	D2O
EXREF	4.65 ppm
BF	0.12 Hz
RGAIN	36

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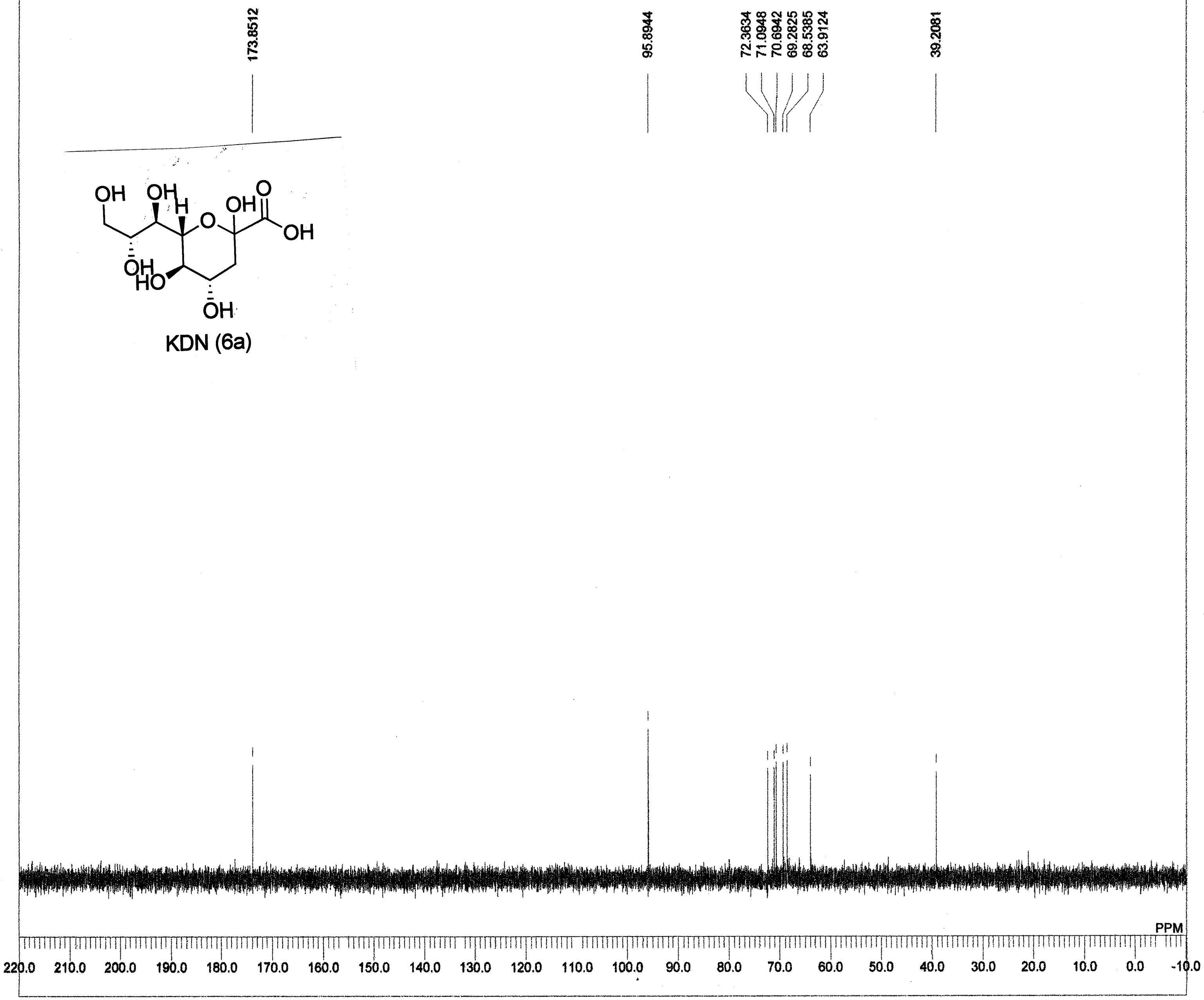
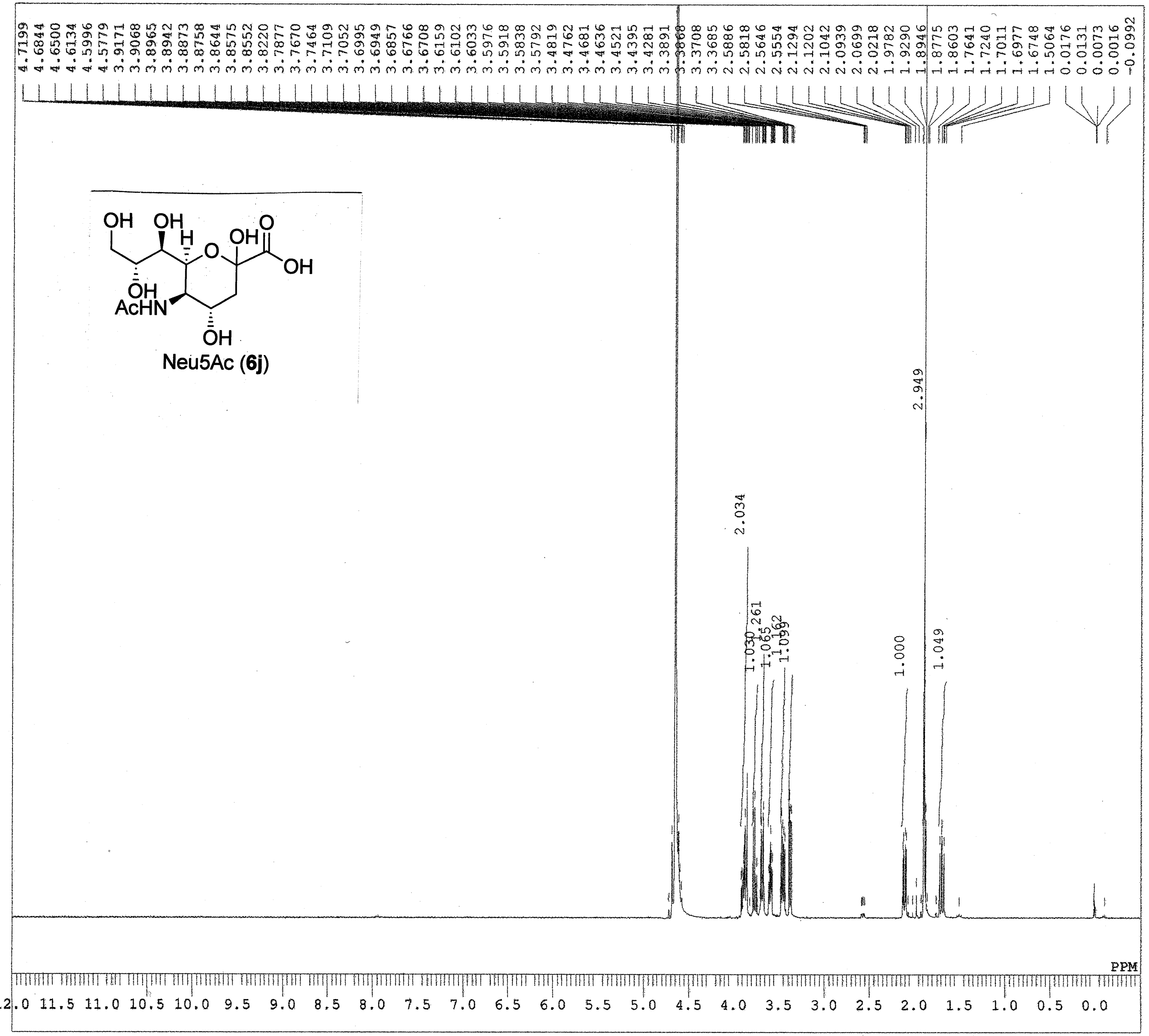


DFILE	kdn acid fresh carb-1-1.jdf
COMNT	
DATIM	2015-08-23 17:25:14
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32767
FREQU	39308.18 Hz
SCANS	2214
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	21.7 c
SLVNT	D2O
EXREF	49.50 ppm
BF	0.12 Hz
RGAIN	60




```
DFILE   wei Neu5Ac HPLC new-1-1.als
COMNT
DATIM   2015-05-12 20:32:38
OBNUC   1H
EXMOD   proton.jxp
OBFRQ        500.16 MHz
OBSET          2.41 KHz
OBFIN          6.01 Hz
POINT         13107
FREQU       7507.51 Hz
SCANS            14
ACQTM        1.7459 sec
PD           5.0000 sec
PW1            5.55 usec
IRNUC   1H
CTEMP          21.5 c
SLVNT   D2O
EXREF          4.65 ppm
BF             0.12 Hz
RGAIN            30
```

6h

DFILE	wei Neu5Ac car-1-1.jdf
COMNT	
DATIM	2015-08-21 01:45:44
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32767
FREQU	39308.18 Hz
SCANS	8000
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	21.7 c
SLVNT	D2O
EXREF	49.50 ppm
BF	0.12 Hz
RGAIN	60

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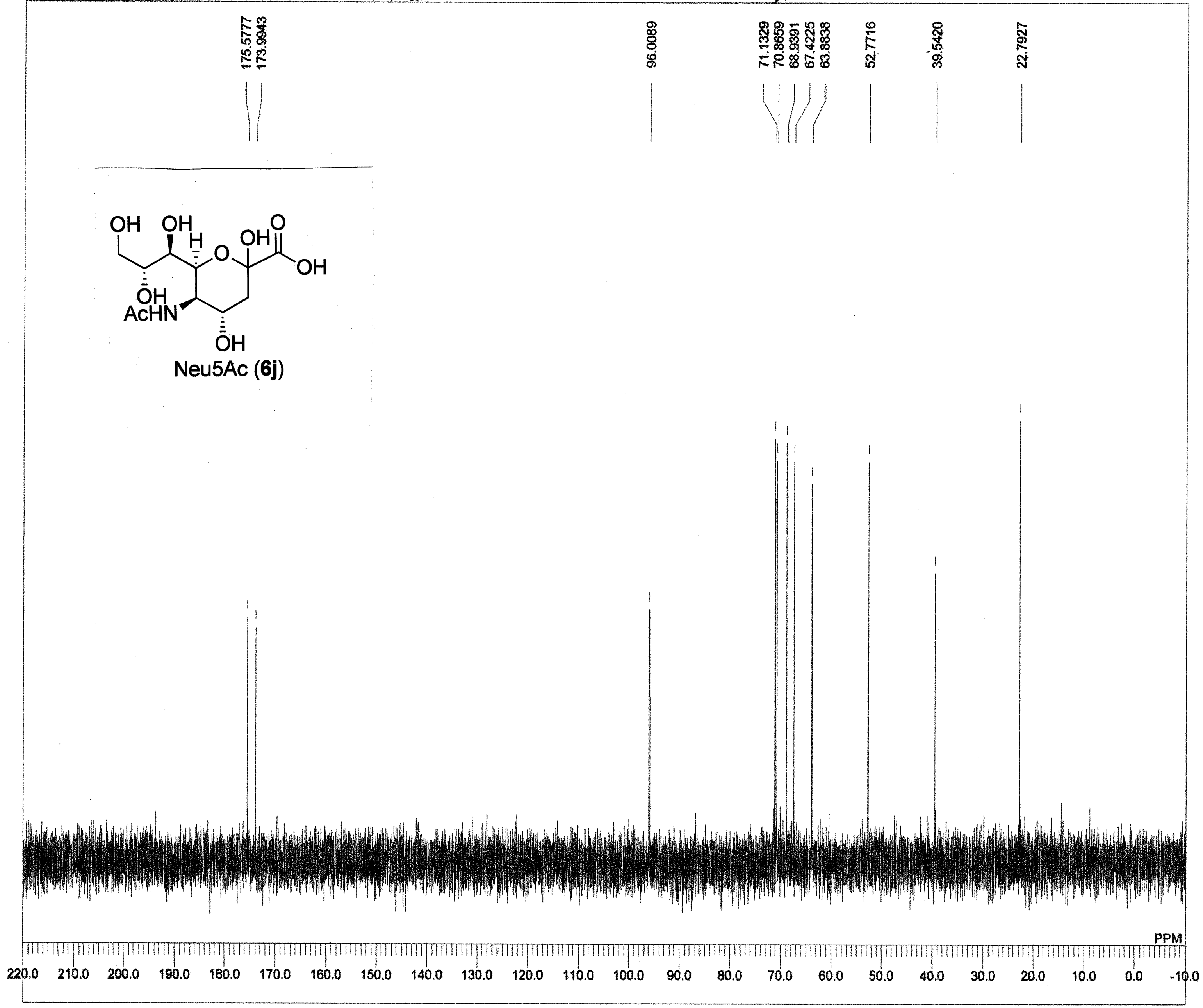
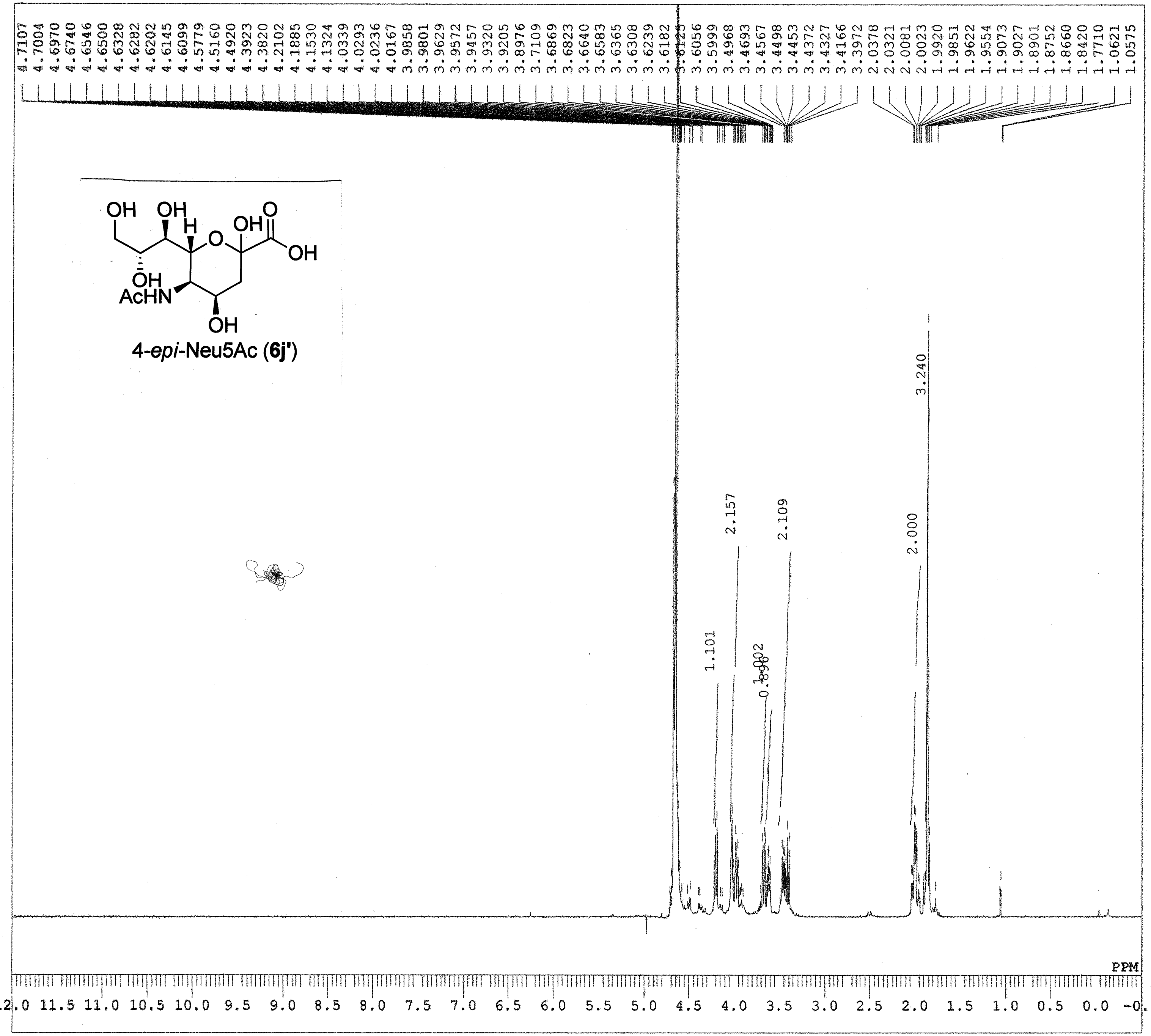
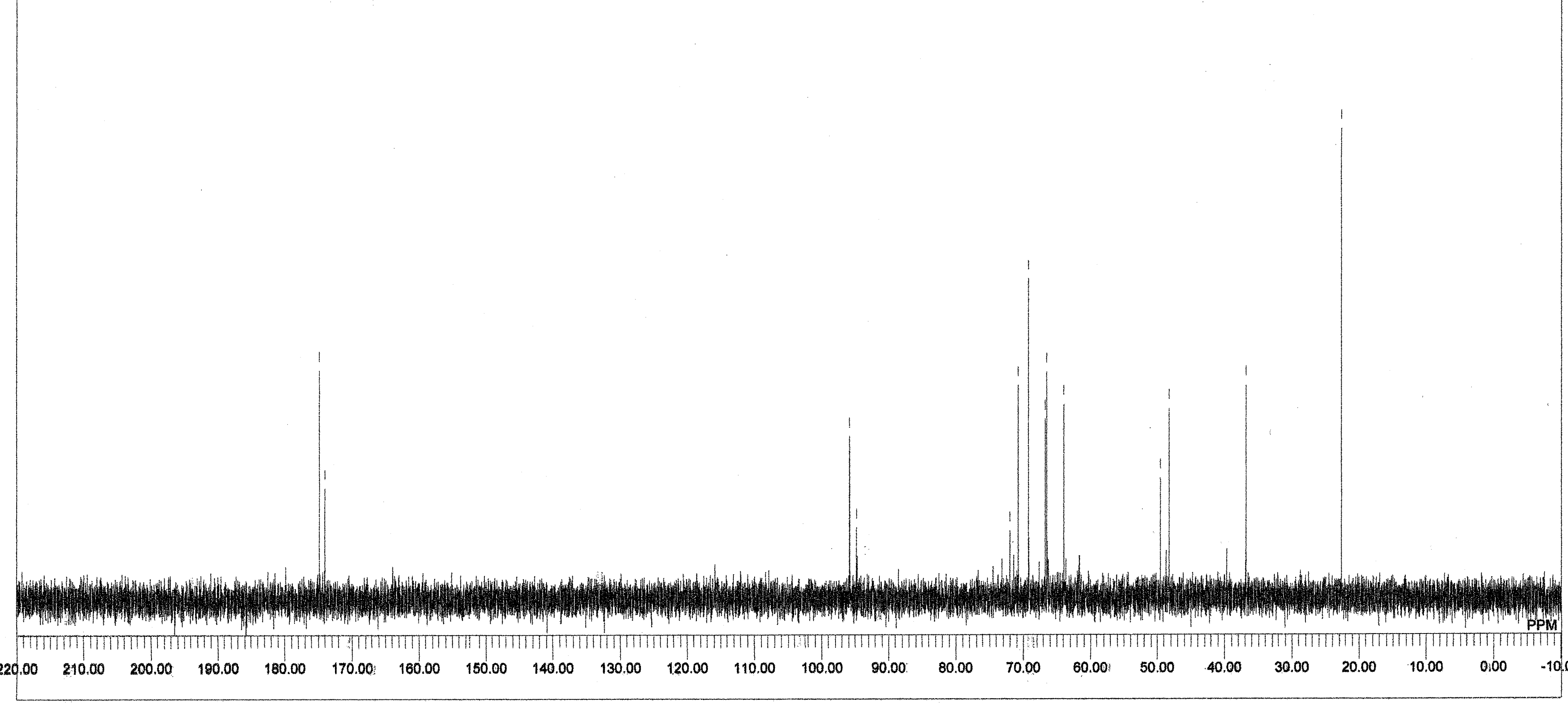
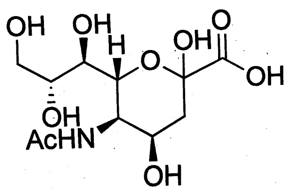




DFILE	wei epi Neu5Ac- most new.jdf	
COMNT		
DATIM	2015-09-02 23:09:55	
OBNUC	1H	
EXMOD	proton.jxp	
OBFRQ	500.16	MHz
OBSET	2.41	KHz
OBFIN	6.01	Hz
POINT	16384	
FREQU	9384.38	Hz
SCANS	14	
ACQTM	1.7459	sec
PD	5.0000	sec
PW1	5.55	usec
IRNUC	1H	
CTEMP	21.2	c
SLVNT	D2O	
EXREF	4.65	ppm
BF	0.12	Hz
RGAIN	34	

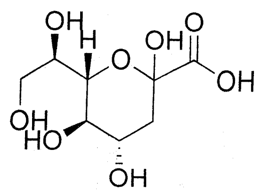
\\172.20.1.201\users\delta\Documents\JEOL\data\wei epi Neu carb_copy1-1-1.jdf



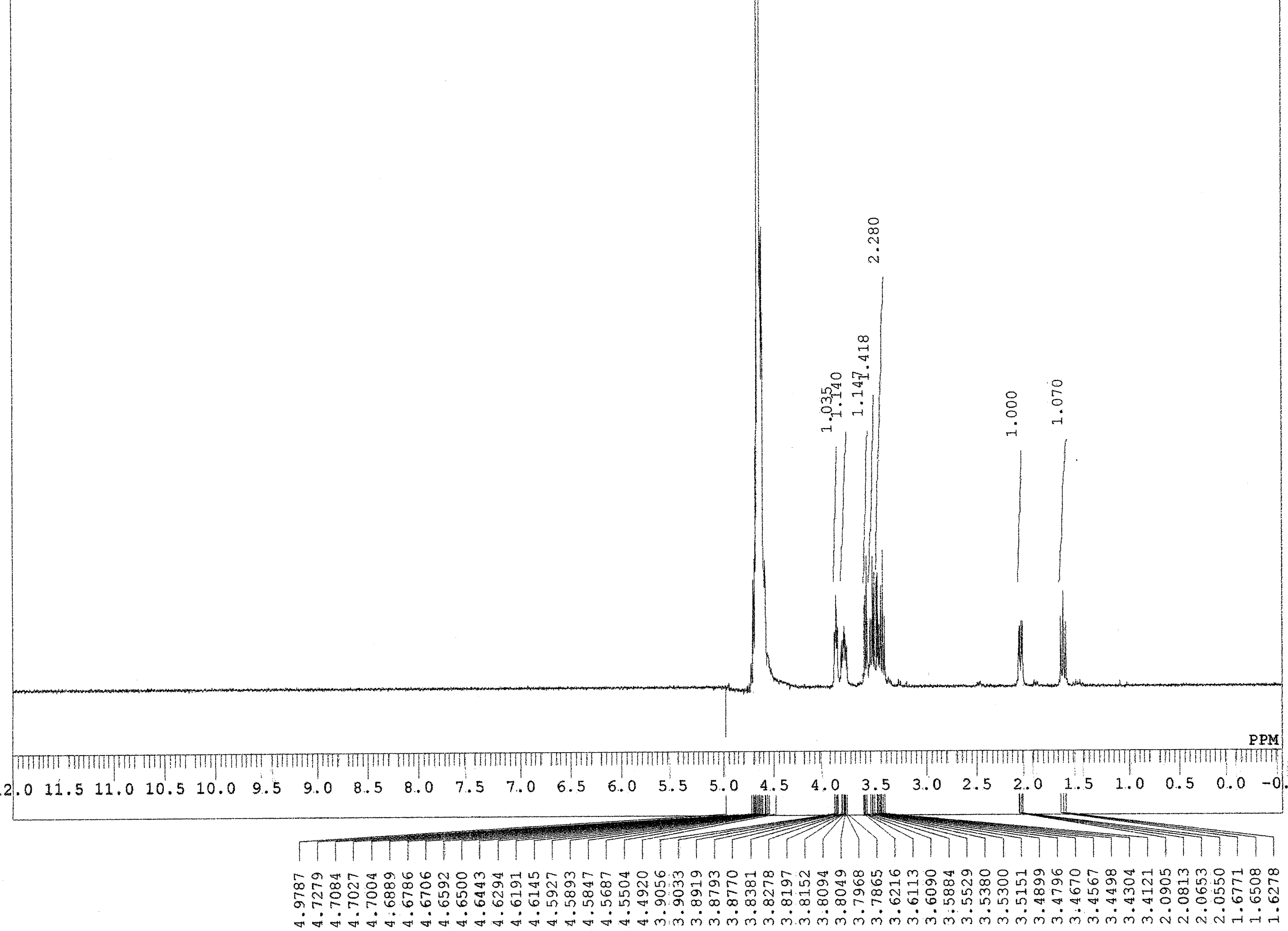


4-*epi*-Neu5Ac (**6j'**)

DFILE	wei epi Neu carb_copy1-1-1
COMNT	
DATIM	2015-09-04 00:01:34
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32767
FREQU	39308.18 Hz
SCANS	10313
ACQTM	0.0000 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	21.5 c
SLVNT	D2O
EXREF	49.50 ppm
BF	0.12 Hz
RGAIN	60



6d



DFILE	wei lyxose acid-new.jdf
COMNT	
DATIM	2015-08-31 23:15:07
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16384
FREQU	9384.38 Hz
SCANS	14
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	5.55 usec
IRNUC	1H
CTEMP	21.4 c
SLVNT	D2O
EXREF	4.65 ppm
BF	0.12 Hz
RGAIN	34

6d

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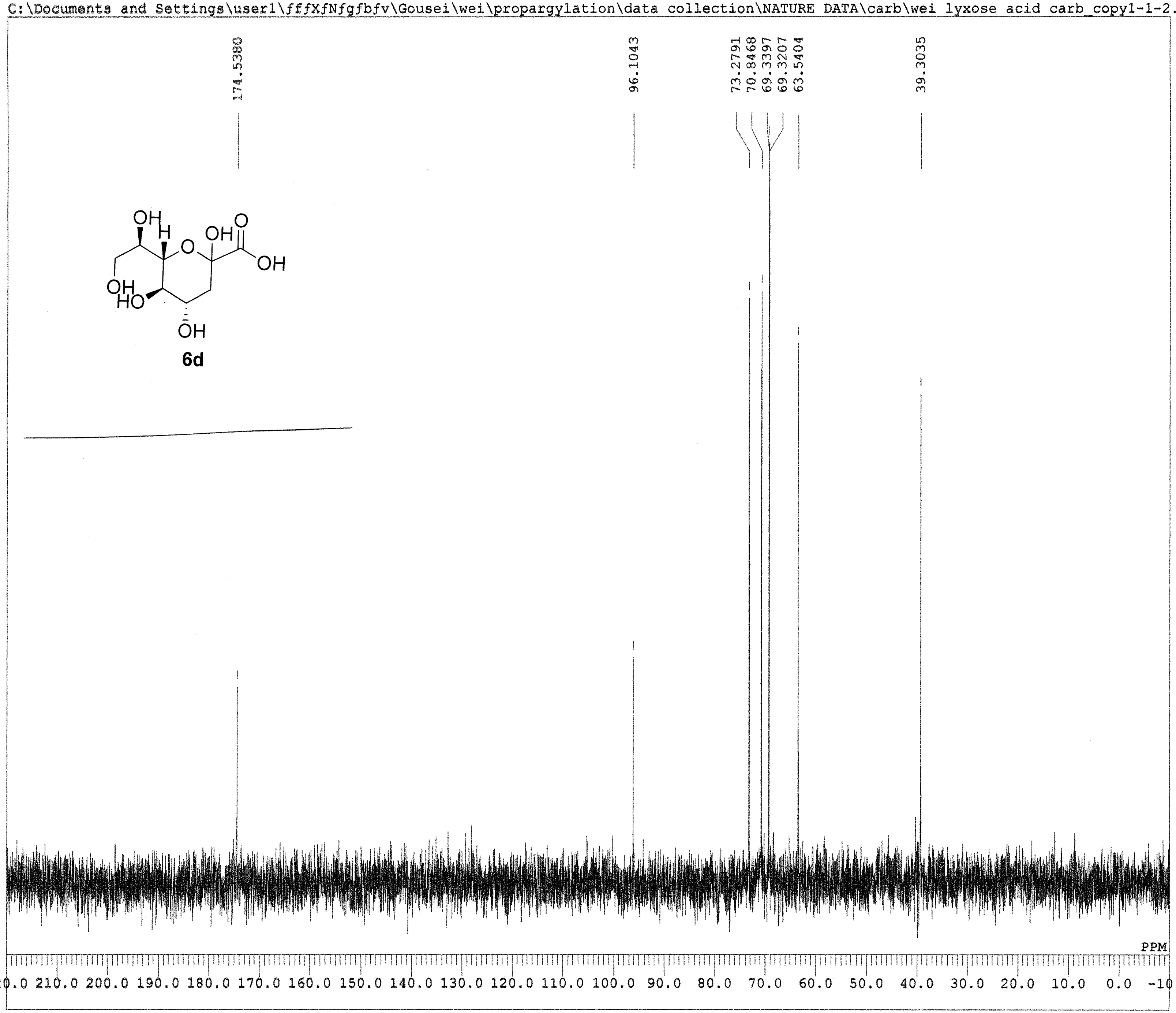

DFILE	wei lyxose acid carb_copy1-1-2.jdf
COMNT	
DATIM	2015-08-20 23:16:12
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	40960
FREQU	49135.22 Hz
SCANS	2912
ACQTM	0.0000 sec
PD	2.0000 sec
PW1	3.40 usec
IRNUC	1H
CTEMP	21.5 c
SLVNT	D2O
EXREF	49.50 ppm
BF	0.12 Hz
RGAIN	60

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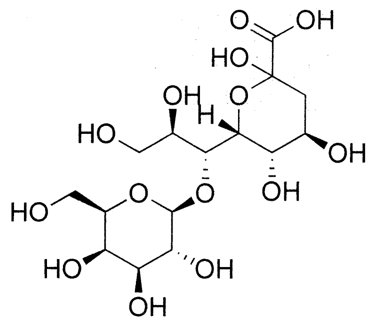
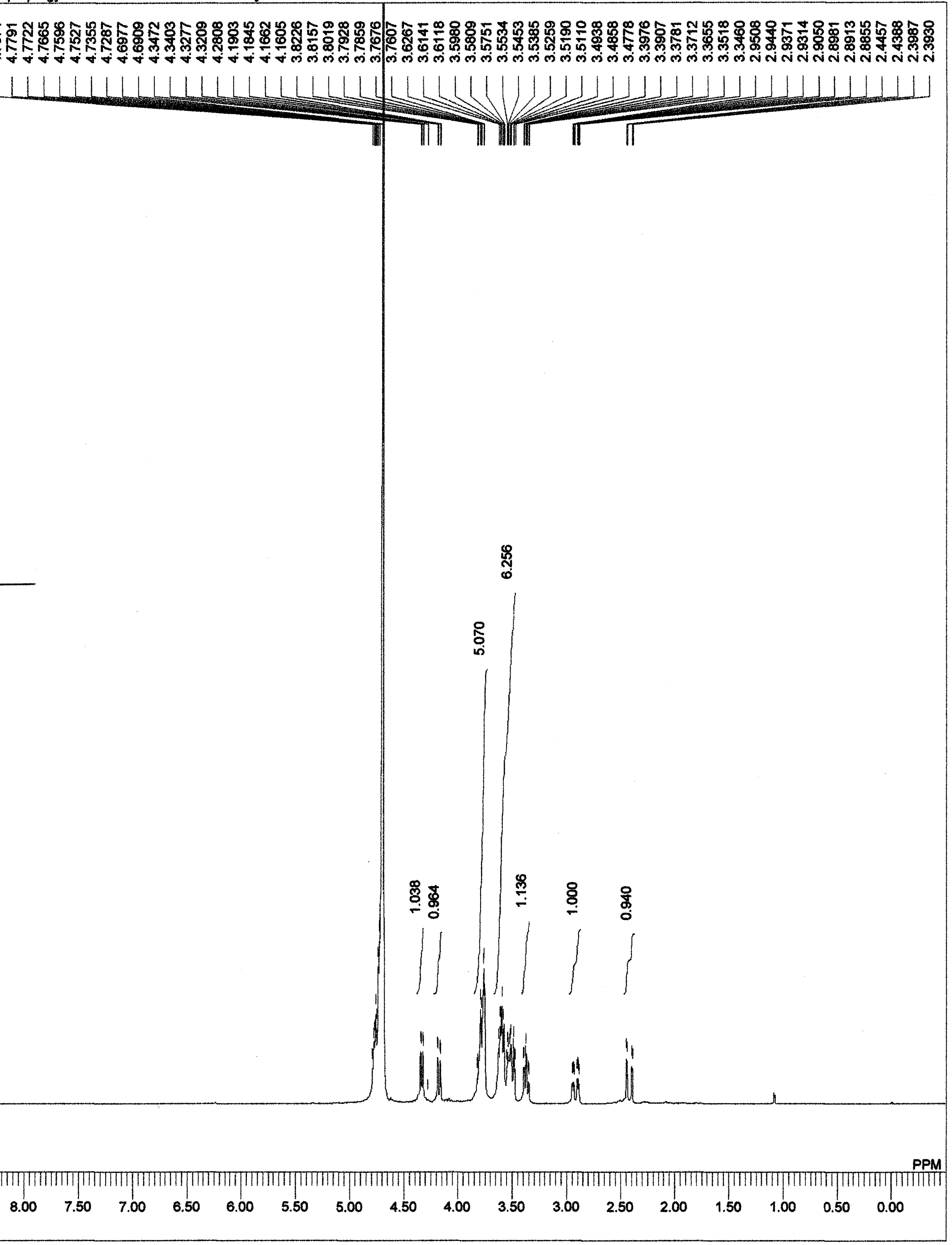


6m



DFILE	wei r-lactose acid-1-1.als
COMNT	
DATIM	23-07-2015 18:11:40
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	391.78 MHz
OBSET	8.51 KHz
OBFIN	3.34 Hz
POINT	13107
FREQU	5882.35 Hz
SCANS	18
ACQTM	2.2282 sec
PD	5.0000 sec
PW1	4.99 usec
IRNUC	1H
CTEMP	21.8 c
SLVNT	D2O
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	30

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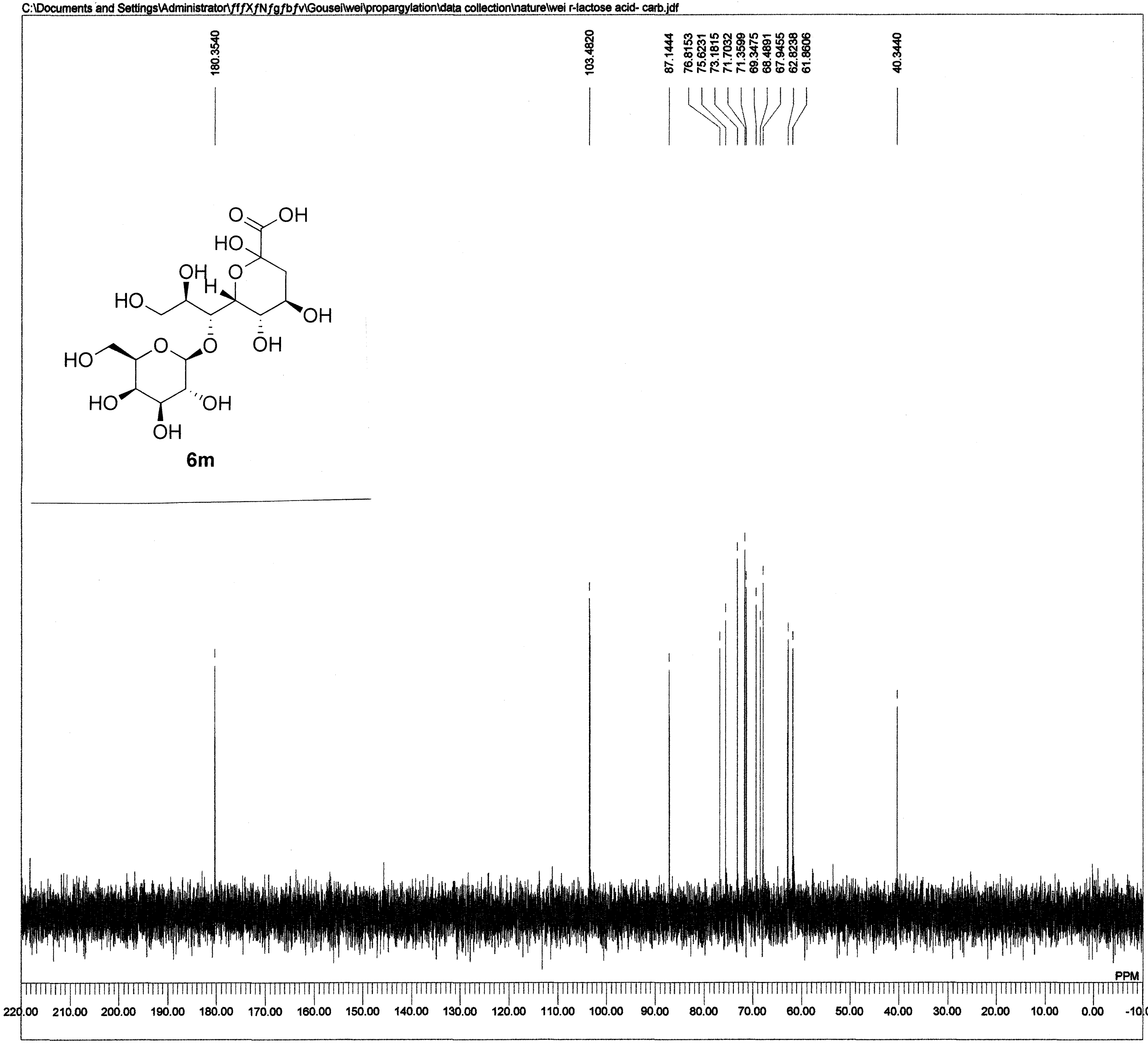

DFILE	wei r-lactose acid- carb.jdf
COMNT	
DATIM	23-07-2015 18:14:23
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	98.52 MHz
OBSET	4.64 KHz
OBFIN	8.74 Hz
POINT	32767
FREQU	30788.18 Hz
SCANS	1053
ACQTM	1.0643 sec
PD	2.0000 sec
PW1	3.16 usec
IRNUC	1H
CTEMP	21.9 c
SLVNT	D2O
EXREF	49.50 ppm
BF	0.12 Hz
RGAIN	60