

Supporting Information

Glycosylation Initiated Cationic Ring-Opening Polymerization of Tetrahydrofuran to Prepare Neo-glycopolymers

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Contents

	page
1. General procedures	S1-S3
2. Preparation and characterization of glycosyl <i>ortho</i> -hexynylbenzoates	S3
3. Preparation and characterization of G-PTHFs	S4-S6
4. Preparation and characterization of G-PTHF 3	S7
5. NMR spectra of new compounds	S8-S23

1. General procedures

Commercial reagents were used without further purification unless specialized. Solvents were dried and redistilled prior to use in the usual way. Thin layer chromatography (TLCs) were performed on precoated plates of Silica Gel HF254 (0.5 mm, Yantai, China). Flash column chromatography was performed on Silica Gel H (10–40 μ , Yantai, China). ^1H and ^{13}C NMR spectra were recorded on a Varian AM 300 or Bruker AM 400 spectrometer with Me_4Si as the internal standard. Chemical shifts are recorded in δ values and J values were given in Hz. Mass spectra were obtained on a Bruker Daltonics, Inc. APEXIII 7.0 TESLA FTMS mass spectrometer. Atomic force microscopy (AFM) was performed on a Veeco Nanoscope IVa MultiMode microscope. Molecular weights (M_n , M_w) and polydispersity indexes (M_w/M_n) of the synthesized G-PTHF samples were measured by conventional gel chromatography (GPC) system equipped with a waters 1515 Isocratic HPLC pump, a Waters 2414 refractive index detector, and a set of Waters Styragel columns (HR3, HR4 and HR5, 7.8 \times 300 mm). GPC measurements were carried out at 35 $^\circ\text{C}$ using THF as eluent with a flow rate of 1.0 mL/min. The system was calibrated with linear Polystyrene standards.

Preparation of $\text{PPh}_3\text{AuNTf}_2$ (commercially available).^{S1}

HNTf_2 (5620 mg, 20 mmol) was dissolved in H_2O (20 mL), Ag_2CO_3 (2760 mg, 10 mmol) was then added. After refluxing for 3 h, the solid was filtered off. The solution was dried in vacuo, and then dissolved in dry CH_2Cl_2 (20 mL). $[\text{Au}(\text{PPh}_3)\text{Cl}]$ (7730 mg, 20 mmol) was added. After stirring in dark for 15 min, the solid (AgCl) was filtered off and the solution was dried in vacuo to provide $\text{PPh}_3\text{AuNTf}_2$ quantitatively.

2. Preparation and characterization of glycosyl *ortho*-hexynylbenzoates

General procedures for the preparation of glycosyl *ortho*-hexynylbenzoates

1a-e. Deca-*O*-acetyl-maltotriose^{S2} (1550 mg, 1.68 mmol), *ortho*-hexynylbenzoic acid^{S1} (372 mg, 1.84 mmol), EDCI (416 mg, 2.18 mmol), and DMAP (205 mg, 1.68 mmol) were dissolved in CH_2Cl_2 (3 mL), then DIPEA (0.6 mL, 3.35 mmol) was added. After stirring for 5 h, the solution was diluted with CH_2Cl_2 , and was then washed with water and brine. The organic layer was dried over Na_2SO_4 , filtered,

and evaporated in vacuo. The residue was purified by silica gel column chromatography (petroleum ether-EtOAc 1.5:1) to give deca-*O*-acetyl-maltotriosyl *ortho*-hexynylbenzoate **1e** (1739 mg, 94%, $\alpha/\beta = 3:17$, which are inseparable) as a white foam.

(S1: Li, Y. et al.; Yang, X.; Liu, Y.; Zhu, C.; Yang, Y.; Yu, B. *Chem. Eur. J.* **2010**, 16, 1871-1882. S2: Buijsman, R.C.; Basten, J. E.; Dreef-Tromp, C. M.; van der Marel, G. A.; van Boeckel, CA. A.; van Boom, J. H. *Bioorg. Med. Chem.* **1999**, 7, 1881-1890)

Characterization of glycosyl *ortho*-hexynylbenzoates: Literature for known compounds and characterization data for new compounds.

Compounds	Literature or characterization data
1a	Yang, Y.; Li, Y.; Yu, B. <i>J. Am. Chem. Soc.</i> 2009 , 131, 12076-12077.
1b	Li, Y.; Yang, X.; Liu, Y.; Zhu, C.; Yang, Y.; Yu, B. <i>Chem. Eur. J.</i> 2010 , 16, 1871-1882.
1c	Li, Y.; Yang, X.; Liu, Y.; Zhu, C.; Yang, Y.; Yu, B. <i>Chem. Eur. J.</i> 2010 , 16, 1871-1882.
1d	Yang, Y.; Li, Y.; Yu, B. <i>J. Am. Chem. Soc.</i> 2009 , 131, 12076-12077.
1e	¹ H NMR (100 MHz, CDCl ₃): δ 8.01 (d, 0.15 H, $J = 7.6$ Hz), 7.90 (d, 0.85 H, $J = 7.6$ Hz), 7.60-7.27 (m, 3 H), 6.54 (s, 0.15 H), 6.01 (d, 0.85 H, $J = 8.0$ Hz), 5.65 (t, 0.15 H, $J = 9.6$ Hz), 5.46-5.26 (m, 4.85 H), 5.14 (t, 1 H, $J = 8.4$ Hz), 5.07 (t, 1 H, $J = 9.6$ Hz), 4.86 (d, 1 H, $J = 10.0$ Hz), 4.76 (d, 1 H, $J = 10.0$ Hz), 4.56-3.88 (m, 11 H), 2.58-2.45 (m, 2 H), 2.20-1.94 (m, 30 H), 1.70-1.45 (m, 4 H), 0.97 (t, 3 H, $J = 7.2$ Hz); ¹³ C NMR (100 MHz, CDCl ₃): δ 170.4, 170.3 (2C), 170.2, 170.1, 170.0, 169.5, 169.4, 169.2, 163.9, 163.0, 134.8, 134.5, 132.3, 130.6, 130.4, 129.8, 129.1, 127.2, 127.0, 125.6, 125.1, 97.1, 96.9, 95.9, 95.7, 95.5, 91.5, 89.4, 79.5, 78.8, 77.2, 74.8, 73.4, 73.3, 72.7, 72.4, 72.3, 71.9, 71.4, 70.8, 70.5, 70.2, 69.9, 69.8, 69.1, 68.8, 68.3, 67.7, 62.6, 62.4, 62.1, 61.2, 30.4, 30.3, 21.8, 21.7, 20.6 (2C), 20.5, 20.4, 20.3 (2C), 20.2, 19.2, 13.4; HRMS (ESI) calcd C ₅₁ H ₆₄ O ₂₇ Na [M+Na] ⁺ 1131.35272, found 1131.35538.

3. Preparation and characterization of G-PTHF's

General procedures for preparation of G-PTHF's. Under an argon atmosphere, to a solution of 3,4,6-tri-*O*-acetyl-2-azido-2-deoxy-D-glucopyranosyl *ortho*-hexynylbenzoate **1a** (206 mg, 0.4 mmol) in dry THF (35 mL) at room temperature was added a THF solution of PPh₃AuNTf₂ (0.2 mmol, 5 mL) under vigorously stirring. After different reaction time, the mixture was poured into ice cold water. The resulting G-PTHF **2a** was precipitated, which was then taken from the solution and dried under vacuum.

For a small scale analysis, a sample was taken from the reaction mixture via syringe and charged into cold water. The resulting aqueous solution was concentrated to give a residue, which was then purified by silica gel column chromatography (petroleum ether-EtOAc 3 : 1 to CH₂Cl₂-MeOH 25 : 1) to provide the G-PTHF **2a**.

The G-PTHF could also be purified by gel column Sephadex LH-20 (CH₂Cl₂-MeOH 1 : 1) or precipitation from *N,N*-dimethylformamide.

The ¹H and ¹³C NMR data of G-PTHF's

Compounds	characterization data
G-PTHF 2a (0.1 M in THF-d ₈ , at 2 d)	¹ H NMR (400 MHz, CDCl ₃): δ 5.48 (dd, 0.54 H, <i>J</i> = 10.0, 0.8 Hz, H4α), 5.09–4.95 (m, 2 H, H1α, H3α, H4β, H3β), 4.39(d, 0.46 H, <i>J</i> = 8.0 Hz, H1β), 4.29 (dd, 1 H, <i>J</i> = 12.4, 4.4 Hz, H6β, H6α), 4.15–4.00 (m, 1.76 H, H6β', H5α, H6α'), 3.62 (m, 0.52 H, H5β), 3.48 (m, 0.46 H, H2β), 3.39 (s, ~1.1 H, CD ₂), 3.28 (m, 0.56 H, H2α), 2.05–1.99 (m, 9 H, Ac), 1.58 (s, ~1.1 H, CD ₂).
G-PTHF 2a (0.01 M in THF, at 15 min)	¹ H NMR (400 MHz, CDCl ₃): δ 5.48 (dd, 0.55 H, <i>J</i> = 10.0, 0.8 Hz, H4α), 5.09–4.95 (m, 2 H, H1α, H3α, H4β, H3β), 4.39(d, 0.46 H, <i>J</i> = 8.0 Hz, H1β), 4.29 (dd, 1 H, <i>J</i> = 12.4, 4.4 Hz, H6β, H6α), 4.15–4.00 (m, 1.85 H, H6β', H5α, H6α'), 3.41 (m, ~390 H),. 2.05–1.99 (m, 9 H, Ac), 1.62 (m, ~390 H); ¹³ C NMR (75 MHz, CDCl ₃): δ 170.4, 169.9, 169.6, 102.0, 97.8, 71.4, 70.7, 70.5, 68.8, 68.5, 67.9, 67.5, 63.7, 62.5, 61.8, 60.8, 26.5, 20.6.
G-PTHF 2b (0.01 M in THF, at 15 min)	¹ H NMR (400 MHz, CDCl ₃): δ 8.03–7.80 (m, 8 H), 7.59–7.25 (m, 12 H), 5.90 (t, 1 H, <i>J</i> = 9.6 Hz), 5.68 (t, 1 H, <i>J</i> = 9.6 Hz), 5.52 (t, 1

THF, at 9 min)	H, $J = 8.0$ Hz), 4.65 (dd, 1 H, $J = 12.4, 2.8$ Hz), 4.51 (dd, 1 H, $J = 12.0, 5.2$ Hz), 4.16 (m, 1 H), 3.41 (m, ~ 700 H), 1.63 (m, ~ 700 H); ^{13}C NMR (75 MHz, CDCl_3): δ 166.1, 165.8, 165.0, 133.4, 133.1(2C), 129.9, 129.8, 129.7 (3C), 128.8, 128.4, 128.3 (2C), 101.2, 72.9, 72.2, 71.9, 70.8 (2C), 70.6, 70.4 (2C), 70.2, 70.1, 70.0, 69.8, 63.2, 62.7, 26.9, 26.7, 26.5, 26.3, 26.2, 26.0.
G-PTHF 2c (0.01 M in THF, at 15 min)	^1H NMR (400 MHz, CDCl_3): δ 8.00 (d, 2 H, $J = 7.6$ Hz), 7.59–7.05 (m, 18 H), 5.25 (t, 1 H, $J = 8.0$ Hz), 4.83–4.70 (m, 2 H), 4.69–4.45 (m, 5 H), 3.90–3.70 (m, 5 H), 3.40 (m, ~ 1092 H), 1.62 (m, ~ 1092 H); ^{13}C NMR (75 MHz, CDCl_3): δ 165.1, 138.1, 137.9, 137.8, 134.2, 134.0, 132.9, 132.0, 130.0, 129.7, 129.3, 129.1, 128.4, 128.3 (2C), 128.2, 128.0, 127.8, 127.7, 127.6, 101.1, 82.8, 78.0, 76.7, 75.2, 75.0, 73.8, 73.5, 71.4, 70.7, 70.6, 70.4, 70.1, 69.7, 69.4, 68.8, 67.4, 62.6, 27.3, 26.8, 26.6, 26.5, 26.3, 26.0.
G-PTHF 2d (0.01 M in THF, at 15 min)	^1H NMR (400 MHz, CDCl_3): δ 7.84–7.71 (m, 4 H), 7.40–7.25 (m, 5 H), 5.79 (t, 1 H, $J = 9.6$ Hz), 5.40 (d, 1 H, $J = 8.4$ Hz), 5.35 (t, 1 H, $J = 9.6$ Hz), 5.26 (d, 1 H, $J = 3.6$ Hz), 4.98 (t, 1 H, $J = 9.6$ Hz), 4.66 (m, 2 H), 4.22–4.00 (m, 4 H), 3.86–3.71 (m, 3 H), 3.62–3.19 (m, ~ 430 H), 2.05 (s, 3 H), 2.04 (s, 3 H), 2.02 (s, 3 H), 1.89 (s, 3 H), 1.75–1.50 (m, ~ 430 H); ^{13}C NMR (75 MHz, CDCl_3): δ 170.4, 169.8, 169.7, 169.6, 137.9, 134.1, 128.4, 127.7, 127.5, 123.4, 98.2, 97.8, 75.7, 74.2, 73.5, 71.5, 70.6, 70.3, 70.1, 69.6, 68.8, 68.3, 68.1, 62.6, 61.4, 61.0, 55.2, 26.9, 26.6, 26.5, 26.3, 26.1, 26.0, 20.6 (2C), 20.5.
G-PTHF 2e (0.1 M in THF-d ₈ , at 15 min)	^1H NMR (400 MHz, CDCl_3): δ 5.52 (t, 0.6 H, $J = 9.2$ Hz), 5.47–5.22 (m, 4.4 H), 5.07 (t, 1 H, $J = 10.0$ Hz), 4.95 (d, 0.6 H, $J = 3.6$ Hz), 4.89–4.70 (m, 3 H), 4.53–4.40 (m, 2.4 H), 4.36–4.16 (m, 3 H), 4.10–3.89 (m, 6 H), 3.39 (s, ~ 0.60 H, CD_2), 2.20–1.95 (m, 30 H), 1.56 (s, ~ 0.60 H, CD_2).
G-PTHF 2e (0.01 M in THF, at 5 min)	^1H NMR (400 MHz, CDCl_3): δ 5.52 (t, 0.6 H, $J = 9.2$ Hz), 5.47–5.22 (m, 4.4 H), 5.07 (t, 1 H, $J = 10.0$ Hz), 4.95 (d, 0.6 H, $J = 3.6$ Hz), 4.89–4.70 (m, 3 H), 4.53–4.40 (m, 2.4 H), 4.36–4.16

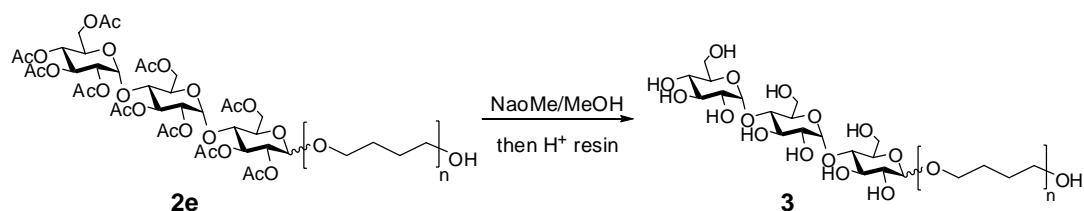
(m, 3 H), 4.10–3.89 (m, 6 H), 3.39 (s, ~570 H), 2.20–1.97 (m, 30 H), 1.56 (s, ~570 H); ^{13}C NMR (75 MHz, CDCl_3): δ 170.6, 170.5, 170.3, 169.7, 95.7, 76.7, 72.6, 72.4, 72.2, 71.5, 70.6, 70.4, 70.2, 68.8, 62.7, 61.3, 26.9, 26.5, 26.1, 20.8 (2C), 20.5.

Characterization of the glycosylation initiated polymerization of THF with donors **1a-e**.^a

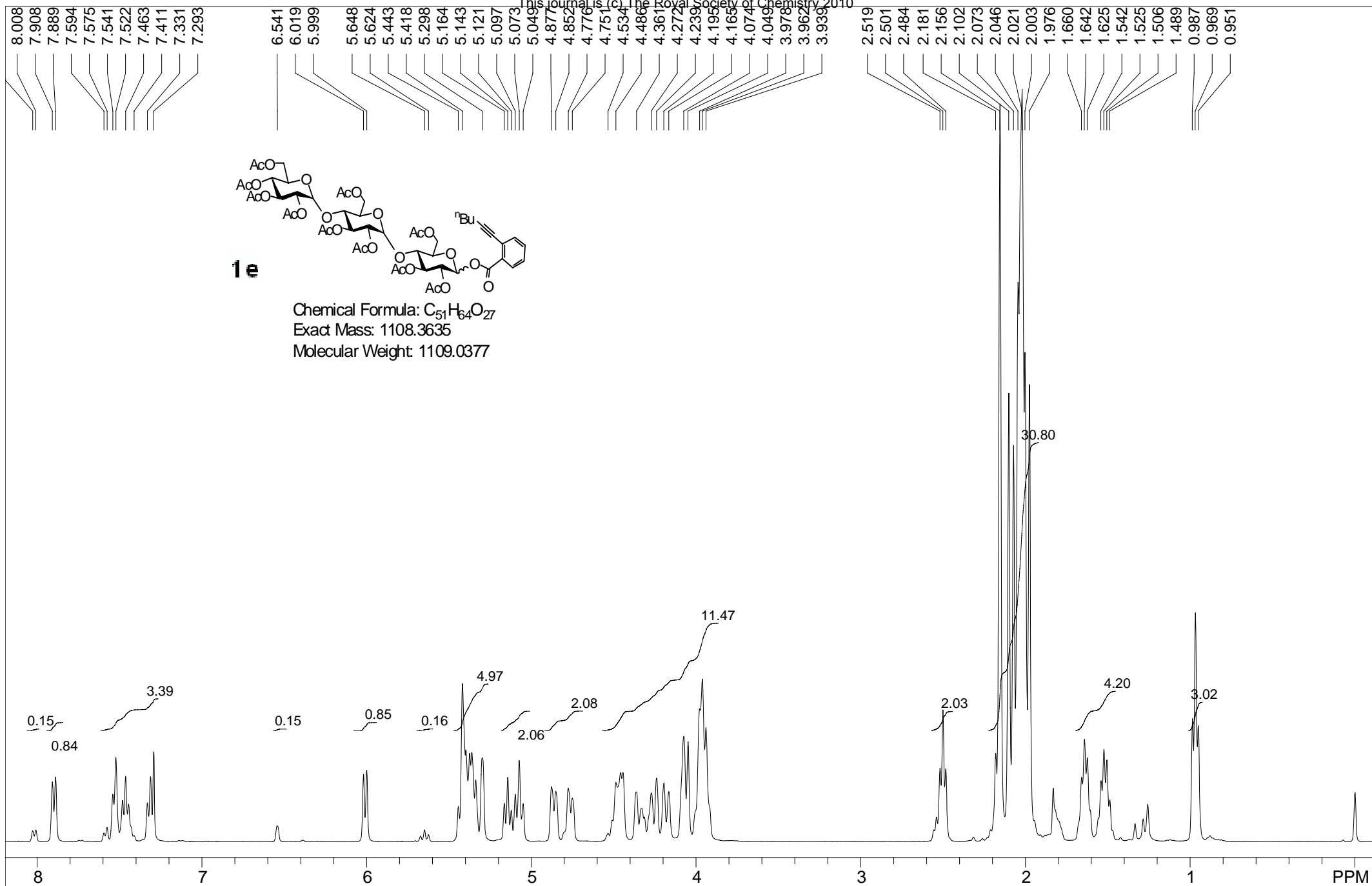
Entry	Donors	conc	Time (min)	Conv. ^c (%)	M_n^d	M_w^d	MP ^d	M_z^d	M_{z+1}^d	M_w/M_n
1 ^e	1a	0.01	5	2.5	8984	11310	12403	13365	15405	1.26
2 ^e	1a	0.01	15	4.7	16010	23351	32655	29534	34481	1.46
3	1a	0.01	45	8.8	20424	33507	35816	46160	56896	1.64
4	1a	0.01	120	9.8	23179	35951	37743	49334	61543	1.55
5 ^e	1b	0.01	3	2.1	8968	10354	10559	11816	13476	1.15
6 ^e	1b	0.01	9	4.6	17561	21622	24051	25105	28553	1.23
7	1b	0.01	17	7.1	25181	33354	41652	39513	44430	1.32
8	1b	0.01	28	8.3	28179	42297	61826	53190	61209	1.50
9	1b	0.01	65	9.1	29295	48365	53137	66246	81783	1.65
10	1b	0.01	120	10.6	31346	49670	54332	66706	81440	1.58
11	1b	0.01	2 d	19.0	73820	100625	102158	129219	155500	1.36
12	1b	0.005	2 d	8.0	50586	82580	87698	117150	147862	1.63
13	1b	0.04	2 d	60.0	32727	59630	57652	87806	115362	1.82
14 ^e	1c	0.01	5	4.0	11823	12496	13121	13061	13551	1.06
15	1c	0.01	15	6.6	28488	29623	30852	30620	31509	1.04
16	1c	0.01	45	8.6	35984	52589	77932	64134	71732	1.46
17	1c	0.01	120	12.1	39663	61685	65898	81259	98753	1.56
18 ^e	1d	0.01	5	2.6	12074	15664	17181	18857	21982	1.30
19	1d	0.01	15	3.3	18902	28351	37253	36903	44410	1.50
20	1d	0.01	45	3.9	20219	33713	39503	46552	57178	1.67
21	1d	0.01	120	5.0	23797	37572	43865	51413	63423	1.58
22 ^e	1e	0.01	5	6.0	19126	23778	24000	28152	32550	1.24
23	1e	0.01	15	7.1	25723	33907	40095	40028	45113	1.32
24	1e	0.01	45	8.2	27115	47141	47614	68925	88290	1.74
25	1e	0.01	120	10.0	40844	74132	77039	107876	140935	1.81

^aIn the presence of $\text{PPh}_3\text{AuNTf}_2$ (0.5 equiv) at rt. ^bThe concentration of donor **1b** in THF ($\text{mol}\cdot\text{L}^{-1}$). ^cThe conversion of THF into G-PTHF (wt%). ^dDetermined by GPC based on polystyrene standards. ^eSmall scale experiments were performed (<20 mg initiator).

4. Preparation and characterization of G-PTHF **3**

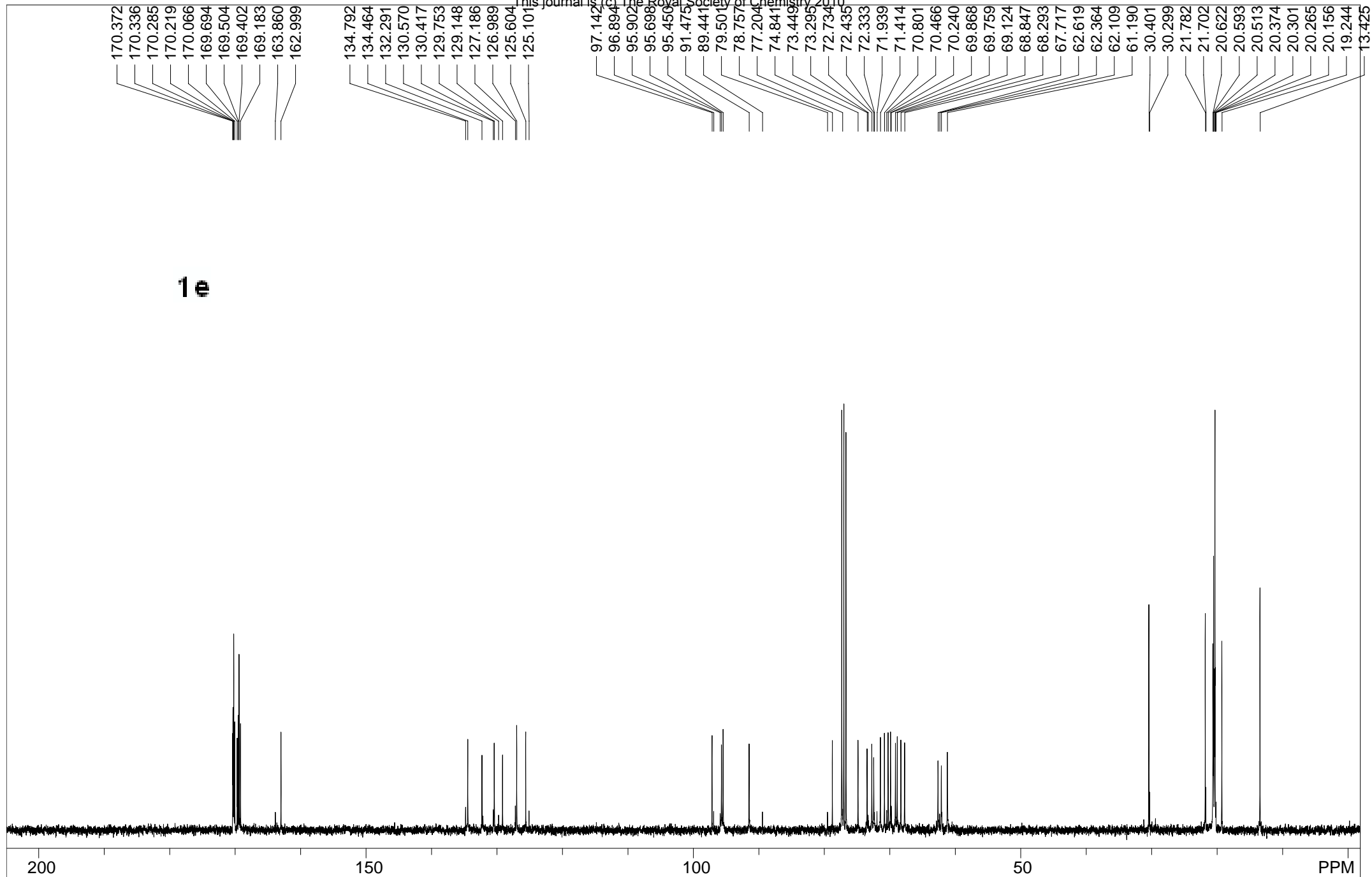


G-PTHF **2e** (60 mg, $M_n = 19126$, $M_w/M_n = 1.24$) was dissolved in MeOH (1.5 mL), NaOMe (4 mg) was added. After stirring overnight, the mixture was neutralized to pH = ~7 with Na⁺ exchange resin. The solution was filtered and dried under vacuum to provide G-PTHF **3** (59 mg, 100%) as a white solid: ¹H NMR (300 MHz, CD₃OD/CDCl₃ = 1 : 1): δ 5.12 (d, 2 H, $J = 3.9$ Hz), 4.80 (d, 0.6 H, $J = 3.9$ Hz), 4.28 (d, 0.4 H, $J = 7.8$ Hz), 3.46 (s, ~570 H), 1.63 (s, ~570 H); ¹³C NMR (100 MHz, CD₃OD/CDCl₃ = 1 : 1): δ 106.9, 105.5, 105.4, 84.6, 84.1, 84.0, 82.0, 80.4, 79.2, 77.7, 77.5, 77.3, 77.2, 76.8, 76.5, 75.9(2C), 75.8, 74.9, 74.8, 74.6, 74.5, 74.3, 74.1, 73.9, 73.3, 71.7, 65.4, 64.8, 64.7(2C), 52.5, 52.3, 52.0, 30.4, 30.2, 30.0, 29.9, 28.8.



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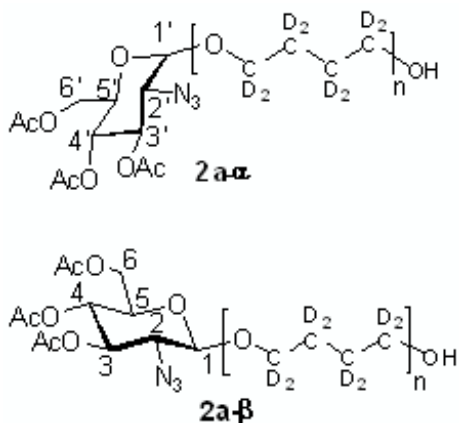
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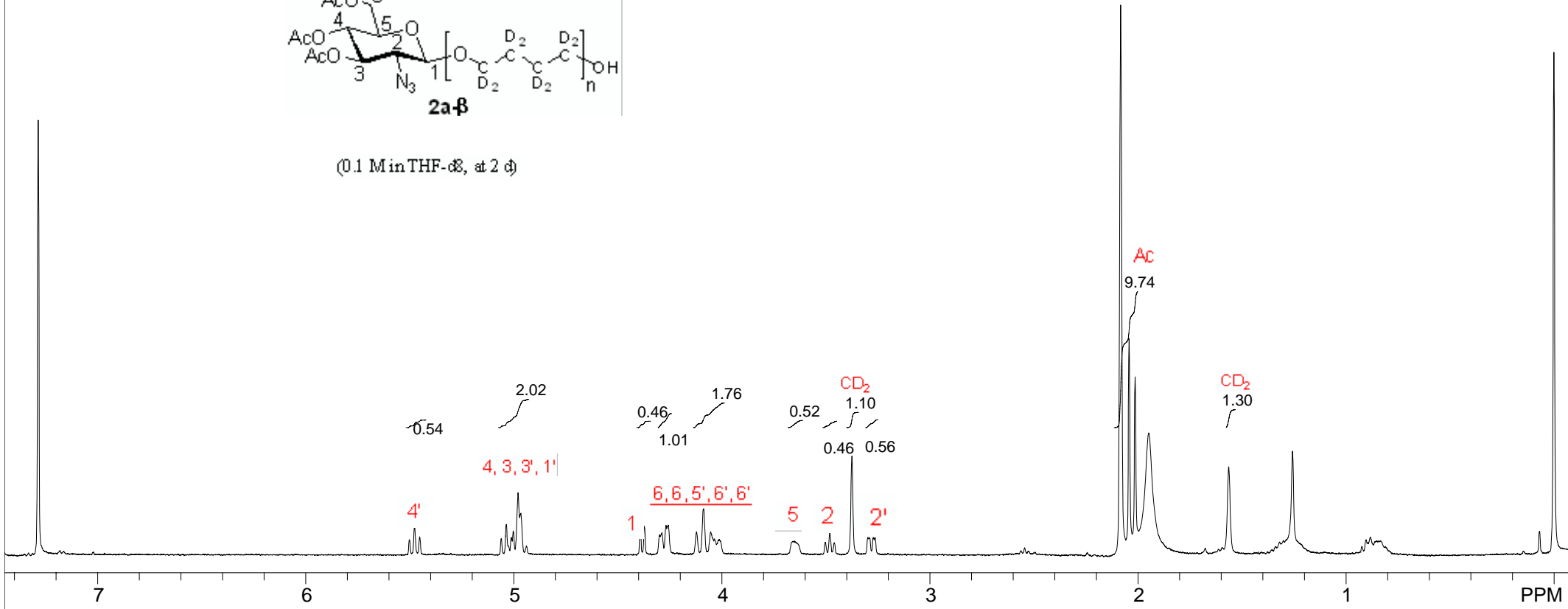
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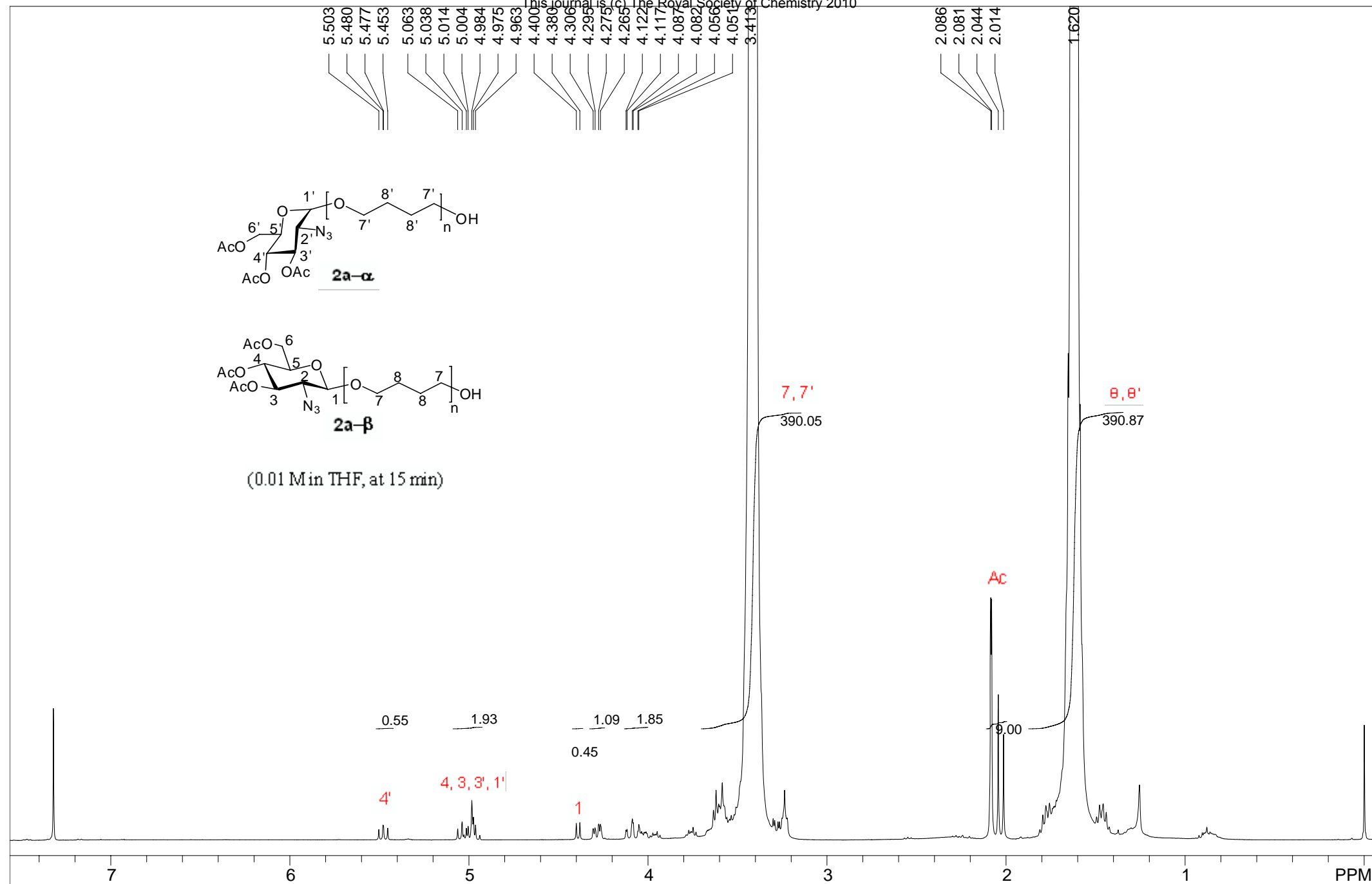


(0.1 M in THF-d₈, at 2 °C)



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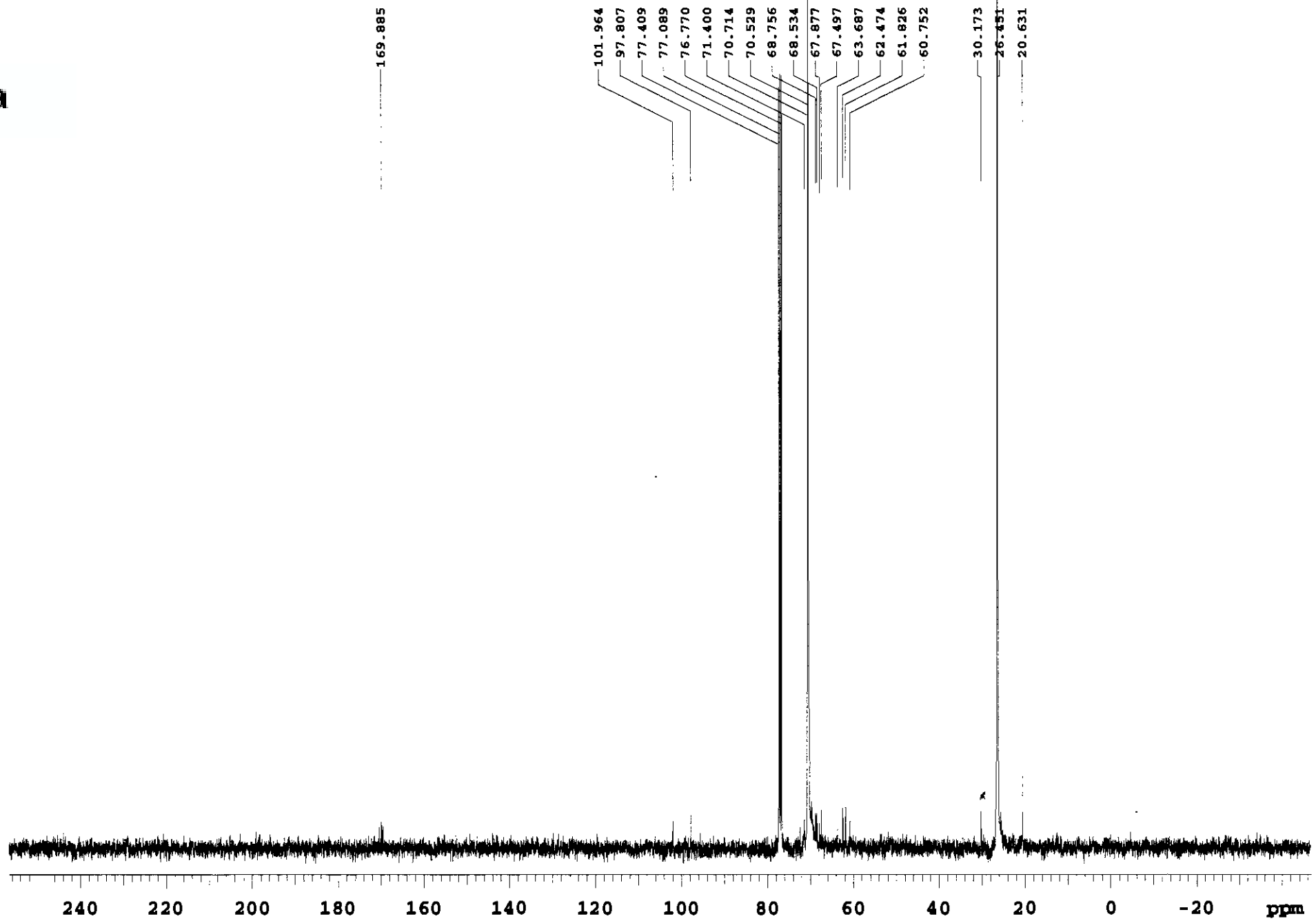


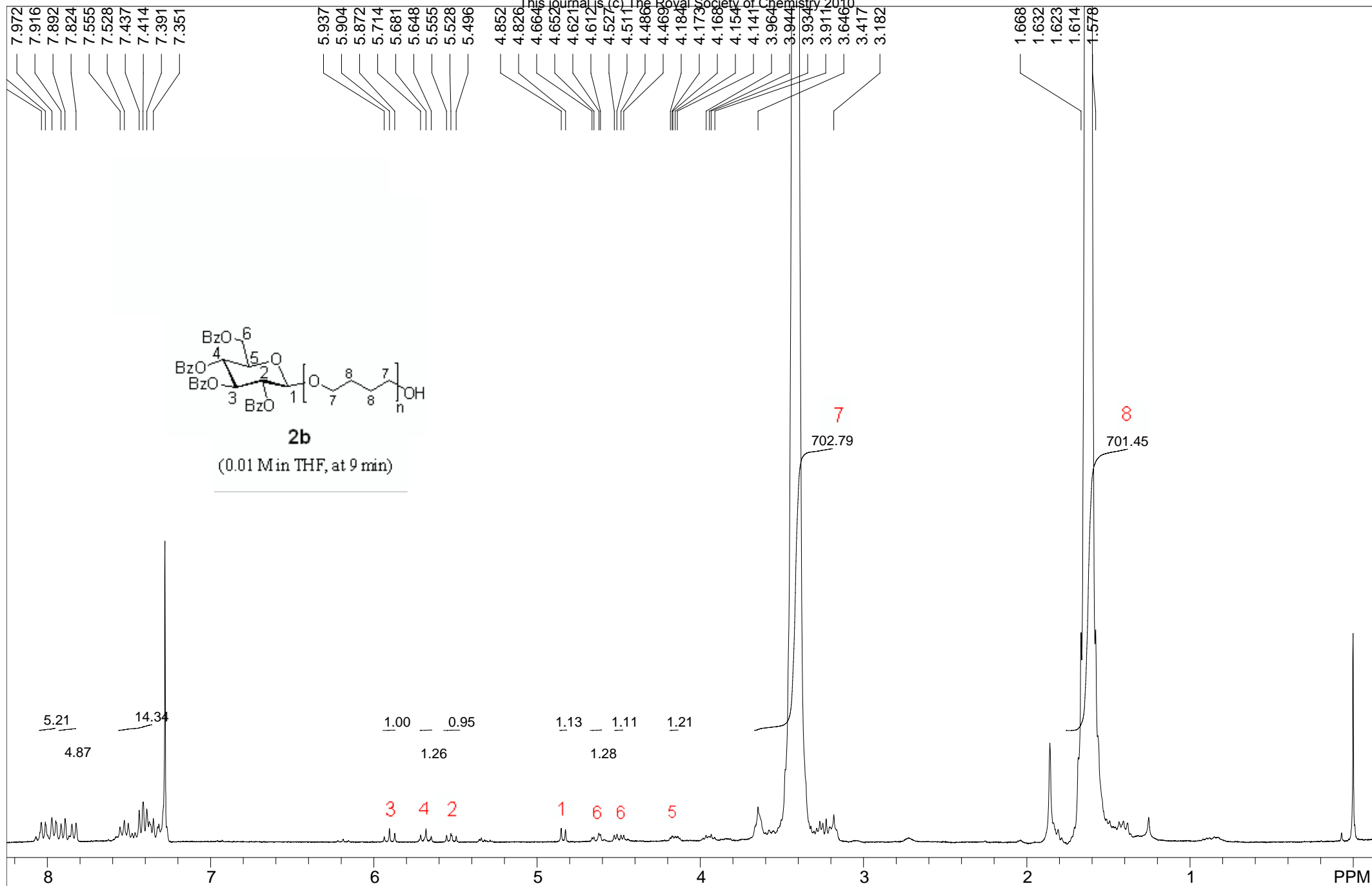
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1-857
11

2a





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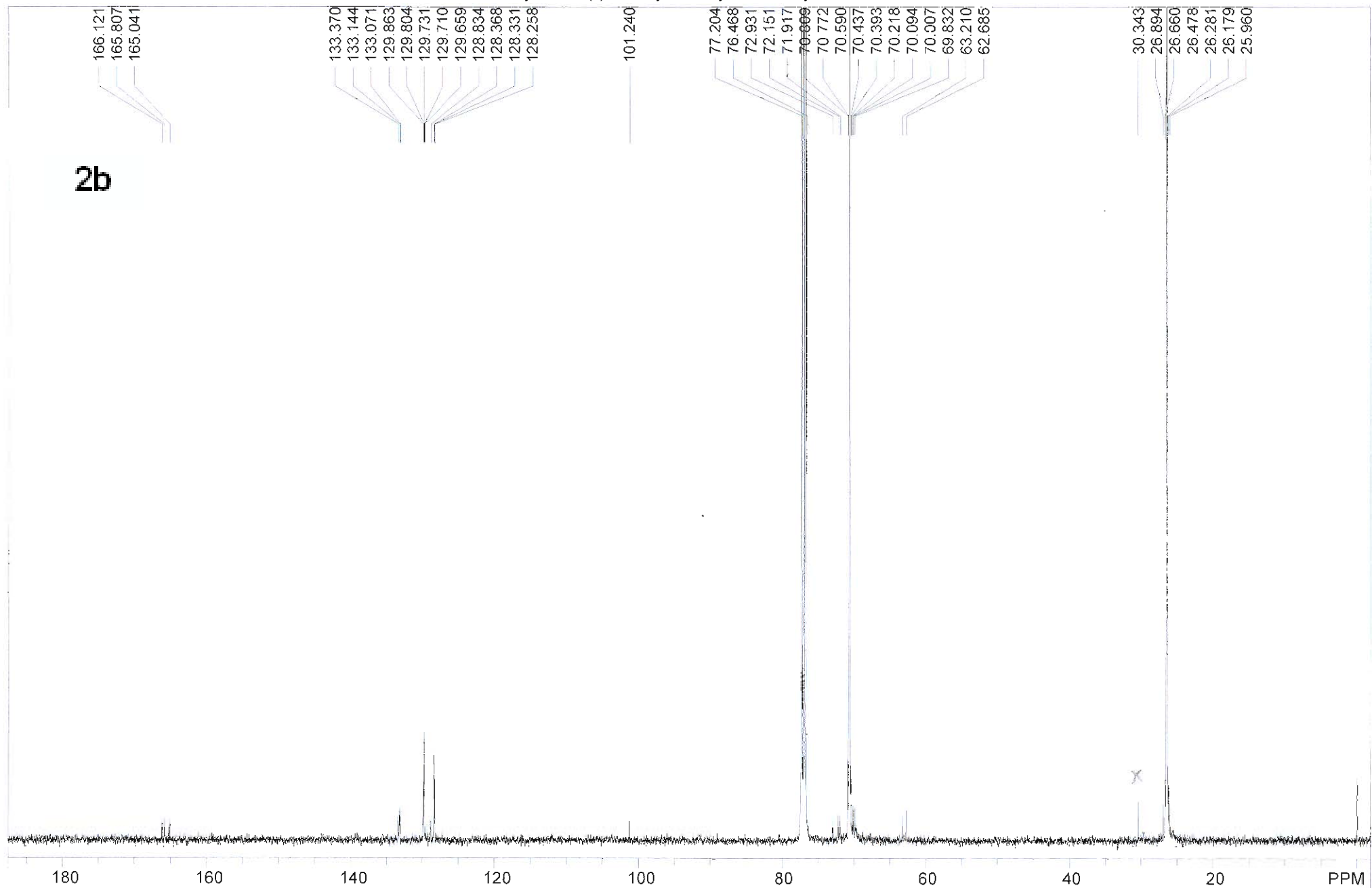
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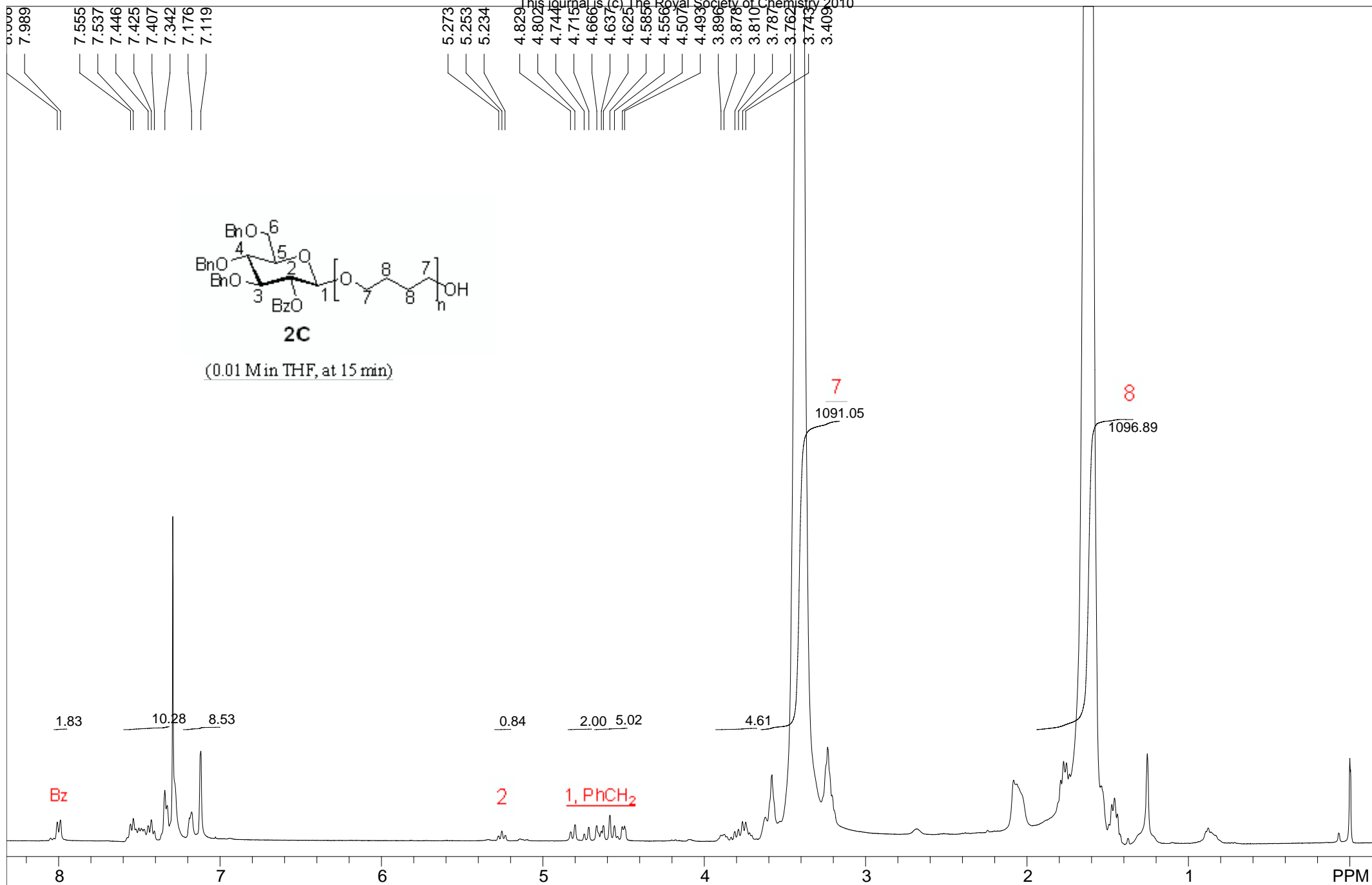
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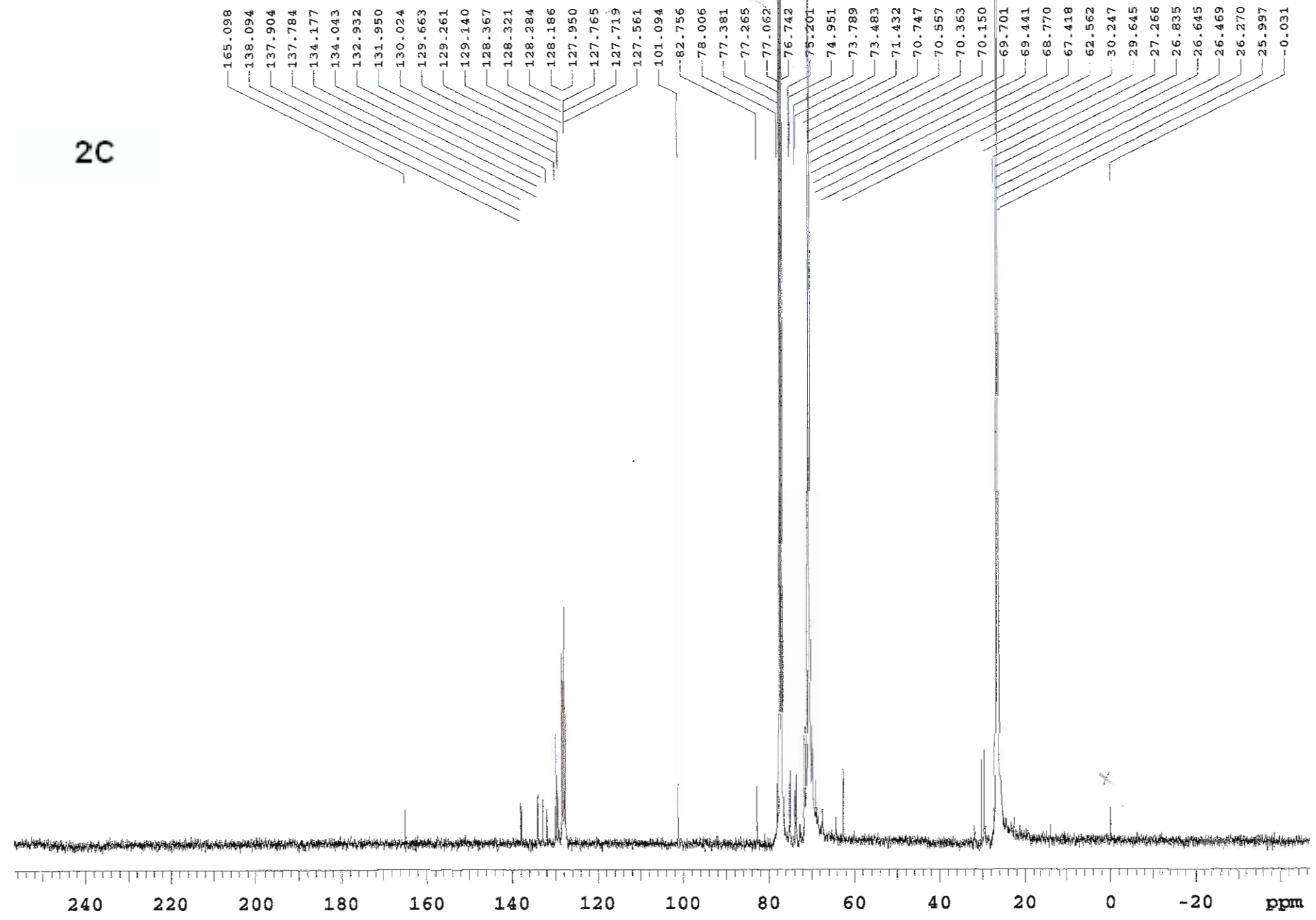


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1484-2P
李浩

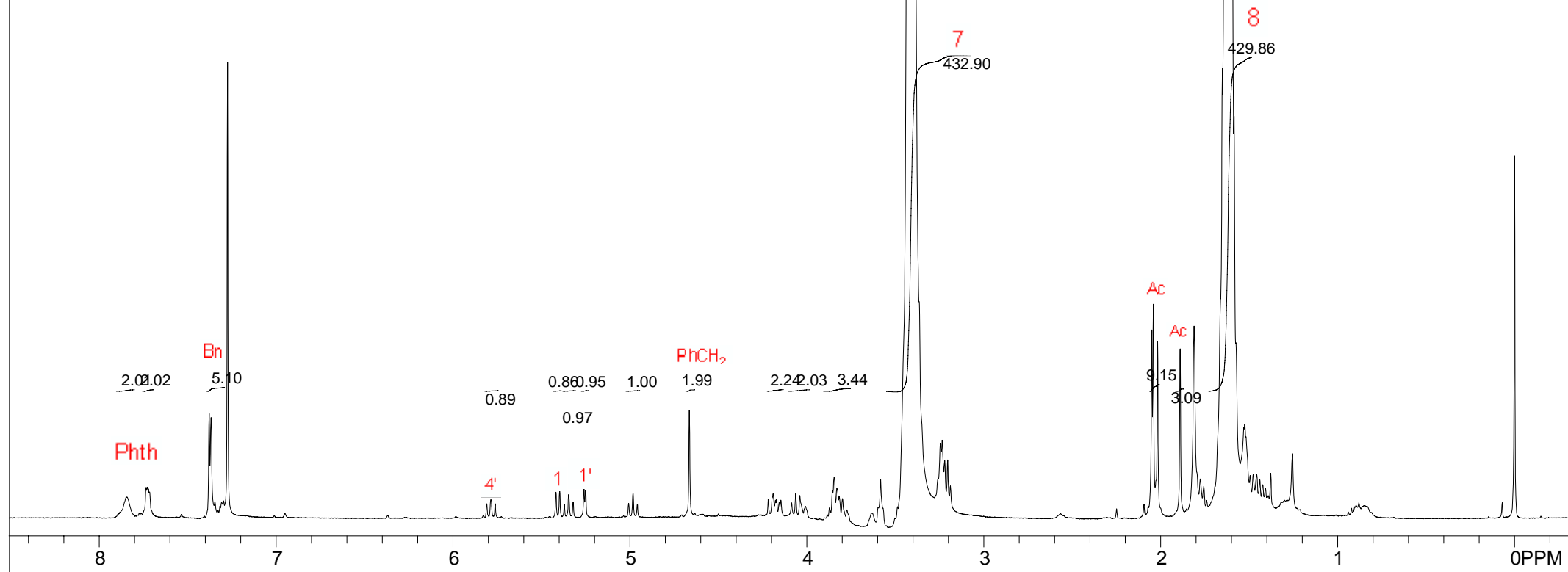
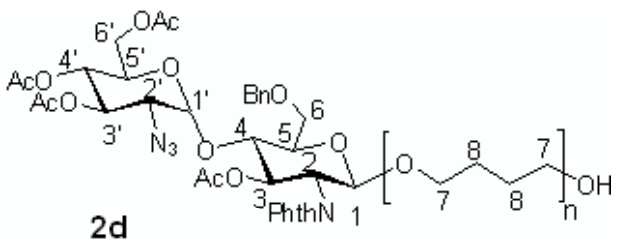
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7.728
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5.321
5.260
5.251
5.007
4.983
4.958
4.664
4.218
4.191
4.177
4.171
4.157
4.147
4.087
4.063
4.040
4.009
4.003
3.856
3.817
3.799
3.412
3.399

2.050
2.042
2.018
1.890
1.652
1.618
1.586
1.575

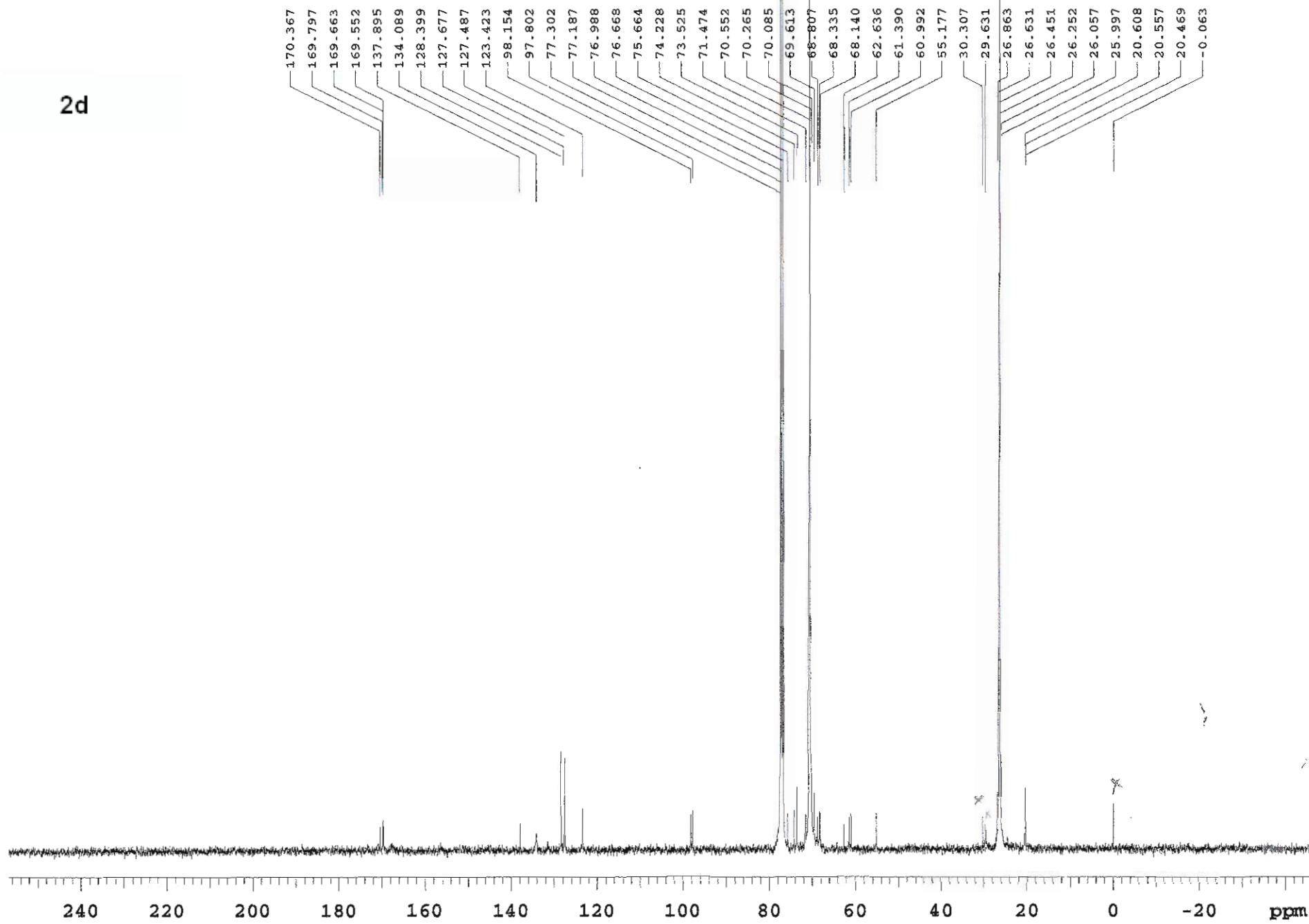


USER: nmr -- DATE: 16:27:23.359 +0800 nmr@EP-ZH107708

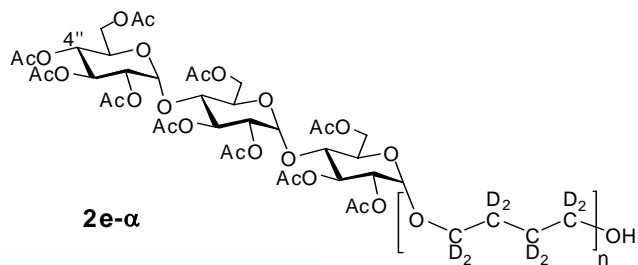
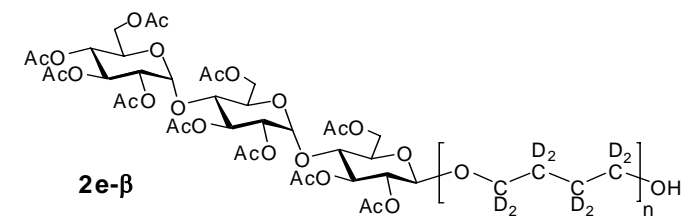
F1: 400.120	F2: 1.000	SW1: 8224	OF1: 2466.9	PTS1d: 32768
EX: zg30	PW: 9.3 us	PD: 1.0 sec	NA: 16	LB: 0.3
				Nuts - \$NMR -1491-2p--.1

1491-2
329

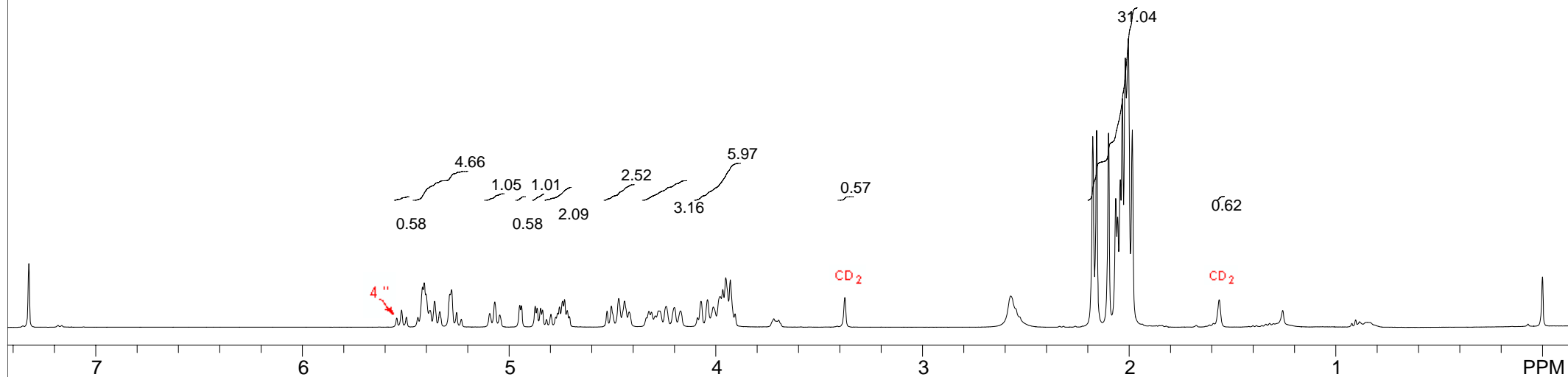
2d



5.544 5.521 5.497 5.442 5.419 5.411 5.402 5.384 5.361 5.336 5.287 5.279 5.254 5.232 5.094 5.070 5.045 4.949 4.940 4.874 4.865 4.848 4.838 4.819 4.798 4.775 4.757 4.708 4.520 4.505 4.469 4.442 4.419 4.336 4.324 4.311 4.293 4.271 4.240 4.200 4.171 4.088 4.071 4.040 4.012 3.980 3.929 3.907 3.719 3.696 3.375 2.176 2.157 2.099 2.065 2.055 2.043 2.033 2.018 2.004 1.984 1.564

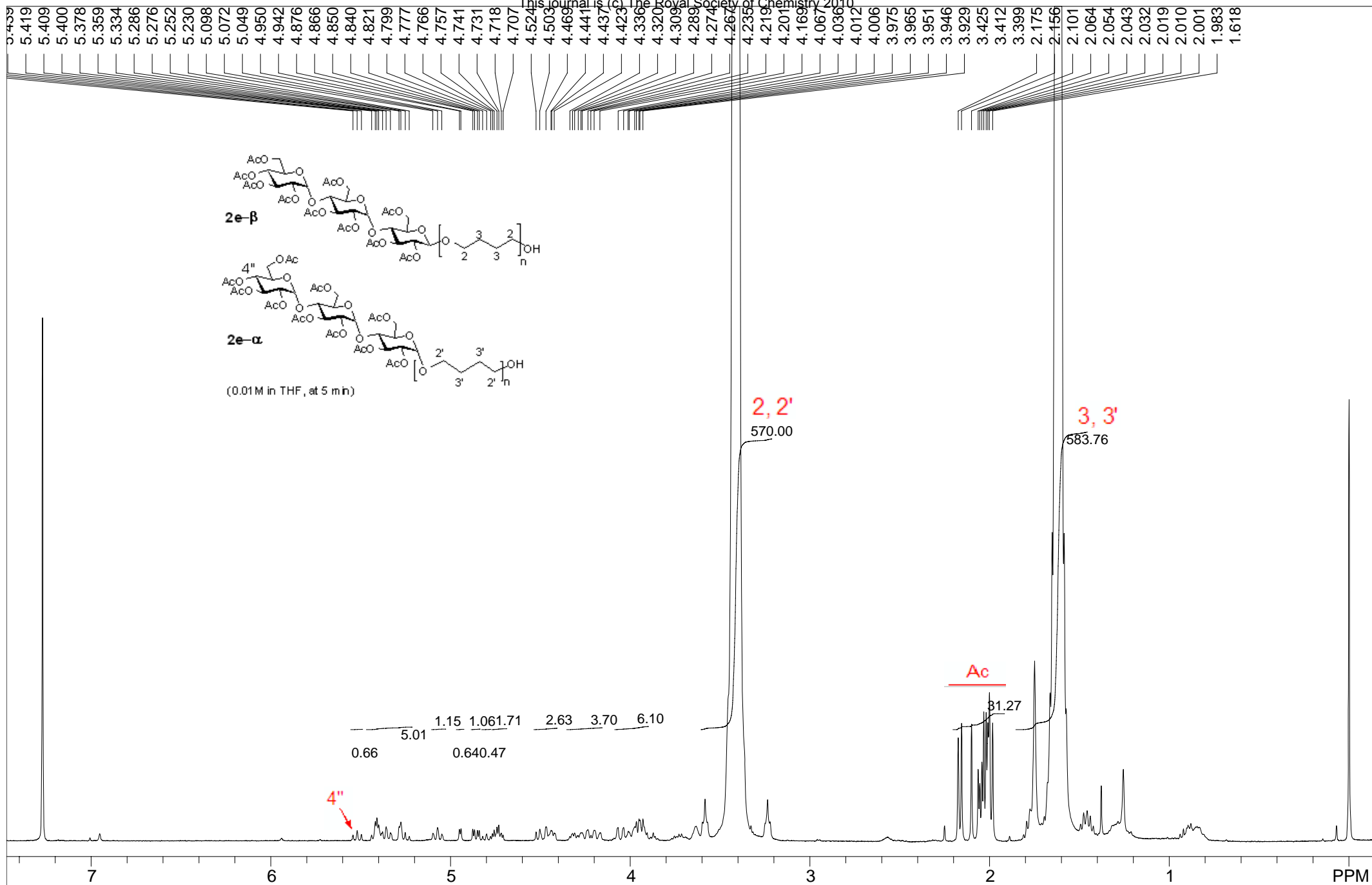


(0.1 M in THF-d₈, at 15 min)



USER: nmr -- DATE: 16:41:32.234 +0800 nmr@EP-ZH107708

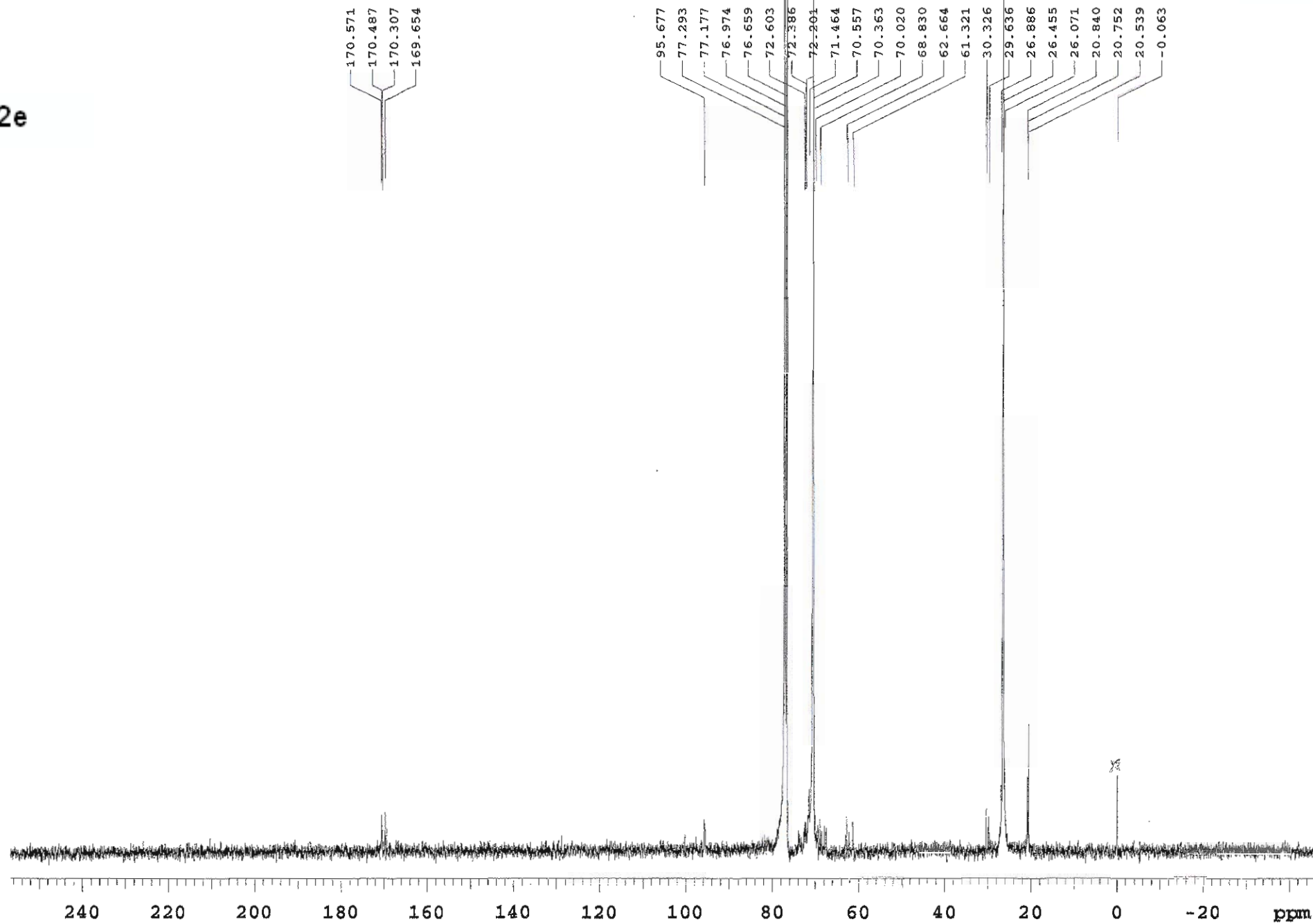
F1: 400.120	F2: 1.000	SW1: 8224	OF1: 2486.9	PTS1d: 32768
EX: zg30	PW: 9.3 us	PD: 1.0 sec	NA: 16	LB: 0.3
				Nuts - \$NMR -1511.1

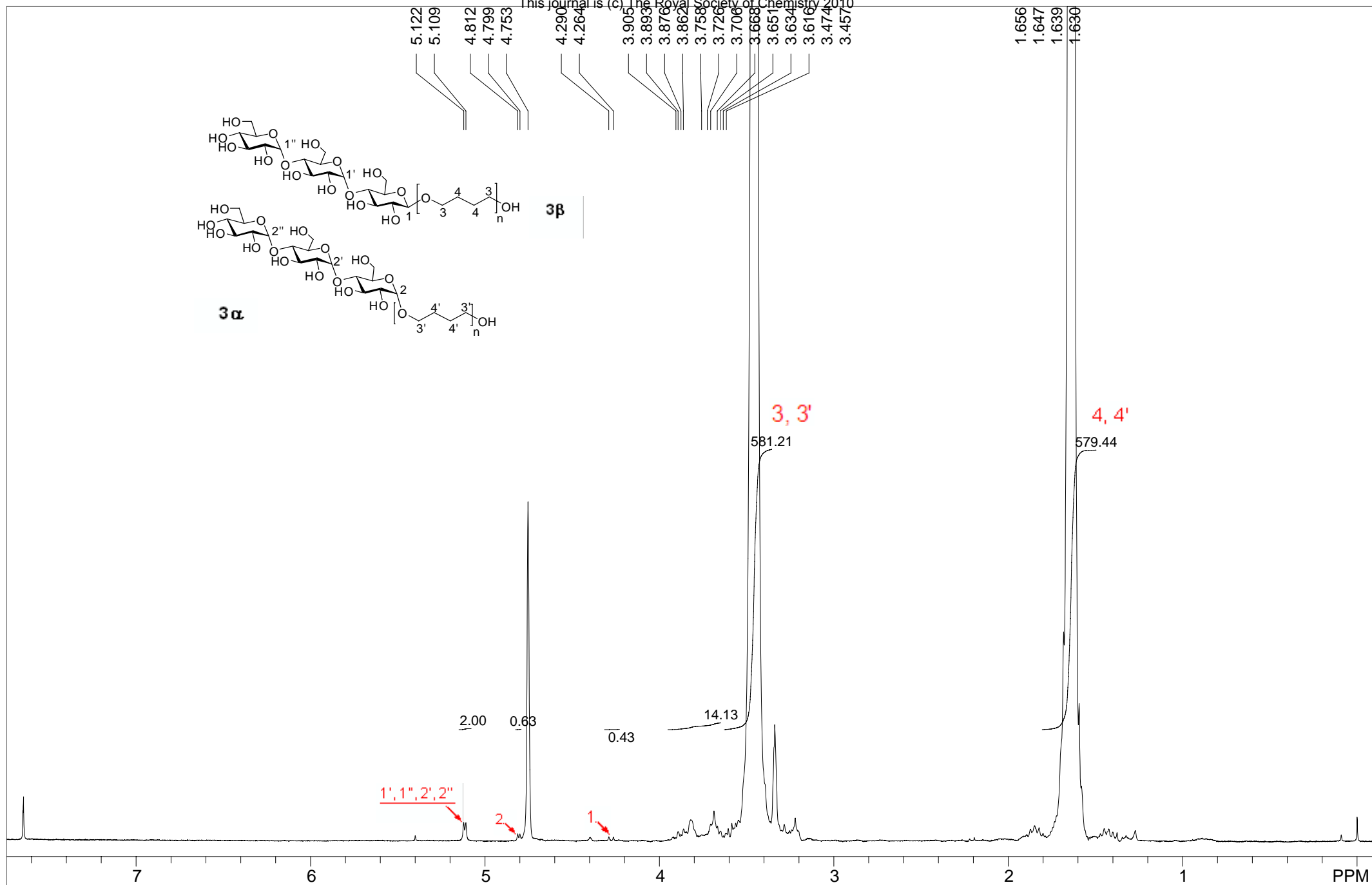


USER: nmr -- DATE: 16:39:59.453 +0800 nmr@EP-ZH107708

F1: 400.120	F2: 1.000	SW1: 8224	OF1: 2465.2	PTS1d: 32768
EX: zg30	PW: 9.3 us	PD: 1.0 sec	NA: 16	LB: 0.3
				Nuts - \$NMR -1495-1p--.1

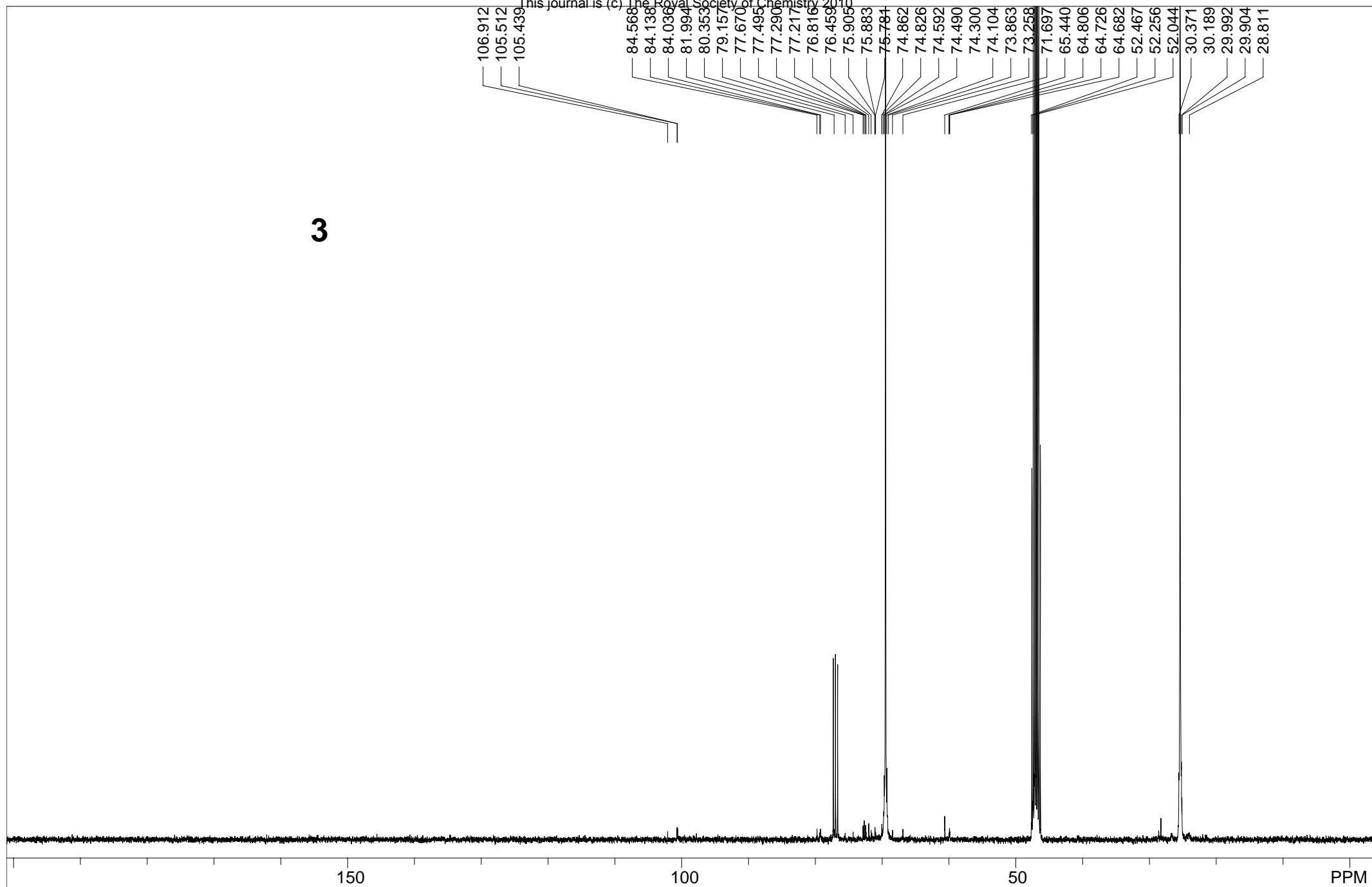
2e





:blank line				USER: -- DATE: Dec 31 2009			
F1: 300.055	F2: 75.456	SW1: 4803		OF1: 629.0		PTS1d: 9587 , 16384	
EX: s2pul	PW: 5.8 us	PD: 1.0 sec		NA: 8	LB: 0.0	Nuts - \$-14100p 31Dec2009	

3



USER: nmr -- DATE: 19:12:39.015 +0800 nmr@EP-ZH107708					
F1: 100.610	F2: 1.000	SW1: 24038	OF1: 9577.7	PTS1d: 32768	
EX: zgpg30	PW: 8.6 us	PD: 2.0 sec	NA: 2024	LB: 1.0	Nuts - \$NMR -14100pC.1