

## Supporting Information

for

### **Chemo-enzymatic modification of poly-*N*-acetyllactosamine (LacNAc) oligomers and *N,N*-diacetyllactosamine (LacDiNAc) based on galactose oxidase treatment**

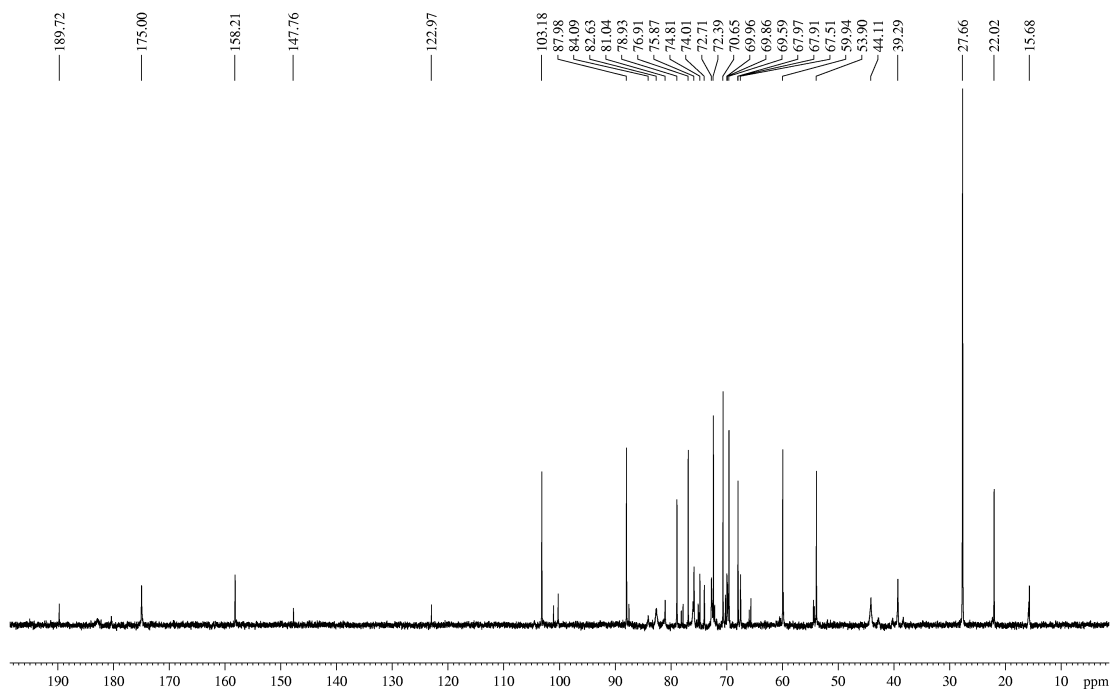
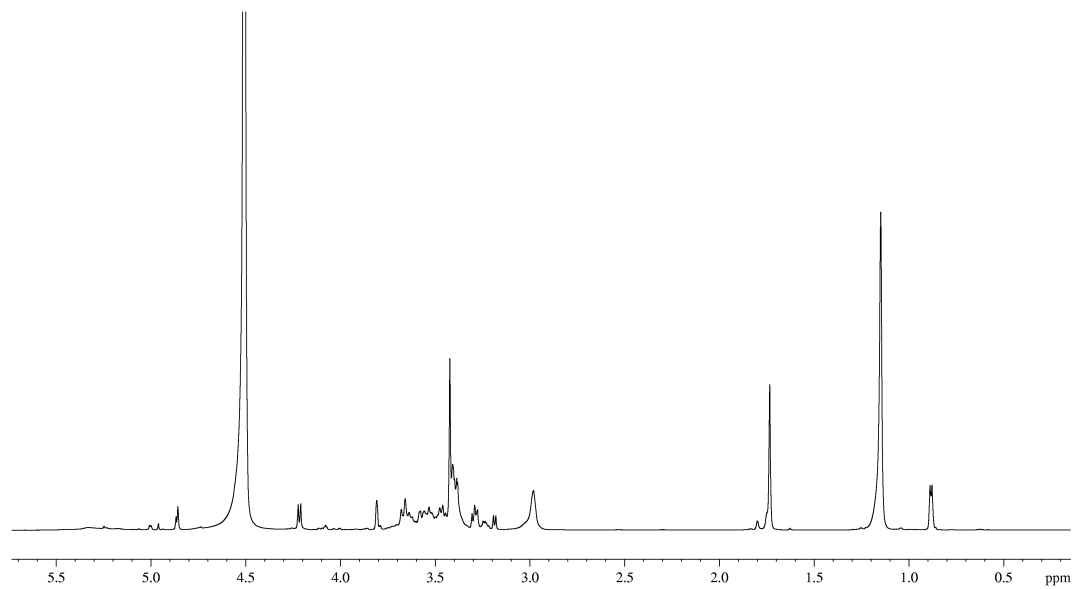
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### **NMR data and spectra**

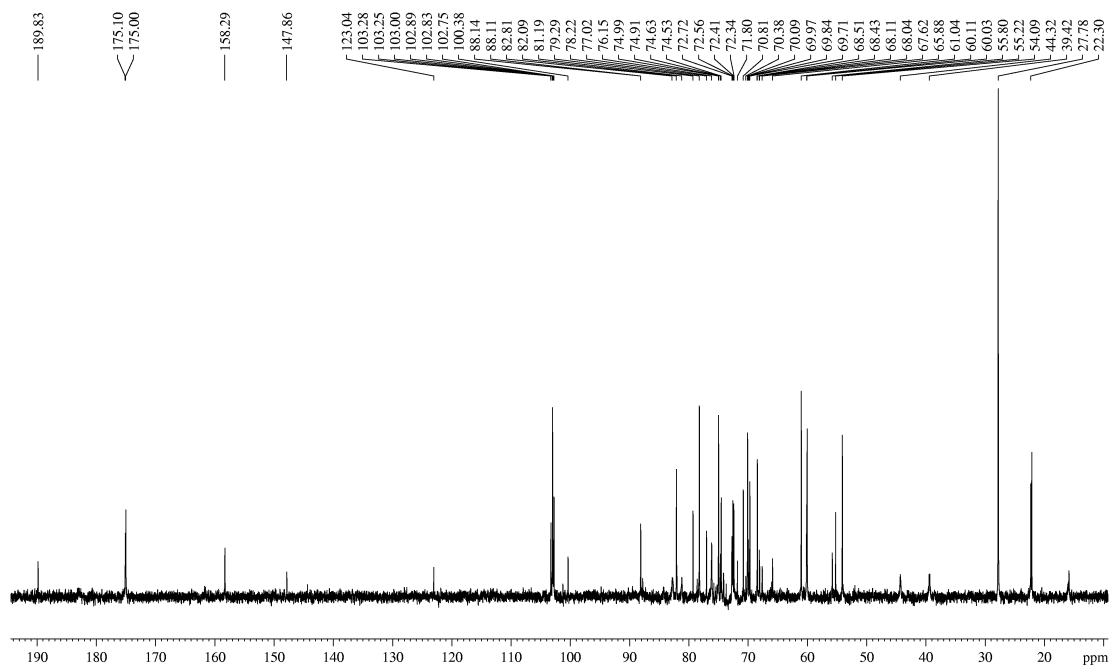
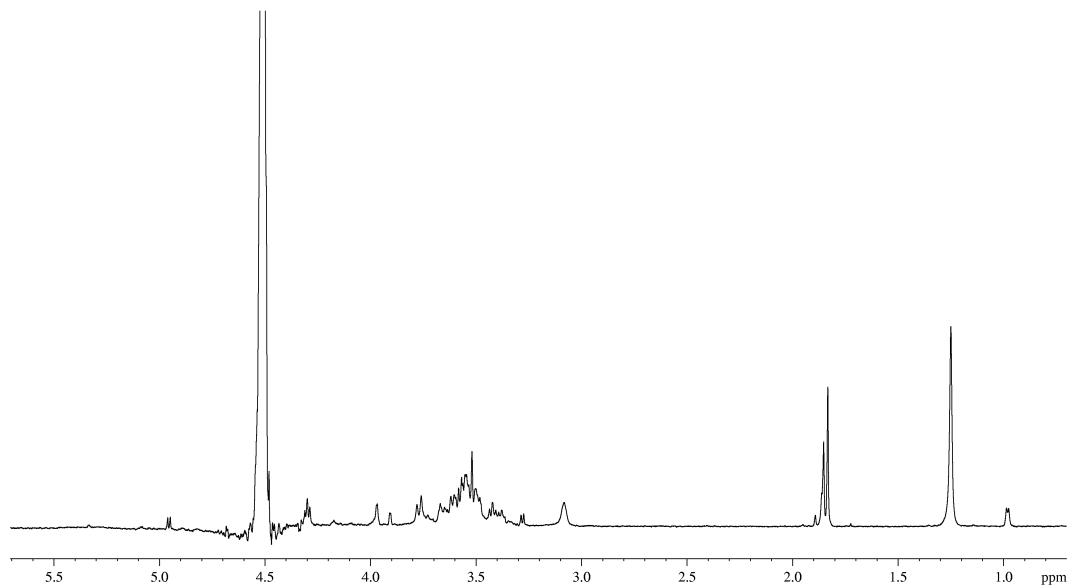


**Figure NMR 1:** <sup>1</sup>H and <sup>13</sup>C NMR spectra of **3a** (600.23 MHz for <sup>1</sup>H, 150.94 MHz for <sup>13</sup>C, D<sub>2</sub>O, 30 °C).

**Table NMR 1:**  $^1\text{H}$  and  $^{13}\text{C}$  NMR data of **3a** (600.23 MHz for  $^1\text{H}$ , 150.94 MHz for  $^{13}\text{C}$ ,  $\text{D}_2\text{O}$ , 30 °C).<sup>a</sup>

	assignment	$\delta_{\text{C}}$	m.	$\delta_{\text{H}}$	$n_{\text{H}}$	m.	$J[\text{Hz}]$	HMBC (C to H)
<b>Boc</b>	<b>CO</b>	158.21	S	-	0			2'
	<b>C</b>	81.04	S	-	0			( $\text{CH}_3$ ) <sub>3</sub>
	<b>(CH<sub>3</sub>)<sub>3</sub></b>	27.66	Q	1.147	9	s		
<b>spacer</b>	<b>1'</b>	44.11	T	3.389	2	m		2 w
	<b>2'</b>	39.29	T	2.980	2	m		
<b>Glc<sup>A</sup></b>	<b>CS</b>	n.d.	S	-	0			
	<b>1</b>	82.63	D	n.d.	1			
	<b>2</b>	53.90	D	3.637	1	m		3, 4 w, 2-Ac
	<b>3</b>	72.71	D	3.532	1	dd	$\Sigma J = 18.0$	4 w
	<b>4</b>	78.93	D	3.460	1	dd	$\Sigma J = 18.1$	<b>1<sup>B</sup></b> , 6u, 6d
	<b>5</b>	75.87	D	3.407	1	m		4
	<b>6</b>	59.94	T	3.668	1	dd		4
				3.570	1	dd	12.5, 4.0	
<b>Gal<sup>B</sup></b>	<b>2-Ac</b>	22.02	Q	1.734	3	s		
	<b>CO</b>	175.00	S	-	0			2-Ac
	<b>1</b>	103.18	D	4.216	1	d	7.8	2, 3, 5, <b>4<sup>A</sup></b>
	<b>2</b>	70.65	D	3.291	1	dd	9.9, 7.8	1 w, 3, 4
	<b>3</b>	72.39	D	3.393	1	dm	9.9	1 w, 3, 4, 5 w
	<b>4</b>	67.97	D	3.808	1	m	$\Sigma J = 5.3$	3, 5, 6 w
	<b>5</b>	76.91	D	3.185	1	m	$\Sigma J = 10.2$	6
	<b>6</b>	87.98	D	4.861	1	dm	7.4, $\Sigma J = 1.8$	4, 5

<sup>a</sup>w: weak signal; n.d.: not detected; u: up-field; d: down-field

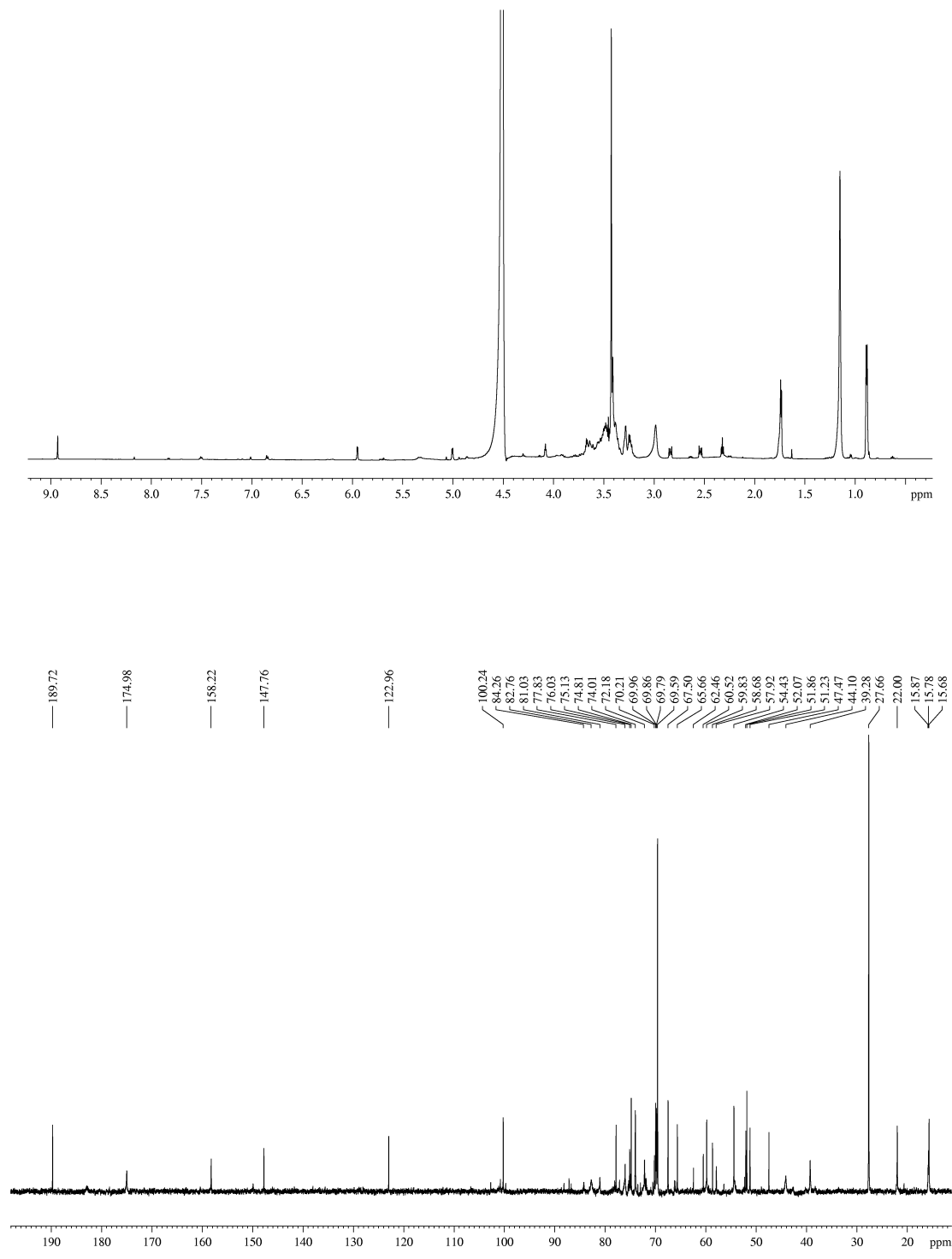


**Figure NMR 2:**  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra of **3b** (600.23 MHz for  $^1\text{H}$ , 150.94 MHz for  $^{13}\text{C}$ ,  $\text{D}_2\text{O}$ , 30 °C).

**Table NMR 2:**  $^1\text{H}$  and  $^{13}\text{C}$  NMR data of **3b** (600.23 MHz for  $^1\text{H}$ , 150.94 MHz for  $^{13}\text{C}$ ,  $\text{D}_2\text{O}$ , 30 °C).<sup>a</sup>

	assignment	$\delta_{\text{C}}$	m.	$\delta_{\text{H}}$	$n_{\text{H}}$	m.	$J[\text{Hz}]$	HMBC (C to H)
<b>Boc</b>	<b>CO</b>	158.29	S	-	0			
	<b>C</b>	81.19	S	-	0			( $\text{CH}_3$ ) <sub>3</sub>
	<b>(CH<sub>3</sub>)<sub>3</sub></b>	27.78	Q	1.249	9	s		
<b>spacer</b>	<b>1'</b>	44.32	T	3.480	2	m		
	<b>2'</b>	39.42	T	3.082	2	m		
<b>Glc<sup>A</sup></b>	<b>CS</b>	n.d.	S	-	0			
	<b>1</b>	82.81	D	n.d.	-			
	<b>2</b>	54.09	D	3.73 <sup>b</sup>	1	m		3, 4, 2-Ac
	<b>3</b>	72.72	D	3.64 <sup>b</sup>	1	m		
	<b>4</b>	78.22	D	3.55 <sup>b</sup>	1	m		6u, 6d, <b>1<sup>B</sup></b>
	<b>5</b>	76.15	D	3.49 <sup>b</sup>	1	m		4, 6d
	<b>6</b>	60.11	T	3.77 <sup>b</sup>	1	m		4
				3.66 <sup>b</sup>	1	m		
	<b>2-Ac</b>	22.13	Q	1.833	3	s		
<b>Gal<sup>B</sup></b>	<b>CO</b>	175.10	S	-	0			2
	<b>1</b>	103.00	D	4.294	1	d	7.2	2, <b>4<sup>A</sup></b>
	<b>2</b>	70.09	D	3.423	1	m		
	<b>3</b>	82.09	D	3.546	1	m		1, 2, 4, <b>1<sup>C</sup></b>
	<b>4</b>	68.43	D	3.970	1	dm	2.4	
	<b>5</b>	74.91	D	3.54 <sup>b</sup>	1	m		1, 4, 6
<b>Glc<sup>C</sup></b>	<b>6</b>	61.04	T	3.57 <sup>b</sup>	2	m		4, 5
	<b>1</b>	102.75	D	4.52 <sup>b</sup>	1	m		2, <b>3<sup>B</sup></b>
	<b>2</b>	55.22	D	3.62 <sup>b</sup>	1	m		3, 2-Ac
	<b>3</b>	72.41	D	3.57 <sup>b</sup>	1	m		
	<b>4</b>	79.29	D	3.54 <sup>b</sup>	1	m		5, 6u, 6d, <b>1<sup>D</sup></b>
	<b>5</b>	74.53	D	3.41 <sup>b</sup>	1	m		6u, 6d
	<b>6</b>	60.03	T	3.77 <sup>b</sup>	1	m		4
			3.66 <sup>b</sup>	1	m			
	<b>2-Ac</b>	22.30	Q	1.852	3	s		
<b>Gal<sup>D</sup></b>	<b>CO</b>	175.01	S	-	0			2, 2-Ac
	<b>1</b>	103.28	D	4.300	1	d	7.0	2, 5
	<b>2</b>	70.81	D	3.379	1	m		
	<b>3</b>	72.56	D	3.488	1	m		
	<b>4</b>	68.11	D	3.907	1	dm	2.6	6
	<b>5</b>	77.02	D	3.280	1	dm	7.4	
	<b>6</b>	88.14	D	4.955	1	d	7.4	1, 5

<sup>a</sup>n.d.: not detected; u: up-field; d: down-field; <sup>b</sup>HSQC readout

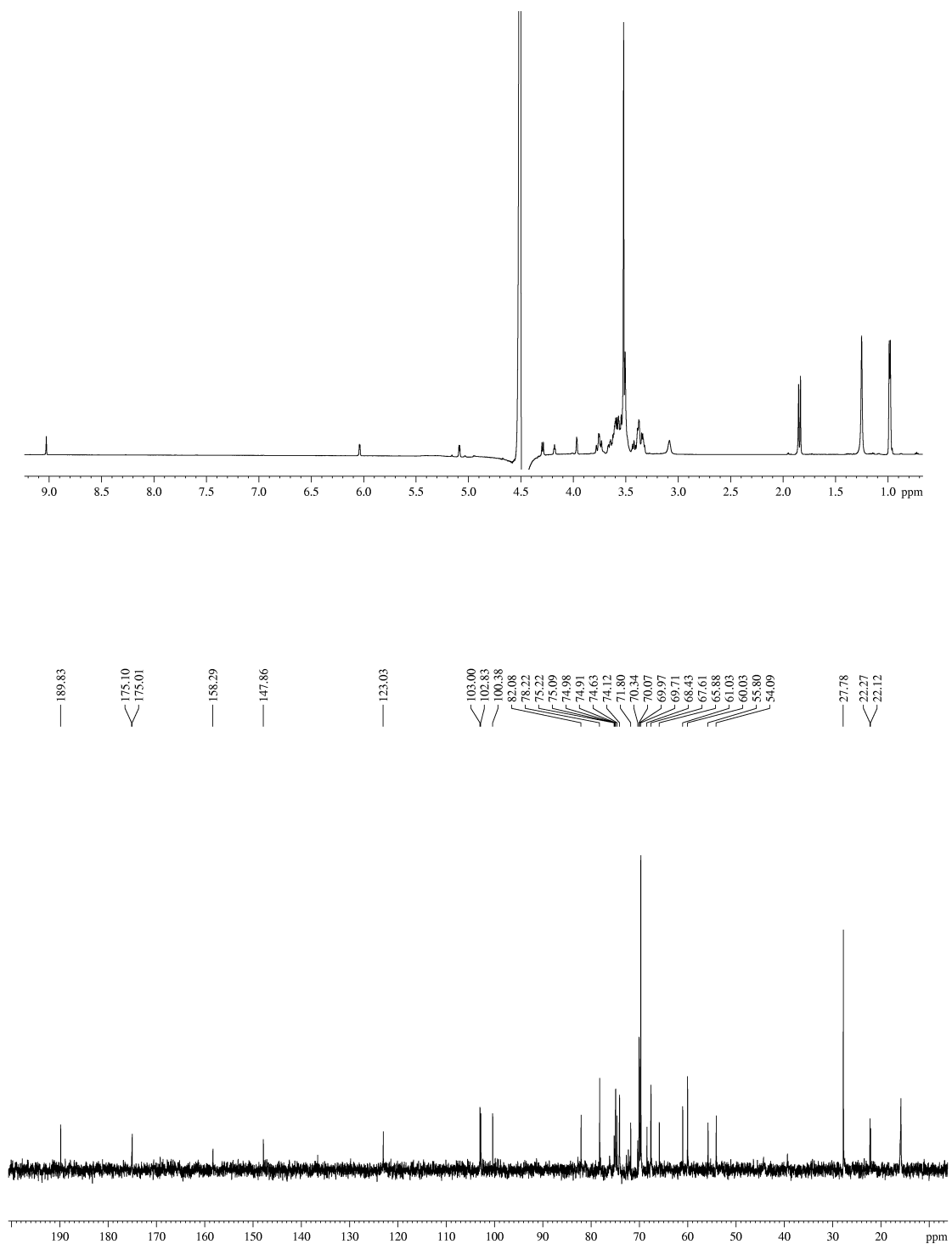


**Figure NMR 3:**  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra of **7a** (600.23 MHz for  $^1\text{H}$ , 150.94 MHz for  $^{13}\text{C}$ , D<sub>2</sub>O, 30 °C).

**Table NMR 3:**  $^1\text{H}$  and  $^{13}\text{C}$  NMR data of **7a** (600.23 MHz for  $^1\text{H}$ , 150.94 MHz for  $^{13}\text{C}$ ,  $\text{D}_2\text{O}$ , 30 °C).<sup>a</sup>

	assignment	$\delta_{\text{C}}$	m.	$\delta_{\text{H}}$	$n_{\text{H}}$	m.	$J[\text{Hz}]$	HMBC (C to H)
<b>Boc</b>	<b>CO</b>	158.22	S	-	0			2'
	<b>C</b>	81.03	S	-	0			( $\text{CH}_3$ ) <sub>3</sub>
<b>spacer</b>	<b>(CH<sub>3</sub>)<sub>3</sub></b>	27.66	Q	1.149	9	s		
	<b>1'</b>	44.10	T	3.39 <sup>b</sup>	2	m		
	<b>2'</b>	39.28	T	2.983	2	m		
<b>Glc<sup>A</sup></b>	<b>CS</b>	n.d.	S	-	0			
	<b>1</b>	82.76	D	n.d.	1			
	<b>2</b>	54.48	D	3.62 <sup>b</sup>	1	m		2-Ac w
	<b>3</b>	75.13	D	3.50 <sup>b</sup>	1	m		
	<b>4</b>	77.83	D	3.53 <sup>b</sup>	1	m		1 <sup>B</sup> , 6d, 5w
	<b>5</b>	76.03	D	3.38 <sup>b</sup>	1	m		
	<b>6</b>	59.83	T	3.65 <sup>b</sup>	1	m		
				3.56 <sup>b</sup>	1	m		
	<b>2-Ac</b>	22.00	Q	1.730	3	s		
<b>Gal<sup>B</sup></b>	<b>CO</b>	174.98	S	-	0			2-Ac
	<b>1</b>	100.24	D	5.005	1	d	5.0	2, 3, 6, 4 <sup>A</sup>
	<b>2</b>	69.79	D	3.671	1	dd	5.0, 4.6	
	<b>3</b>	65.66	D	4.079	1	dd	4.6, 3.9	
	<b>4</b>	122.96	D	5.949	1	d	3.9	2, 3
	<b>5</b>	147.76	S	-	0			1, 3, 4, 6
	<b>6</b>	189.72	D	8.931	1	s		4, 3

<sup>a</sup>n.d.: not detected; u: up-field; d: down-field; <sup>b</sup>HSQC readout



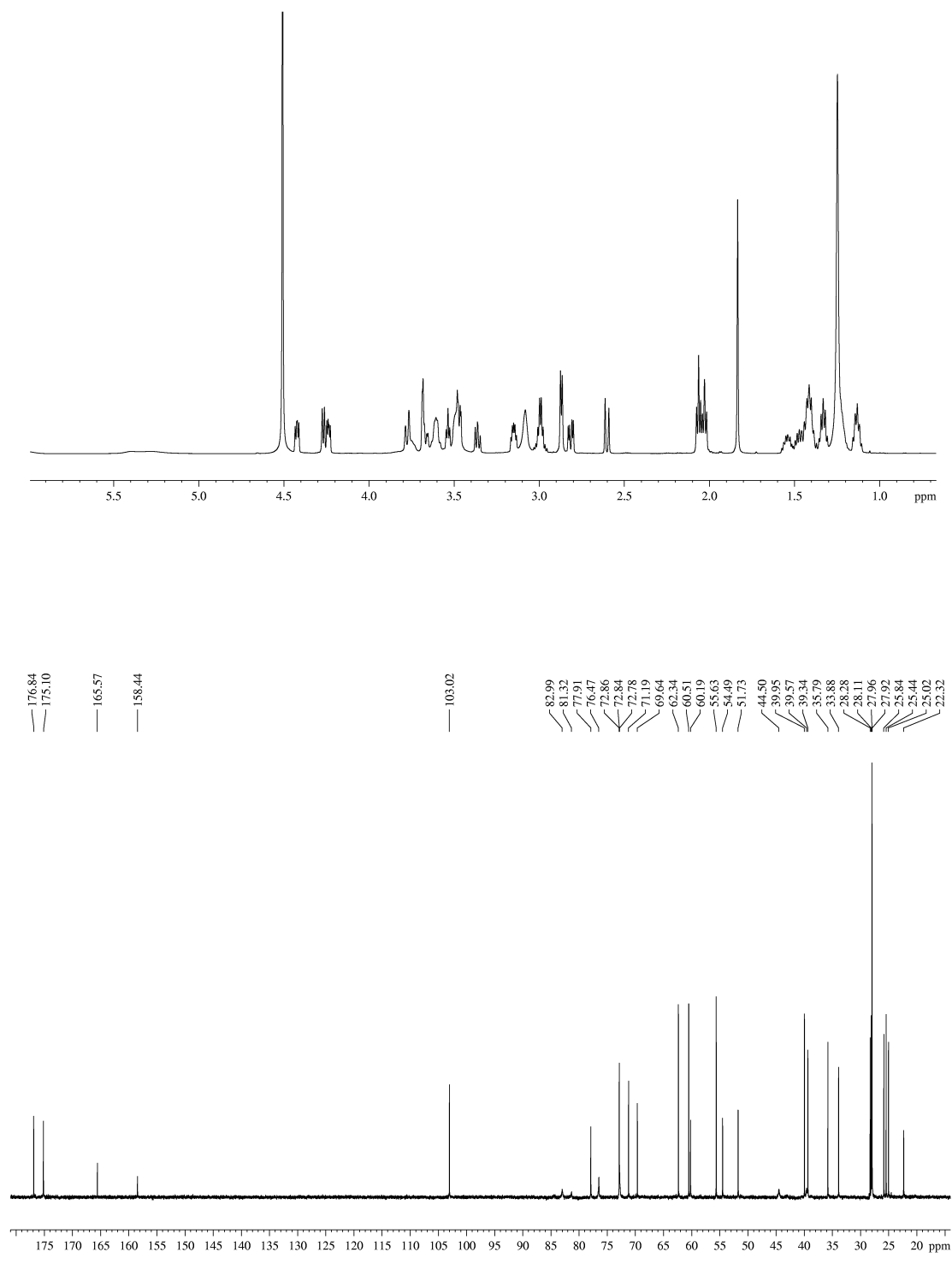
**Figure NMR 4:** <sup>1</sup>H and <sup>13</sup>C NMR spectra of **7b** (600.23 MHz for <sup>1</sup>H, 150.94 MHz for <sup>13</sup>C, D<sub>2</sub>O, 30 °C).



**Table NMR 4:**  $^1\text{H}$  and  $^{13}\text{C}$  NMR data of **7b** (600.23 MHz for  $^1\text{H}$ , 150.94 MHz for  $^{13}\text{C}$ ,  $\text{D}_2\text{O}$ , 30 °C).

	assignment	$\delta_{\text{C}}$	m.	$\delta_{\text{H}}$	$n_{\text{H}}$	m.	$J[\text{Hz}]$	HMBC (C to H)
<b>Boc</b>	<b>CO</b>	158.29	S	-	0			
	<b>C</b>	81.2 <sup>a</sup>	S	-	0			( $\text{CH}_3$ ) <sub>3</sub>
	<b>(CH<sub>3</sub>)<sub>3</sub></b>	27.78	Q	1.250	9	s		
<b>spacer</b>	<b>1'</b>	44.3 <sup>a</sup>	T	3.48 <sup>b</sup>	2	m		
	<b>2'</b>	39.3 <sup>a</sup>	T	3.083	2	m		
<b>Glc<sup>A</sup></b>	<b>CS</b>	n.d. <sup>c</sup>	-	-	0			
	<b>1</b>	n.d. <sup>c</sup>	-	n.d.	-			
	<b>2</b>	54.09	D	3.74 <sup>b</sup>	1	m		
	<b>3</b>	72.7 <sup>a</sup>	D	3.63 <sup>b</sup>	1	m		
	<b>4</b>	78.22	D	3.55 <sup>b</sup>	1	m		<b>1<sup>B</sup></b>
	<b>5</b>	76.12	D	3.49 <sup>b</sup>	1	m		
	<b>6</b>	60.03	T	3.77 <sup>b</sup>	1	m		4
<b>Gal<sup>B</sup></b>				3.65 <sup>b</sup>	1	m		
	<b>2-Ac</b>	22.12	Q	1.833	3	s		
	<b>CO</b>	175.10	S	-	0			2-Ac
	<b>1</b>	103.00	D	4.292	1	d	7.9	2, 3
	<b>2</b>	69.97	D	3.419	1	dd	10.9, 7.9	1, 3, 4
	<b>3</b>	82.08	D	3.53 <sup>b</sup>	1	m		1, 2, 4, <b>1<sup>C</sup></b>
	<b>4</b>	68.43	D	3.966	1	d	2.9	3
<b>Glc<sup>C</sup></b>	<b>5</b>	75.09	D	3.54 <sup>b</sup>	1	m		
	<b>6</b>	61.03	T	3.57 <sup>b</sup>	2	m		4, 5
	<b>1</b>	102.83	D	4.51 <sup>b</sup>	1	m		2, <b>3<sup>B</sup></b>
	<b>2</b>	55.80	D	3.60 <sup>b</sup>	1	m		3
	<b>3</b>	71.80	D	3.50 <sup>b</sup>	1	m		
	<b>4</b>	78.22	D	3.61 <sup>b</sup>	1	m		<b>1<sup>D</sup></b>
	<b>5</b>	74.63	D	3.38 <sup>b</sup>	1	m		
<b>Gal<sup>D</sup></b>	<b>6</b>	60.03	T	3.74 <sup>b</sup>	1	m		4
				3.64 <sup>b</sup>	1	m		
	<b>2-Ac</b>	22.27	Q	1.851	3	s		
	<b>CO</b>	175.01	S	-	0			2, 2-Ac
	<b>1</b>	100.38	D	5.086	1	d	5.1	2, 3, 6, <b>4<sup>C</sup></b>
	<b>2</b>	70.07	D	3.752	1	dd	5.1, 4.7	1, 3, 4
	<b>3</b>	65.88	D	4.179	1	dd	4.7, 3.8	2, 4
<b>4</b>	123.03	D	6.037	1	d	3.8	2, 3	
<b>5</b>	147.86	S	-	0			1, 3, 4	
<b>6</b>	189.83	D	9.026	1	s		5	

<sup>a</sup>broad signal; <sup>b</sup>HSQC readout; <sup>c</sup>n.d.: not detected

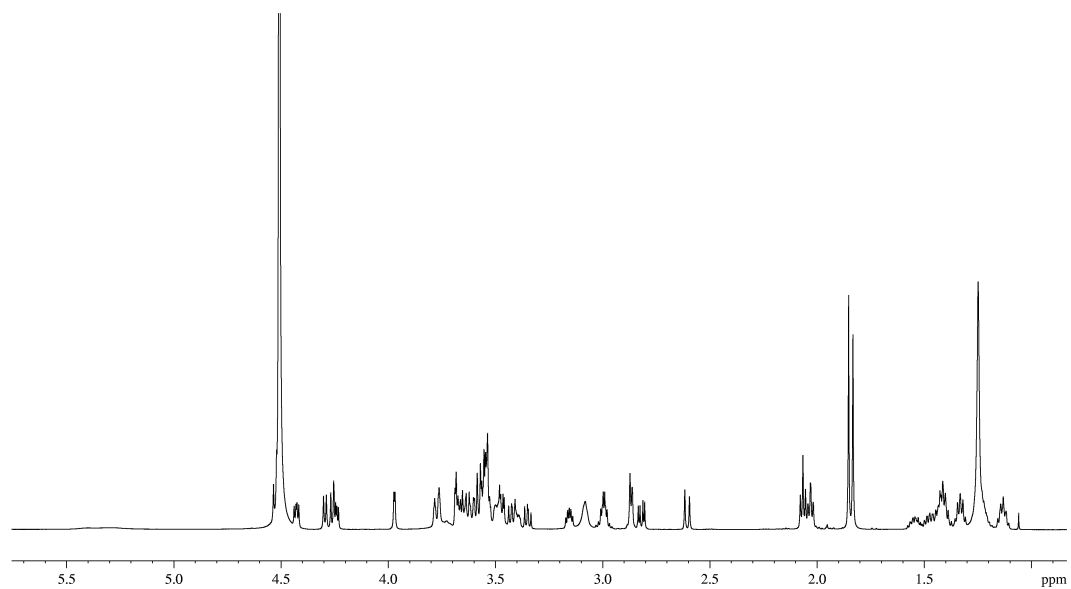


**Figure NMR 5:**  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra of **15a** (600.23 MHz for  $^1\text{H}$ , 150.94 MHz for  $^{13}\text{C}$ ,  $\text{D}_2\text{O}$ ,  $30\text{ }^\circ\text{C}$ ).

**Table NMR 5:**  $^1\text{H}$  and  $^{13}\text{C}$  NMR data of **15a** (600.23 MHz for  $^1\text{H}$ , 150.94 MHz for  $^{13}\text{C}$ ,  $\text{D}_2\text{O}$ , 30 °C).

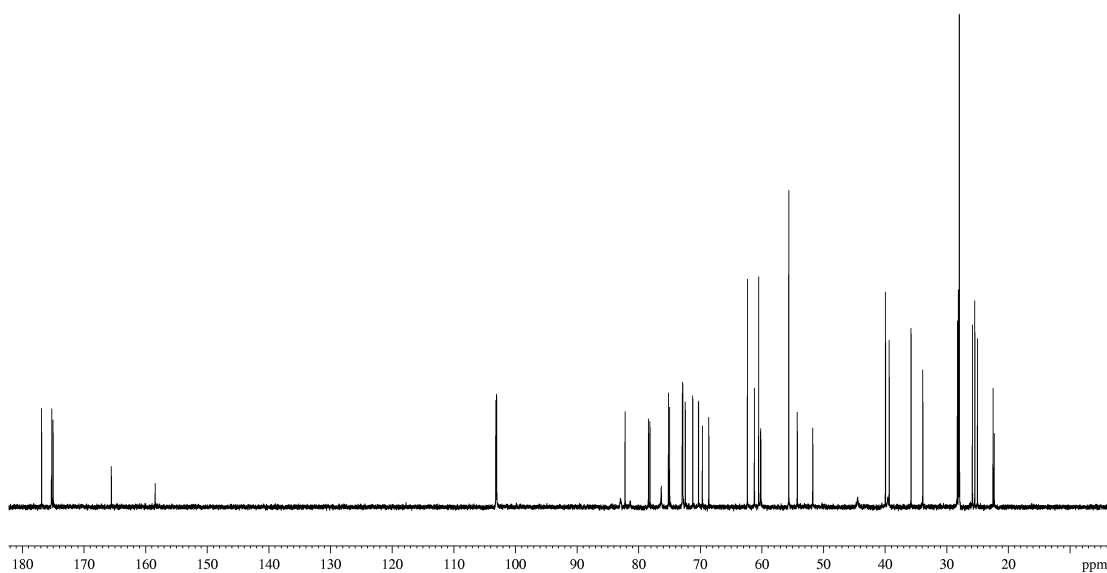
	assignment	$\delta_{\text{C}}$	m.	$\delta_{\text{H}}$	$n_{\text{H}}$	m.	$J[\text{Hz}]$	HMBC (C to H) <sup>d</sup>
<b>Boc</b>	<b>CO</b>	158.44	S	-	0			2' w
	<b>C</b>	81.32	S	-	0			(CH <sub>3</sub> ) <sub>3</sub>
	<b>(CH<sub>3</sub>)<sub>3</sub></b>	27.96	Q	1.247	9	s		(CH <sub>3</sub> ) <sub>3</sub>
<b>spacer</b>	<b>1'</b>	44.50	T	3.48 <sup>a</sup>	2	m		
	<b>2'</b>	39.57	T	3.081	2	m		
<b>Glc<sup>A</sup></b>	<b>CS</b>	183.1 <sup>b</sup>	S	-	0			
	<b>1</b>	82.99	D	5.40, 5.28 <sup>a</sup>	1	br s		
	<b>2</b>	54.49	D	3.75 <sup>a</sup>	1	m		2-Ac
	<b>3</b>	72.78	D	3.61 <sup>a</sup>	1	m		
	<b>4</b>	77.91	D	3.60 <sup>a</sup>	1	m		5, 6d, 1 <sup>B</sup>
	<b>5</b>	76.47	D	3.49 <sup>a</sup>	1	m		6d
	<b>6</b>	60.19	T	3.776	1	dd	12.4, 1.5	
				3.668	1	dd	12.4, 4.4	
<b>Gal<sup>B</sup></b>	<b>2-CO</b>	175.10	S	-	0			2-Ac
	<b>2-Ac</b>	22.32	Q	1.834	3	s		
	<b>1</b>	103.22	D	4.269	1	d	7.8	2, 3, 5, 4 <sup>A</sup>
	<b>2</b>	71.19	D	3.361	1	dd	10.0, 7.8	1, 3, 4
	<b>3</b>	72.84 <sup>c</sup>	D	3.471	1	dd	10.0, 3.3	
	<b>4</b>	69.64	D	3.685	1	d	3.3	3, 6
	<b>5</b>	72.86 <sup>c</sup>	D	3.581	1	t	6.2	
<b>spacer</b>	<b>6</b>	51.73	T	2.870	2	d	6.2	4, 5
	<b>1''</b>	175.10	S	-	0			2'', 3''
	<b>2''</b>	33.88	T	2.028	2	t		3'', 4''
	<b>3''</b>	25.02	T	1.416	2	m		2'', 4'', 5''
	<b>4''</b>	25.84	T	1.131	2	m		2'', 3'', 5'', 6''
	<b>5''</b>	28.28	T	1.331	2	m		6''
	<b>6''</b>	39.34	T	3.011	1	dt	13.5, 6.8	2, 4'', 5''
<b>biotin</b>				2.979	1	dt	13.5, 6.8	
	<b>1</b>	176.84	S	-	0			6'', 2, 3u, 3d
	<b>2</b>	35.79	T	2.063	2	m		3u, 3d, 4
	<b>3</b>	25.44	T	1.46 <sup>a</sup>	2	m		2, 4, 5u, 5d
	<b>4</b>	28.11	T	1.230	2	m		2
	<b>5</b>	27.95	T	1.547	1	m		6, 7, 9u
				1.404	1	m		
	<b>6</b>	55.63	D	3.150	1	m		4, 5u, 5d, 7, 8,
	<b>7</b>	62.34	D	4.238	1	dd	7.8, 4.5	5u, 5d, 8, 9u, 9d
	<b>8</b>	60.51	D	4.423	1	dd	7.8, 5.0	7, 9u
<b>9</b>	39.95	T	2.815	1	dd	13.0, 5.0	7, 8	
			2.602	1	d	13.0		
	<b>CO(NH)<sub>2</sub></b>	165.57	S	-	0			7, 8

<sup>a</sup>HSQC readout; <sup>b</sup>broad signal; <sup>c</sup>might be interchanged; <sup>d</sup>u: up-field, d: down-field



176.87  
 175.26  
 175.19  
 174.99  
 165.58  
 158.46

103.18  
 103.09  
 103.03  
 82.90  
 81.38  
 78.40  
 78.16  
 76.31  
 75.16  
 74.97  
 72.92  
 72.84  
 72.43  
 71.21  
 70.28  
 69.66  
 68.62  
 62.34  
 61.21  
 60.51  
 60.21  
 60.17  
 55.64  
 54.26  
 51.75  
 44.46  
 39.95  
 39.57  
 39.34  
 35.78  
 33.89  
 28.27  
 28.09  
 27.95  
 27.92  
 25.83  
 25.44  
 25.00  
 22.48  
 22.30



**Figure NMR 6:**  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra of **15b** (600.23 MHz for  $^1\text{H}$ , 150.94 MHz for  $^{13}\text{C}$ ,  $\text{D}_2\text{O}$ , 30 °C).

**Table NMR 6:**  $^1\text{H}$  and  $^{13}\text{C}$  NMR data of **15b** (600.23 MHz for  $^1\text{H}$ , 150.94 MHz for  $^{13}\text{C}$ ,  $\text{D}_2\text{O}$ , 30 °C).

	assignment	$\delta_{\text{C}}$	m.	$\delta_{\text{H}}$	$n_{\text{H}}$	m.	$J[\text{Hz}]$	HMBC (C to H) <sup>c</sup>
<b>Boc</b>	<b>CO</b>	158.46	S	-	0			2' w
	<b>C</b>	81.38	S	-	0			(CH <sub>3</sub> ) <sub>3</sub>
	<b>(CH<sub>3</sub>)<sub>3</sub></b>	27.95	Q	1.249	9	s		(CH <sub>3</sub> ) <sub>3</sub>
<b>spacer</b>	<b>1'</b>	44.46	T	3.483	2	m		
	<b>2'</b>	39.57	T	3.082	2	m		
<b>Glc<sup>A</sup></b>	<b>CS</b>	183.1 <sup>b</sup>	S	-	0			
	<b>1</b>	82.90	D	5.29, 5.41 <sup>a</sup>	1	br s		
	<b>2</b>	54.26	D	3.73 <sup>a</sup>	1	m		4 w, 2-Ac
	<b>3</b>	72.88	D	3.62 <sup>a</sup>	1	m		<b>1<sup>B</sup></b>
	<b>4</b>	78.40	D	3.55 <sup>a</sup>	1	m		4, 6d
	<b>5</b>	76.31	D	3.50 <sup>a</sup>	1	m		
	<b>6</b>	60.21	T	3.77 <sup>a</sup>	1	m		
				3.66 <sup>a</sup>	1	m		
<b>Gal<sup>B</sup></b>	<b>2-CO</b>	175.26	S	-	0			2-Ac
	<b>2-Ac</b>	22.30	Q	1.833	3	s		
	<b>1</b>	103.19	D	4.296	1	d	7.9	2, <b>4<sup>A</sup></b>
	<b>2</b>	70.28	D	3.424	1	dd	9.9, 7.9	1, 3, 4
	<b>3</b>	82.20	D	3.547	1	m		1, 2, 4, <b>1<sup>C</sup></b>
	<b>4</b>	68.62	D	3.972	1	d	3.3	3
<b>Glc<sup>C</sup></b>	<b>5</b>	75.16	D	3.542	1	m		1, 4, 6
	<b>6</b>	61.21	T	3.569	2	m		4, 5
	<b>1</b>	103.03	D	4.529	1	d	8.5	2, <b>3<sup>B</sup></b>
	<b>2</b>	55.64	D	3.638	1	dd	9.9, 8.5	1, 3
	<b>3</b>	72.43	D	3.537	1	dd	9.9, 9.5	1, 2, 4
	<b>4</b>	78.16	D	3.585	1	m		3, 6d, <b>1<sup>D</sup></b>
<b>Gal<sup>D</sup></b>	<b>5</b>	74.94	D	3.401	1	ddd	9.7, 4.6,	1, 4, 6u, 6d
	<b>6</b>	60.17	T	3.776	1	m		4
					3.670	1	m	
	<b>2-CO</b>	174.99	S	-	0			2-Ac
	<b>2-Ac</b>	22.48	Q	1.853	3	s		
	<b>1</b>	103.09	D	4.262	1	d	7.9	2, <b>4<sup>C</sup></b>
<b>spacer</b>	<b>2</b>	71.21	D	3.349	1	dd	9.9, 7.9	1, 3, 4
	<b>3</b>	72.84	D	3.471	1	dd	9.9, 3.4	1, 2
	<b>4</b>	69.66	D	3.686	1	m		3, 5, 6
	<b>5</b>	72.92	D	3.535	1	m		6
	<b>6</b>	51.75	T	2.868	2	d	6.2	4, 5
	<b>1''</b>	175.19	S	-	0			2'', 3''
<b>spacer</b>	<b>2''</b>	33.89	T	2.032	2	m		3'', 4''
	<b>3''</b>	25.00	T	1.415	2	m		2'', 4'', 5''
	<b>4''</b>	25.83	T	1.132	2	m		2'', 3'', 5'', 6''
	<b>5''</b>	28.27	T	1.333	2	m		4'', 6''
	<b>6''</b>	39.34	T	3.009	1	dt	13.5, 6.8	4'', 5'', <b>2</b>
					2.908	1	dt	13.5, 6.8

<b>biotin</b>	<b>1</b>	176.87	S	-	0			2, 3, <b>6''</b>
	<b>2</b>	35.78	T	2.066	2	m		3, 4
	<b>3</b>	25.44	T	1.46 <sup>a</sup>	2	m		2, 4, 5u, 5d
	<b>4</b>	28.09	T	1.231	2	m		2, 6
	<b>5</b>	27.92	T	1.547	1	m		6, 7 w, 9u w
				1.405	1	m		
	<b>6</b>	55.64	D	3.156	1	m		4, 5u, 5d, 7, 8,
	<b>7</b>	62.34	D	4.243	1	dd	7.9, 4.6	5u, 5d, 8, 9u
	<b>8</b>	60.51	D	4.429	1	ddd	7.9, 5.0,	7, 9u
<b>9</b>	39.95	T	2.819	1	dd	13.0, 5.0	7, 8	
			2.607	1	d	13.0		
<b>CO(NH)<sub>2</sub></b>	165.58	S	-	0				7, 8, 9u

<sup>a</sup>HSQC readout; <sup>b</sup>broad signal; <sup>c</sup>w: weak signal, u: up-field, d: down-field

**Table NMR 7:** <sup>15</sup>N NMR data of **15b** (600.23 MHz for <sup>1</sup>H, 60.82 MHz for <sup>15</sup>N, D<sub>2</sub>O, 30 °C).

assignment	$\delta_N^a$	HMBC (N to H)
2-NHAc <sup>A</sup>	121.0	2-Ac <sup>A</sup> , 3 <sup>A</sup>
2-NHAc <sup>C</sup>	121.0	2-Ac <sup>C</sup> , 3 <sup>C</sup>
6 <sup>D</sup> -NH	65.2	5 <sup>D</sup> , 6 <sup>D</sup>
1''-NH	146.6	6 <sup>D</sup> , 2''
6''-NH	125.6	6'', b2
b7-NH	90.7	b7, b9-u, b9-d
b8-NH	81.9	b6, b8

<sup>a</sup><sup>1</sup>H, <sup>15</sup>N HMBC readouts