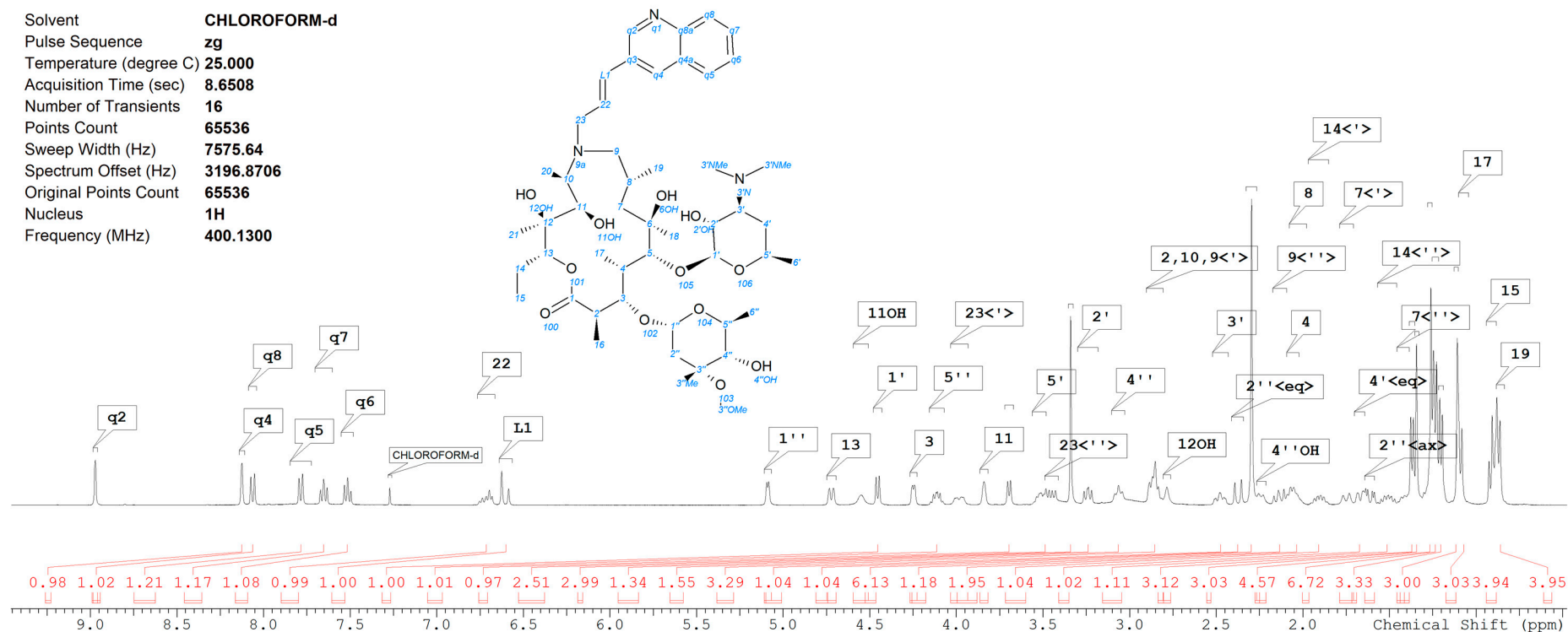


# New Bicyclic Azalide Macrolides Obtained by Tandem Palladium Catalysed Allylic Alkylation/Conjugated Addition Reaction

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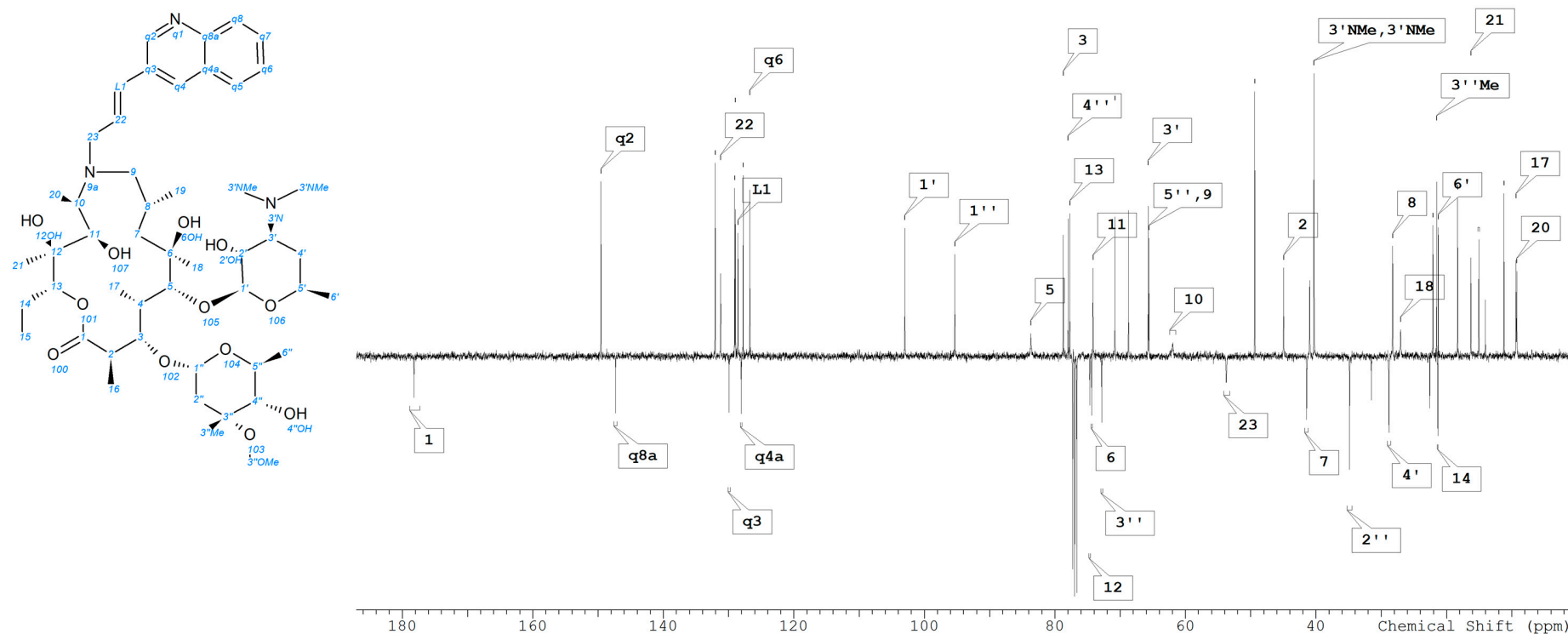
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Solvent **CHLOROFORM-d**  
Pulse Sequence **zg**  
Temperature (degree C) **25.000**  
Acquisition Time (sec) **8.6508**  
Number of Transients **16**  
Points Count **65536**  
Sweep Width (Hz) **7575.64**  
Spectrum Offset (Hz) **3196.8706**  
Original Points Count **65536**  
Nucleus **<sup>1</sup>H**  
Frequency (MHz) **400.1300**



Shift1 (ppm)	H's	Type	J (Hz)	Atom1	Multiplet1	Shift1 (ppm)	H's	Type	J (Hz)	Atom1	Multiplet1	Shift1 (ppm)	H's	Type	J (Hz)	Atom1	Multiplet1
0.872	3.000	br d	7.1	19	M01	2.140	1.000	dd	11.0, 10.5	9<">	M38	4.456	1.000	d	7.2	1'	M22
0.908	3.000	t	7.4	15	M45	2.243	1.000	br d	10.2	4"OH	M10	4.553	1.000	m	-	11OH	M23
1.092	3.000	d	7.3	17	M02	2.297	6.000	s	-	3'NMe, 3'NMe	M37	4.723	1.000	br dd	9.4, 1.6	13	M24
1.109	3.000	s	-	21	M44	2.375	1.000	d	15.0	2"<eq>	M36	5.090	1.000	br d	4.6	1"	M25
1.203	3.000	d	6.1	6'	M03	2.479	1.000	ddd	12.9, 9.8, 3.6	3'	M11	6.606	1.000	d	16.0	L1	M26
1.238	7.000	br d	6.9	4'<ax>, 20, 16	M42	2.786	1.000	br s	-	12OH	M12	6.718	1.000	ddd	16.0, 7.0, 6.0	22	M27
1.261	3.000	s	-	3"Me	M43	2.858	3.000	m	-	2, 10, 9<">	M35	7.516	1.000	t	7.2	q6	M28
1.344	3.000	s	-	18	M04	3.064	1.000	br t	8.6	4"	M13	7.654	1.000	t	7.6	q7	M29
1.370	3.000	d	6.1	6"	M40	3.242	1.000	br dd	9.8, 7.5	2'	M14	7.786	1.000	d	8.2	q5	M30
1.422	1.000	dd	15.7, 7.4	7<">	M41	3.341	3.000	s	-	3"OMe	M15	8.064	1.000	d	8.3	q8	M31
1.510	1.000	ddq	14.6, 9.3, 7.4	14<">	M05	3.457	1.000	br dd	14.4, 7.4	23<">	M16	8.126	1.000	s	-	q4	M32
1.613	1.000	dd	15.2, 4.8	2"<ax>	M06	3.516	1.000	dq	10.4, 6.0	5'	M34	8.972	1.000	s	-	q2	M33
1.668	1.000	br ddd	12.9, 2.0	4'<eq>	M39	3.697	1.000	d	6.7	5	M17						
1.750	1.000	br d	14.2	7<">	M07	3.842	1.000	d	1.2	11	M18						
1.907	1.000	dqd	14.4, 7.7, 1.4	14<">	M08	3.985	1.000	br dd	13.8, 4.9	23<">	M19						
2.029	1.000	m	-	8	M46	4.115	1.000	dq	9.6, 6.0	5"	M20						
2.062	1.000	br q	7.4	4	M09	4.249	1.000	br dd	4.0, 1.6	3	M21						

**Figure S1.** <sup>1</sup>H spectrum, structure, numbering and assignment of **3** in CDCl<sub>3</sub> at 25 °C



Shift1 (ppm)	C's	Type	Atom1	Multiplet1	Shift1 (ppm)	C's	Type	Atom1	Multiplet1	Shift1 (ppm)	C's	Type	Atom1	Multiplet1	Shift1 (ppm)	C's	Type	Atom1	Multiplet1
9.258	1.000	s	20	M01	40.989	1.000	s		M14	78.014	1.000	s	4"	M23	149.586	1.000	s	q2	M36
9.389	1.000	s	17	M02	41.441	1.000	s	7	M45	78.744	1.000	s	3	M24	178.240	1.000	s	1	M37
11.212	1.000	s	15	M03	44.985	1.000	s	2	M15	83.716	1.000	br s	5	M26					
15.076	1.000	s	16	M04	49.403	1.000	s	3"OMe	M16	95.353	1.000	s	1"	M27					
16.286	1.000	s	21	M06	53.778	1.000	br s	23	M44	103.024	1.000	s	1'	M28					
18.328	1.000	s	6"	M07	61.959	1.000	br s	10	M43	126.778	1.000	s	q6	M29					
21.317	1.000	s	6'	M08	65.619	2.000	s	5", 9	M17	127.799	1.000	s	q5	M30					
21.347	1.000	s	14	M48	65.736	1.000	s	3'	M18	128.076	1.000	s	q4a	M40					
21.536	1.000	s	3"Me	M09	68.755	1.000	s	5'	M19	128.601	1.000	s	L1	M31					
22.105	1.000	s	19	M10	70.840	1.000	s	2'	M20	128.980	1.000	s	q7	M32					
27.048	1.000	s	18	M11	72.838	1.000	s	3"	M42	129.112	1.000	s	q8	M33					
28.273	1.000	s	8	M12	74.179	1.000	s	11	M21	129.957	1.000	s	q3	M39					
28.857	1.000	s	4'	M47	74.383	1.000	s	6	M41	131.255	1.000	s	22	M34					
34.894	1.000	s	2"	M46	74.675	1.000	s	12	M25	132.087	1.000	s	q4	M35					
40.333	2.000	s	3'NMe, 3'NMe	M13	77.752	1.000	s	13	M22	147.354	1.000	s	q8a	M38					

Solvent **CHLOROFORM-d**

Pulse Sequence **deptqgpsp.2**

Temperature (degree C) **25.000**

Acquisition Time (sec) **0.3408**

Number of Transients **1000**

Points Count **16384**

Sweep Width (Hz) **24036.99**

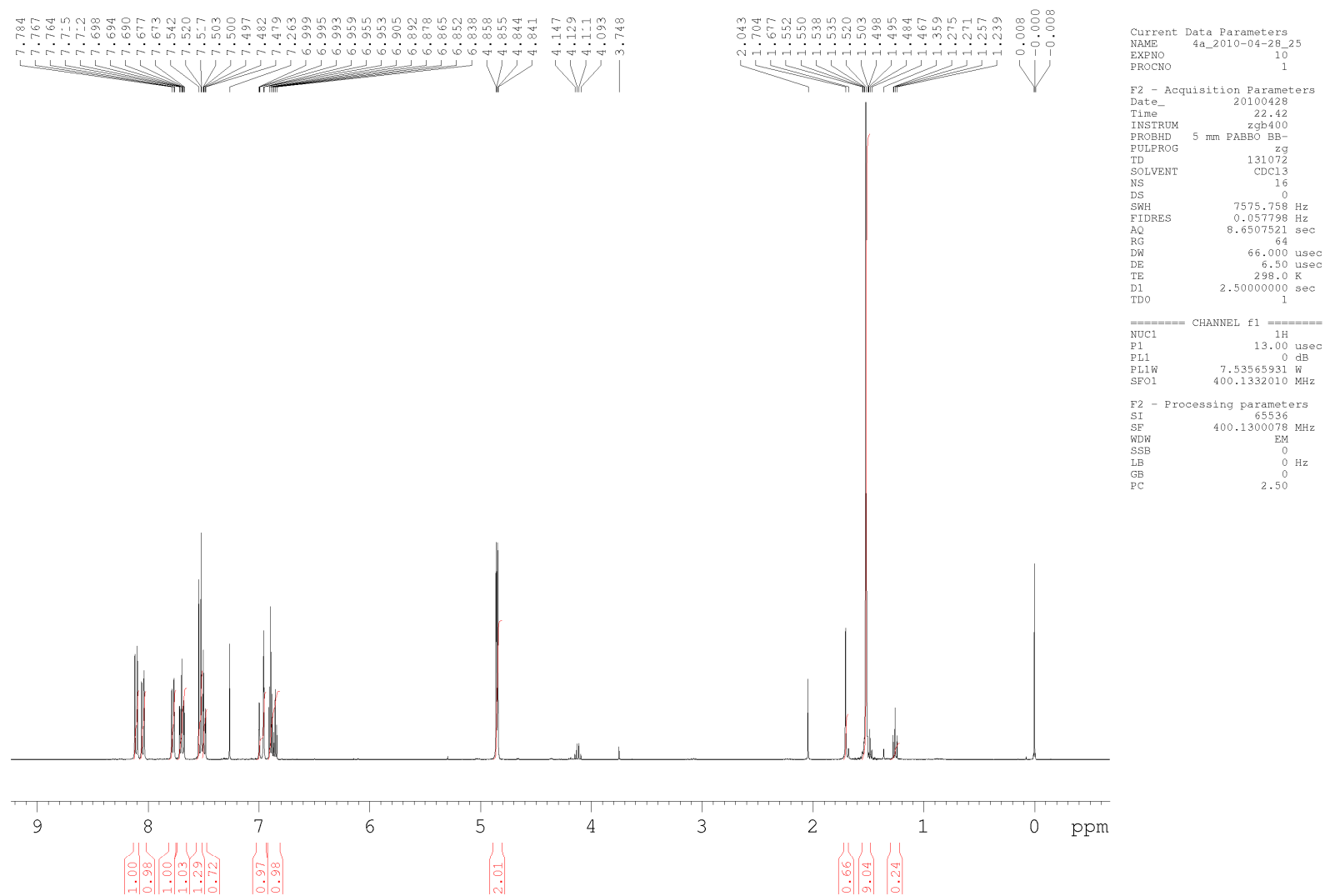
Spectrum Offset (Hz) **11063.1299**

Original Points Count **8192**

Nucleus **13C**

Frequency (MHz) **100.6128**

**Figure S2.**  $^{13}\text{C}$ -DEPTq spectrum, structure, numbering and assignment of **3** in  $\text{CDCl}_3$  at 25 °C



**Figure S3.**  $^1\text{H}$  spectrum of **8a** in  $\text{CDCl}_3$  at 25  $^\circ\text{C}$

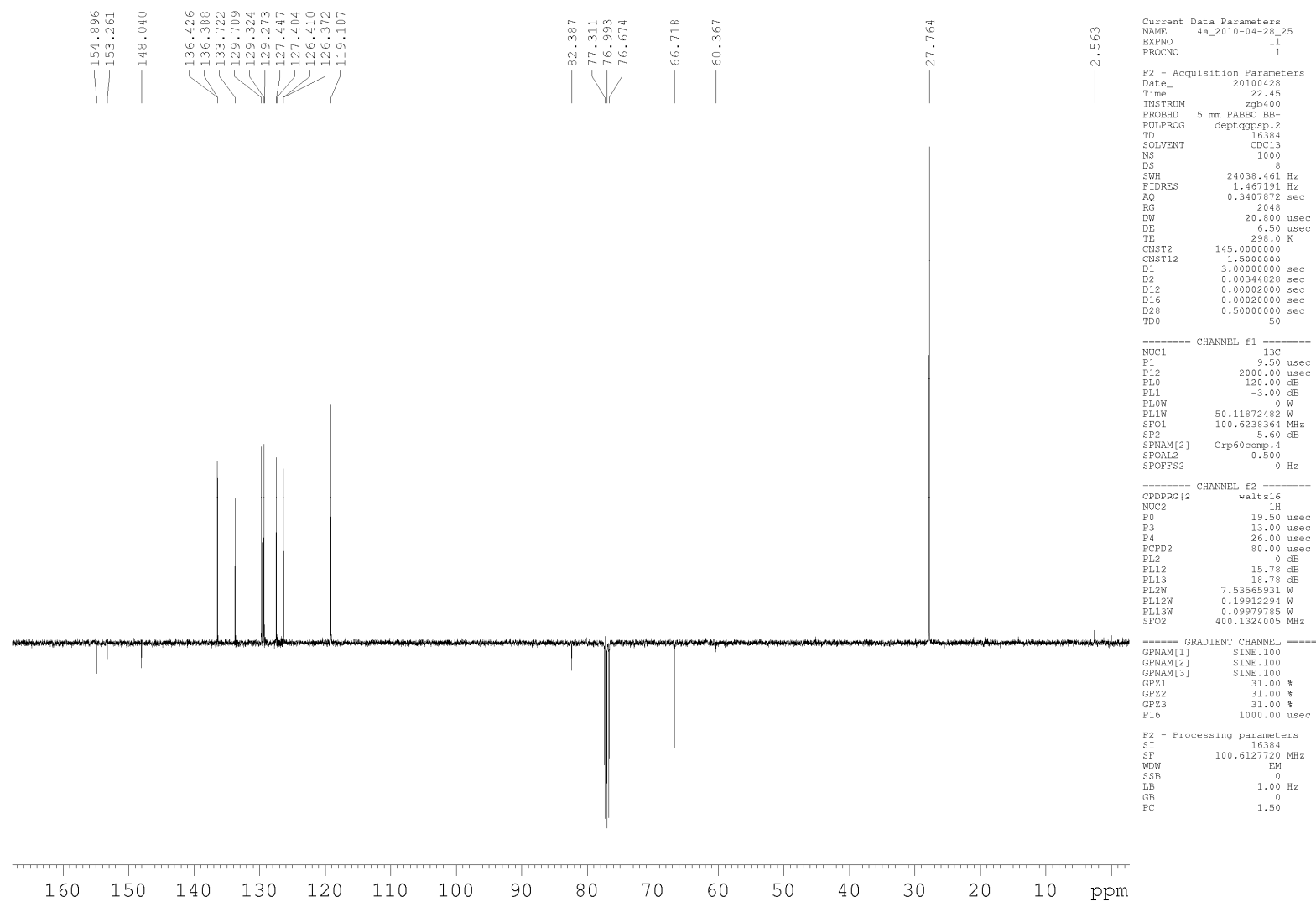
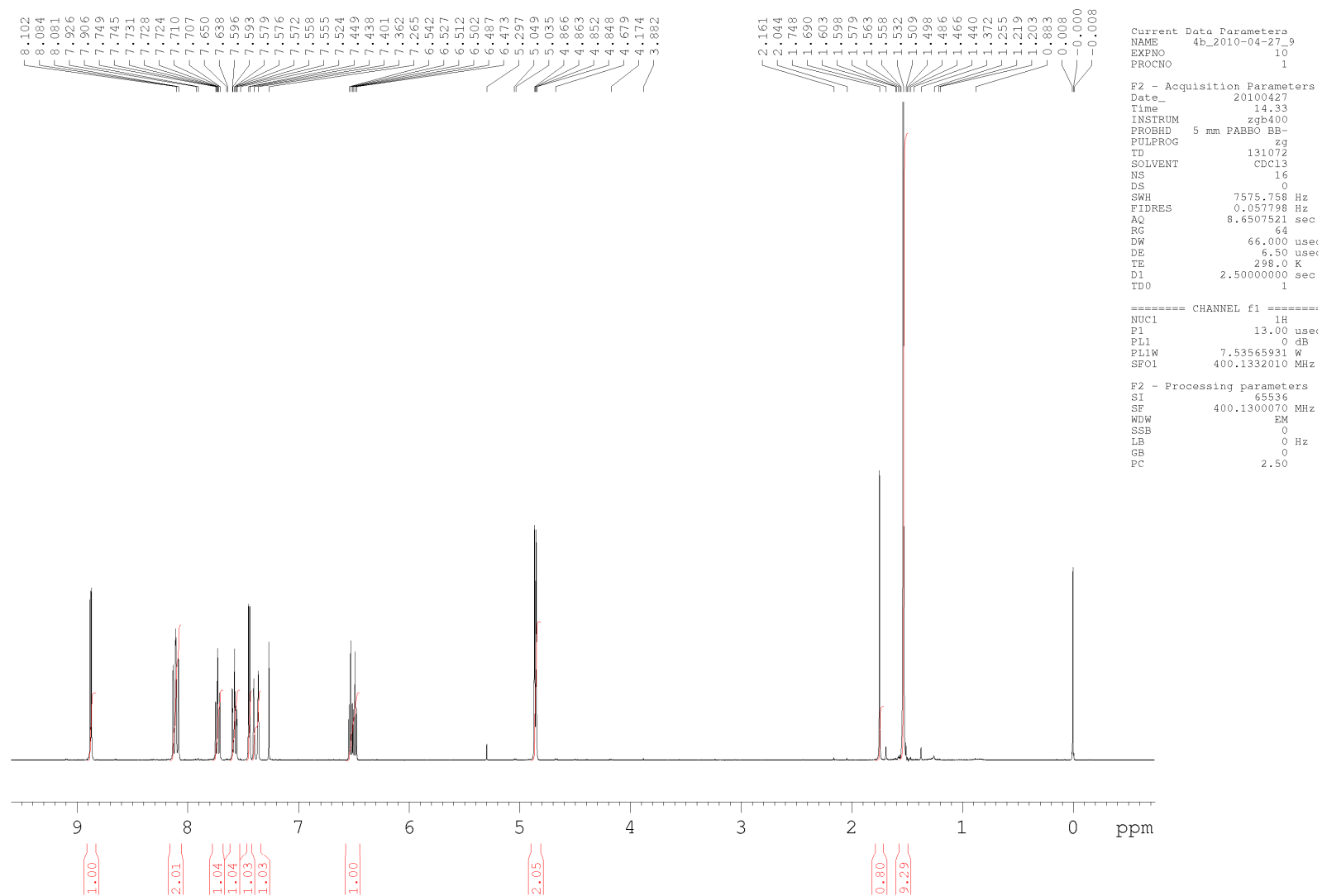
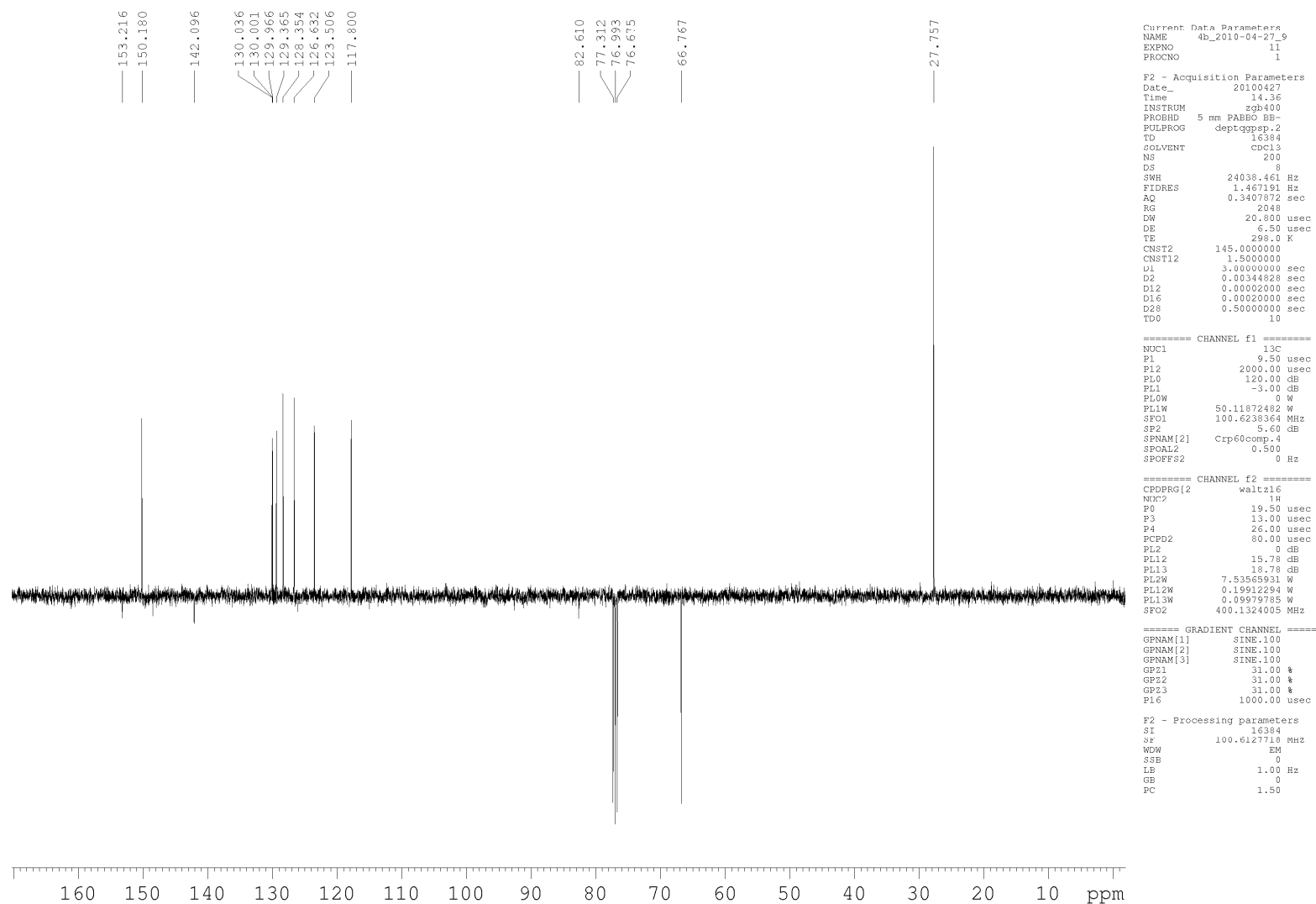


Figure S4.  $^{13}\text{C}$ -- DEPTq spectrum of **8a** in  $\text{CDCl}_3$  at 25  $^{\circ}\text{C}$

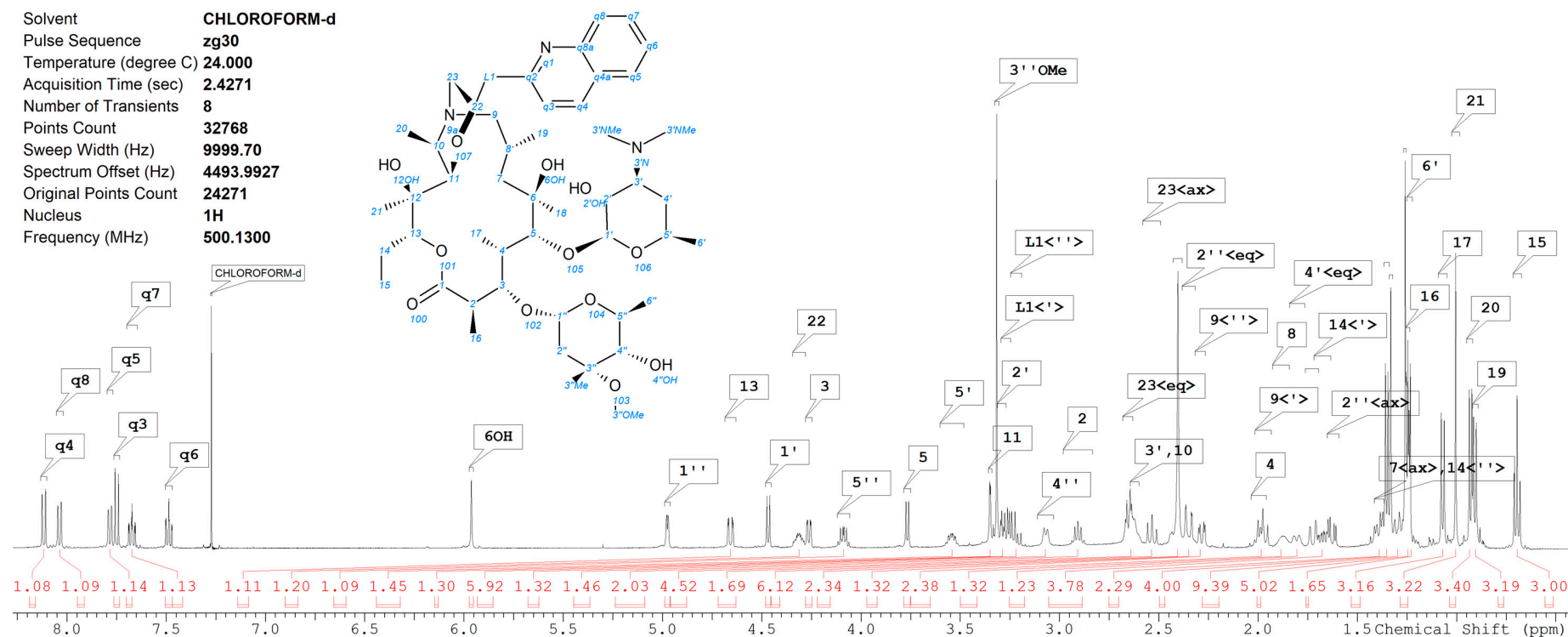


**Figure S5.**  $^1\text{H}$  spectrum of **8b** in  $\text{CDCl}_3$  at 25 °C



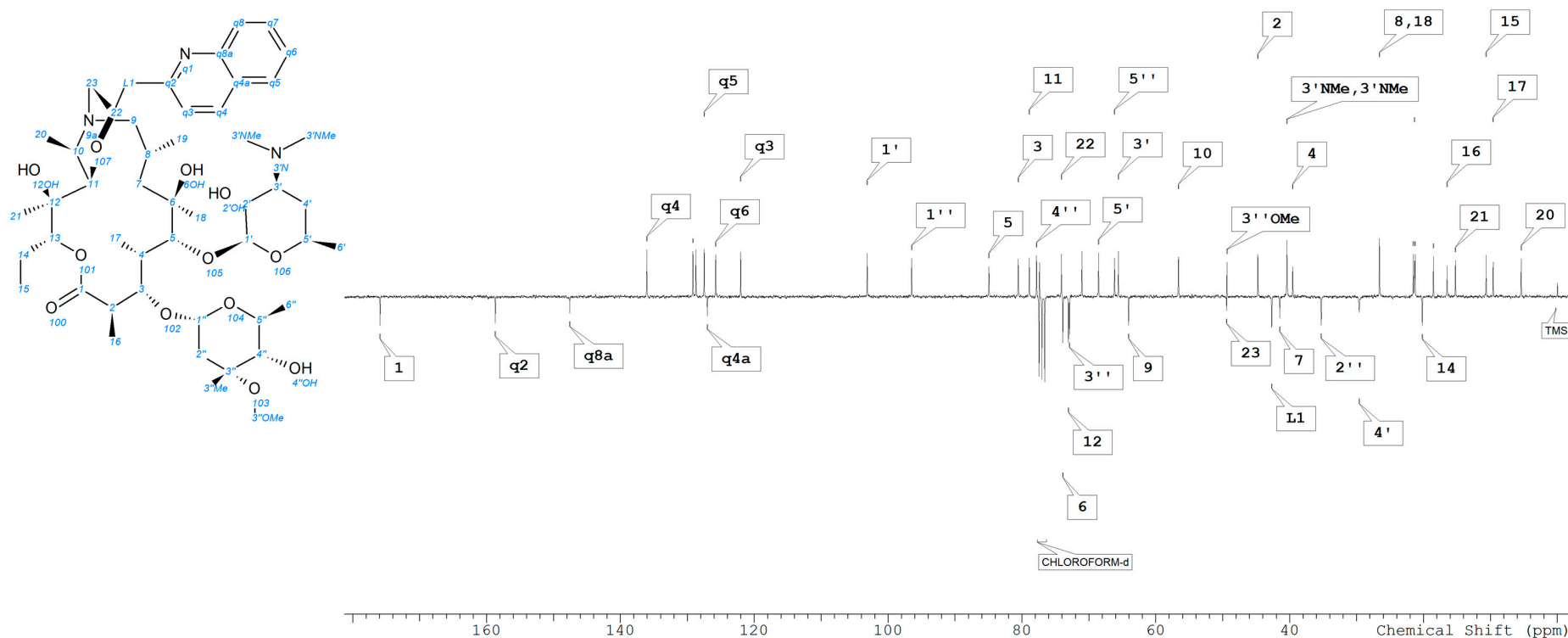
**Figure S6.**  $^{13}\text{C}$ -DEPTq spectrum of **8b** in  $\text{CDCl}_3$  at 25 °C

Solvent **CHLOROFORM-d**  
Pulse Sequence **zg30**  
Temperature (degree C) **24.000**  
Acquisition Time (sec) **2.4271**  
Number of Transients **8**  
Points Count **32768**  
Sweep Width (Hz) **9999.70**  
Spectrum Offset (Hz) **4493.9927**  
Original Points Count **24271**  
Nucleus **<sup>1</sup>H**  
Frequency (MHz) **500.1300**



Shift1 (ppm)	H's	Type	J (Hz)	Atom1	Multiplet1	Shift1 (ppm)	H's	Type	J (Hz)	Atom1	Multiplet1	Shift1 (ppm)	H's	Type	J (Hz)	Atom1	Multiplet1
0.696	3.000	t	7.5	15	M01	1.872	1.000	m	-	8	M47	3.543	1.000	dqd	10.2, 6.4, 2.1	5'	M15
0.909	3.000	d	7.0	19	M02	1.977	1.000	br t	12.1	9<'>	M08	3.768	1.000	d	7.0	5	M16
0.930	3.000	d	6.4	20	M38	1.988	1.000	quind	7.5, 1.8	4	M48	4.089	1.000	dq	8.8, 6.2	5"	M17
1.006	3.000	s	-	21	M03	2.284	1.000	dd	12.5, 3.4	9<'>	M09	4.264	1.000	dd	8.7, 1.8	3	M18
1.071	3.000	d	7.3	17	M04	2.350	1.000	br dd	14.6, 1.5	2"<eq>	M36	4.315	1.000	br dddd	10.7, 8.2, 4.9, 2.9	22	M29
1.240	3.000	br d	6.1	6'	M05	2.404	6.000	s	-	3'NMe, 3'NMe	M35	4.468	1.000	d	7.3	1'	M19
1.248	3.000	br d	7.3	16	M39	2.535	1.000	dd	11.6, 10.7	23<ax>	M10	4.659	1.000	dd	11.1, 2.0	13	M20
1.260	3.000	s	-	3"Me	M40	2.624	2.000	m	-	3', 10	M11	4.976	1.000	dd	4.6, 1.4	1"	M21
1.301	1.000	td	11.3, 10.2	4'<ax>	M43	2.654	1.000	dd	11.6, 3.1	23<eq>	M34	5.964	1.000	s	-	6OH	M22
1.333	3.000	s	-	18	M06	2.909	1.000	dq	8.7, 7.2	2	M12	7.487	1.000	ddd	8.1, 7.0, 1.1	q6	M23
1.353	3.000	d	6.1	6"	M41	3.070	1.000	br d	8.5	4"	M13	7.673	1.000	ddd	8.4, 6.9, 1.5	q7	M24
1.389	2.000	m	-	7<ax>, 14<'>	M42	3.219	1.000	dd	15.0, 8.2	L1<'>	M14	7.750	1.000	d	8.5	q3	M25
1.628	1.000	dd	15.0, 4.9	2"<ax>	M07	3.273	1.000	dd	14.0, 4.9	L1<'>	M31	7.784	1.000	dd	8.2, 0.9	q5	M28
1.674	1.000	dqd	14.4, 7.5, 1.8	14<'>	M45	3.295	1.000	dd	10.4, 7.6	2'	M33	8.039	1.000	d	8.2	q8	M26
1.726	1.000	d	14.6	7<eq>	M37	3.317	3.000	s	-	3"OMe	M32	8.117	1.000	d	8.5	q4	M27
1.807	1.000	ddd	12.5, 4.0, 2.1	4'<eq>	M44	3.351	1.000	d	1.8	11	M30						

**Figure S7.** <sup>1</sup>H spectrum, structure, numbering and assignment of **10a** in CDCl<sub>3</sub> at 25 °C



Shift1 (ppm)	C's	Type	Atom1	Multiplet1	Shift1 (ppm)	C's	Type	Atom1	Multiplet1	Shift1 (ppm)	C's	Type	Atom1	Multiplet1	Shift1 (ppm)	C's	Type	Atom1	Multiplet1
5.421	1.000	s	20	M02	41.490	1.000	s	7	M17	77.392	1.000	s	13	M48	158.728	1.000	s	q2	M46
9.607	1.000	s	17	M03	42.687	1.000	s	L1	M18	77.844	1.000	s	4"	M32	175.934	1.000	s	1	M47
10.662	1.000	s	15	M04	44.773	1.000	s	2	M19	78.923	1.000	s	11	M33					
15.253	1.000	s	21	M05	49.379	1.000	s	3"OMe	M20	80.564	1.000	s	3	M34					
16.498	1.000	s	16	M06	49.419	1.000	s	23	M21	84.949	1.000	s	5	M35					
18.551	1.000	s	6"	M07	56.634	1.000	s	10	M22	96.501	1.000	s	1"	M36					
20.201	1.000	s	14	M08	64.080	1.000	s	9	M23	103.146	1.000	s	1'	M37					
21.279	1.000	s	6'	M09	65.618	1.000	s	3'	M24	122.057	1.000	s	q3	M38					
21.350	1.000	s	19	M10	66.189	1.000	s	5"	M25	125.783	1.000	s	q6	M39					
21.548	1.000	s	3"Me	M11	68.583	1.000	s	5'	M26	127.036	1.000	s	q4a	M40					
26.591	2.000	s	8, 18	M12	71.065	1.000	s	2'	M27	127.496	1.000	s	q5	M41					
29.636	1.000	s	4'	M13	72.920	1.000	s	3"	M28	128.733	1.000	s	q8	M42					
35.313	1.000	s	2"	M14	73.079	1.000	s	12	M29	129.193	1.000	s	q7	M43					
39.563	1.000	s	4	M15	73.904	1.000	s	6	M30	136.075	1.000	s	q4	M44					
40.427	2.000	s	3'NMe, 3'NMe	M16	74.118	1.000	s	22	M31	147.596	1.000	s	q8a	M45					

Solvent **CHLOROFORM-d**

Pulse Sequence **jmod**

Temperature (degree C) **26.160**

Acquisition Time (sec) **1.6712**

Number of Transients **2425**

Points Count **32768**

Sweep Width (Hz) **19607.25**

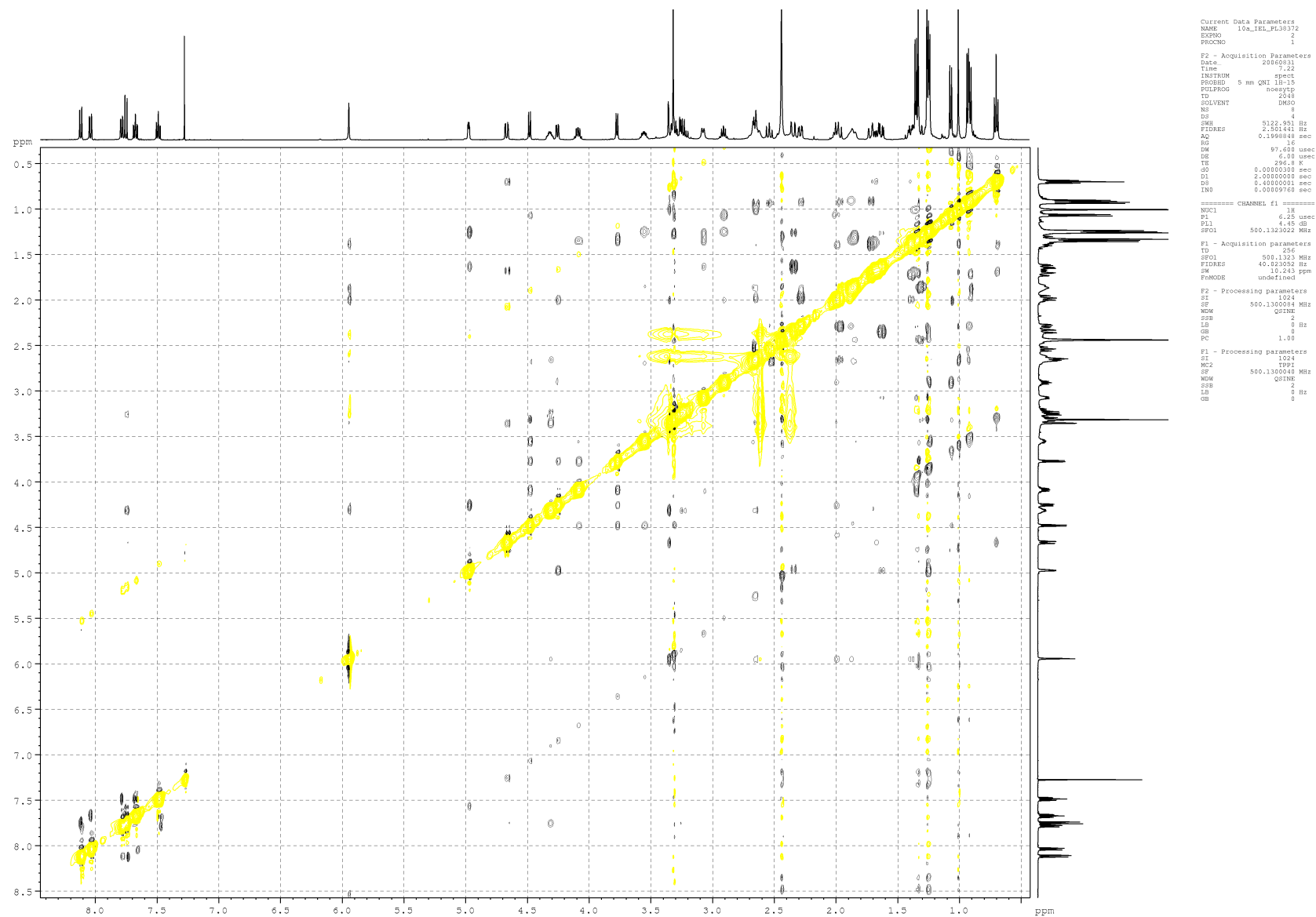
Spectrum Offset (Hz) **9027.3271**

Original Points Count **32768**

Nucleus  **$^{13}\text{C}$**

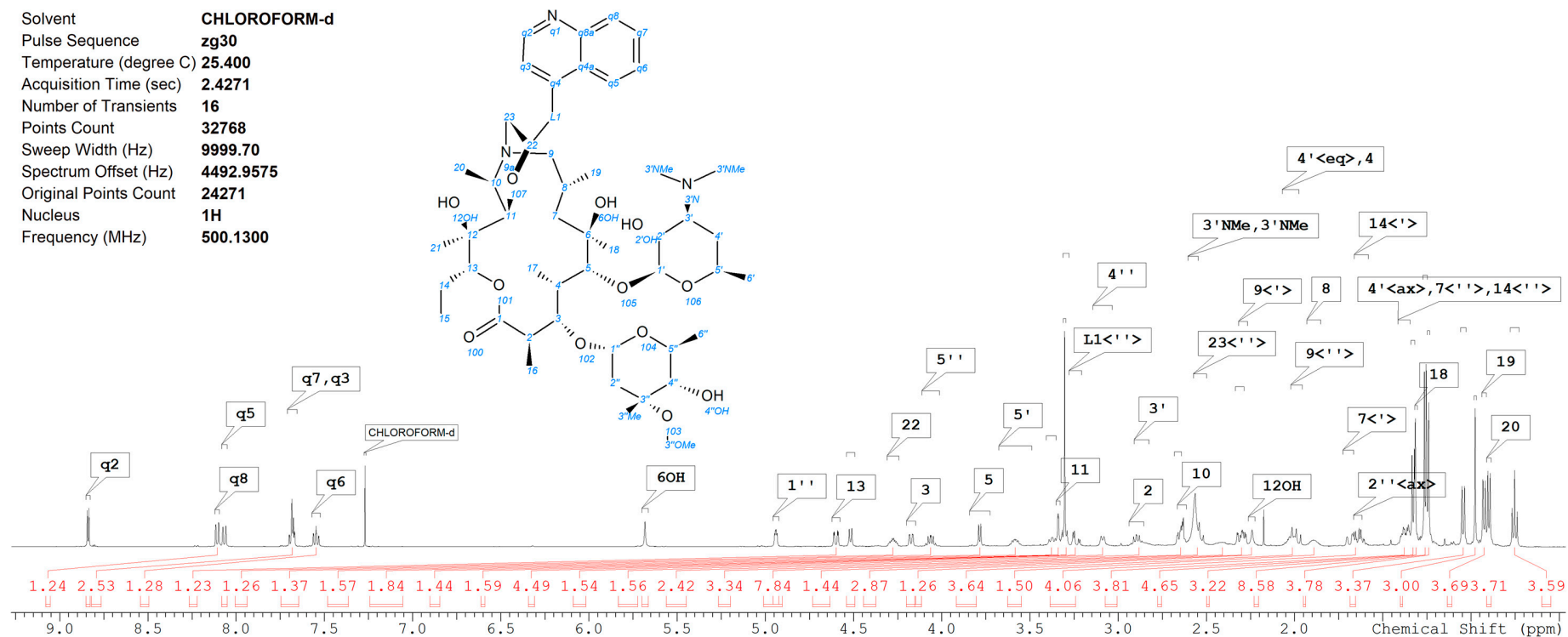
Frequency (MHz) **75.4678**

**Figure S8.**  $^{13}\text{C}$ -DEPTq spectrum, structure, numbering and assignment of **10a** in  $\text{CDCl}_3$  at 25 °C



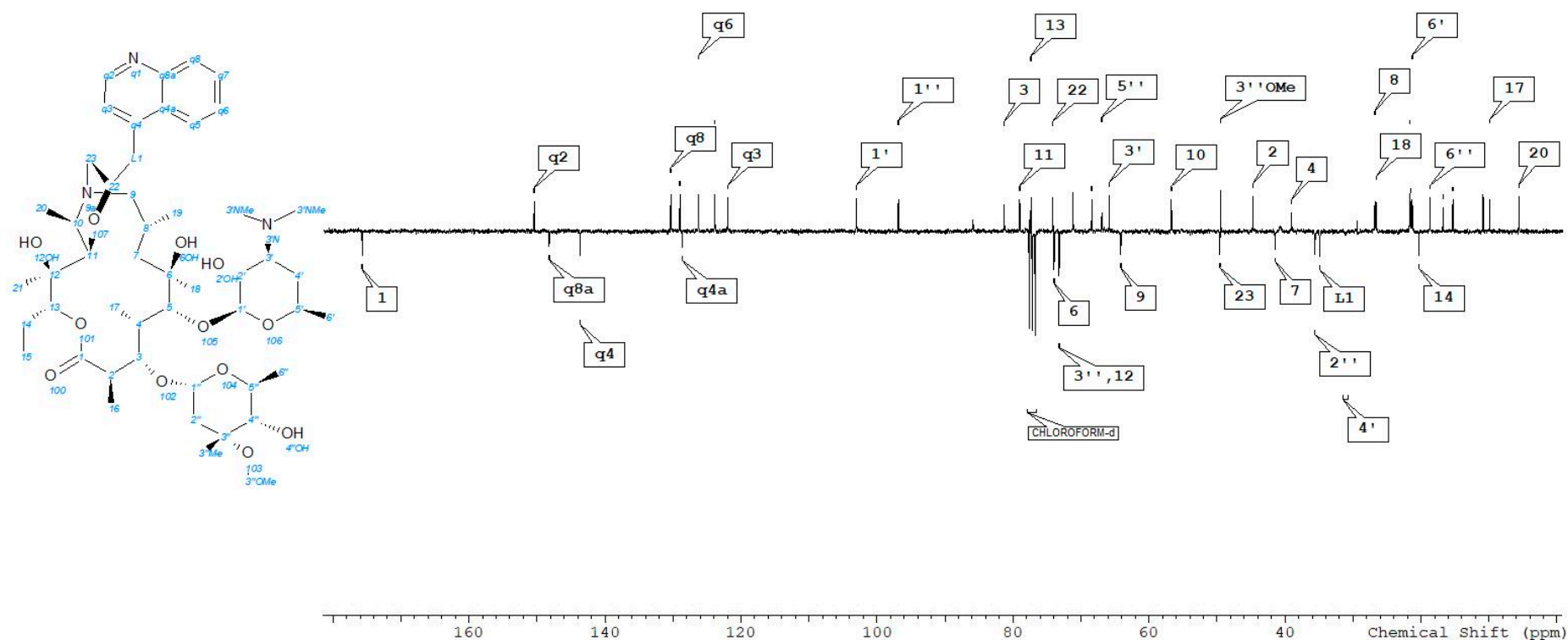
**Figure S9.** NOESY spectrum of **10a** in  $\text{CDCl}_3$  at 25 °C

Solvent	<b>CHLOROFORM-d</b>
Pulse Sequence	<b>zg30</b>
Temperature (degree C)	<b>25.400</b>
Acquisition Time (sec)	<b>2.4271</b>
Number of Transients	<b>16</b>
Points Count	<b>32768</b>
Sweep Width (Hz)	<b>9999.70</b>
Spectrum Offset (Hz)	<b>4492.9575</b>
Original Points Count	<b>24271</b>
Nucleus	<b>1H</b>
Frequency (MHz)	<b>500.1300</b>



Shift1 (ppm)	H's	Type	J (Hz)	Atom1	Multiplet1	Shift1 (ppm)	H's	Type	J (Hz)	Atom1	Multiplet1	Shift1 (ppm)	H's	Type	J (Hz)	Atom1	Multiplet1
0.753	3.000	t	7.3	15	M01	2.242	1.000	br s	-	12OH	M13	3.585	1.000	dqd	10.2, 6.1, 2.0	5'	M20
0.899	3.000	br d	6.4	20	M02	2.292	1.000	br dd	12.4, 3.5	9<'>	M42	3.786	1.000	d	6.1	5	M21
0.927	3.000	br d	7.0	19	M34	2.310	1.000	br dd	14.6, 2.4	2"<eq>	M12	4.064	1.000	dq	8.8, 6.4	5"	M22
0.978	3.000	s	-	21	M03	2.410	1.000	m	-		M14	4.175	1.000	dd	9.0, 1.7	3	M23
1.045	3.000	d	7.3	17	M04	2.542	1.000	t	11.6	23<'>	M40	4.278	1.000	ddt	10.8, 7.2, 3.7	22	M24
1.241	3.000	s	-	3"Me	M06	2.565	6.000	br s	-	3'NMe, 3"NMe	M41	4.518	1.000	d	7.3	1'	M25
1.260	6.000	d	5.2	16, 6'	M47	2.644	1.000	br s	-	10	M16	4.601	1.000	dd	11.1, 2.0	13	M26
1.316	3.000	s	-	18	M08	2.656	1.000	dd	8.5, 3.1	23<'>	M15	4.941	1.000	dd	4.3, 2.4	1"	M27
1.329	3.000	d	6.4	6"	M46	2.842	1.000	br s	-	3'	M17	5.683	1.000	s	-	6OH	M28
1.373	3.000	m	-	4'<ax>, 7<'>, 14<'>	M07	2.897	1.000	dq	9.2, 7.3	2	M39	7.548	1.000	ddd	8.2, 7.0, 1.2	q6	M29
1.630	1.000	dqd	14.4, 7.3, 1.8	14<'>	M45	3.089	1.000	br d	7.9	4"	M18	7.682	2.000	m	-	q7, q3	M30
1.645	1.000	dd	14.8, 4.7	2"<ax>	M44	3.237	1.000	dd	15.6, 4.0	L1<'>	M19	8.068	1.000	d	8.2	q5	M31
1.688	1.000	br dd	14.8, 1.1	7<'>	M09	3.304	3.000	s	-	3"OMe	M38	8.109	1.000	d	7.6	q8	M32
1.891	1.000	m	-	8	M10	3.305	1.000	d	15.0	L1<'>	M37	8.840	1.000	d	4.6	q2	M33
1.991	1.000	t	12.5	9<'>	M11	3.341	1.000	d	1.8	11	M36						
2.026	2.000	m	-	4'<eq>, 4	M43	3.375	1.000	br dd	9.9, 7.5	2'	M35						

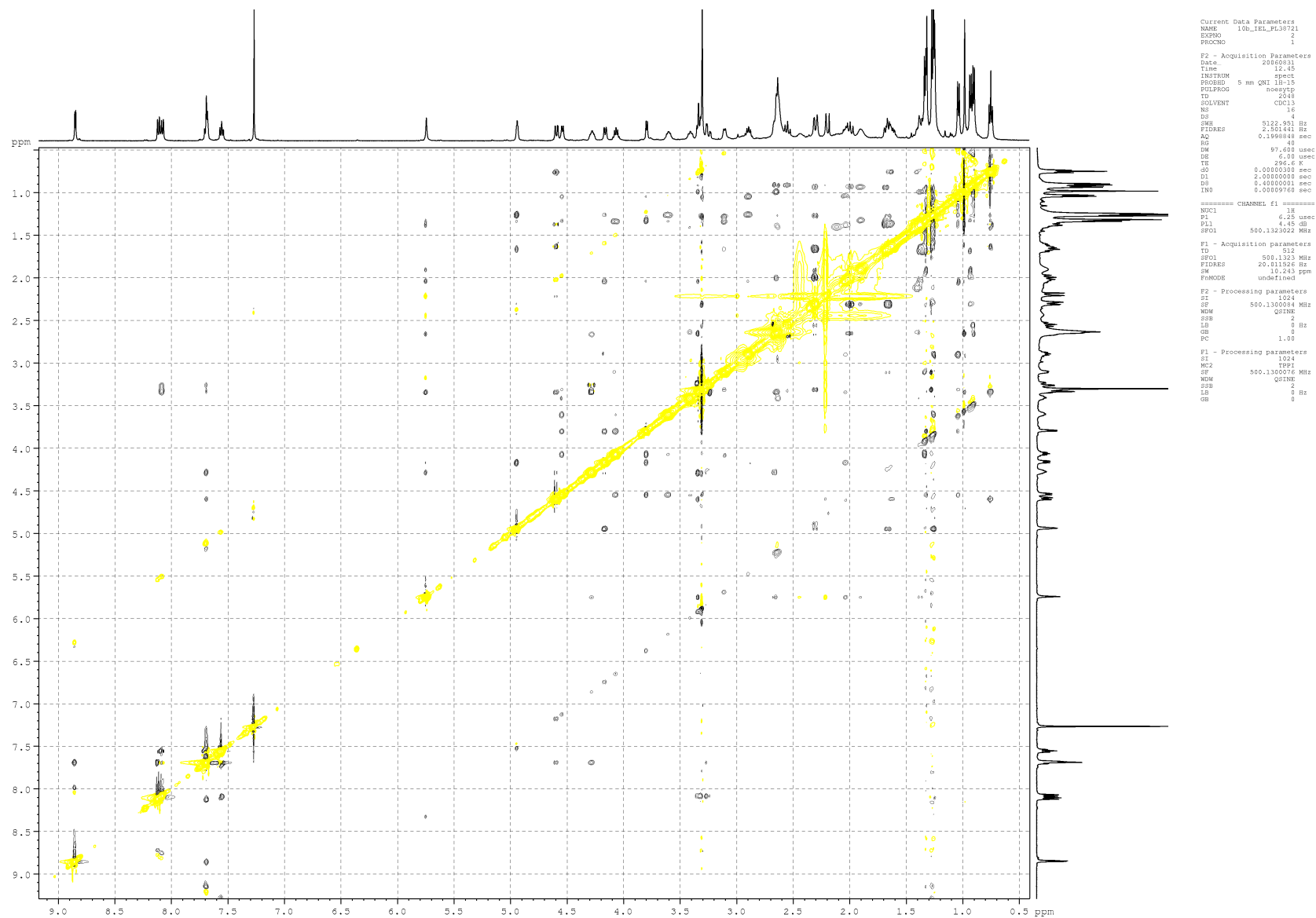
**Figure S10.**  $^1\text{H}$  spectrum, structure, numbering and assignment of **10b** in  $\text{CDCl}_3$  at 25 °C



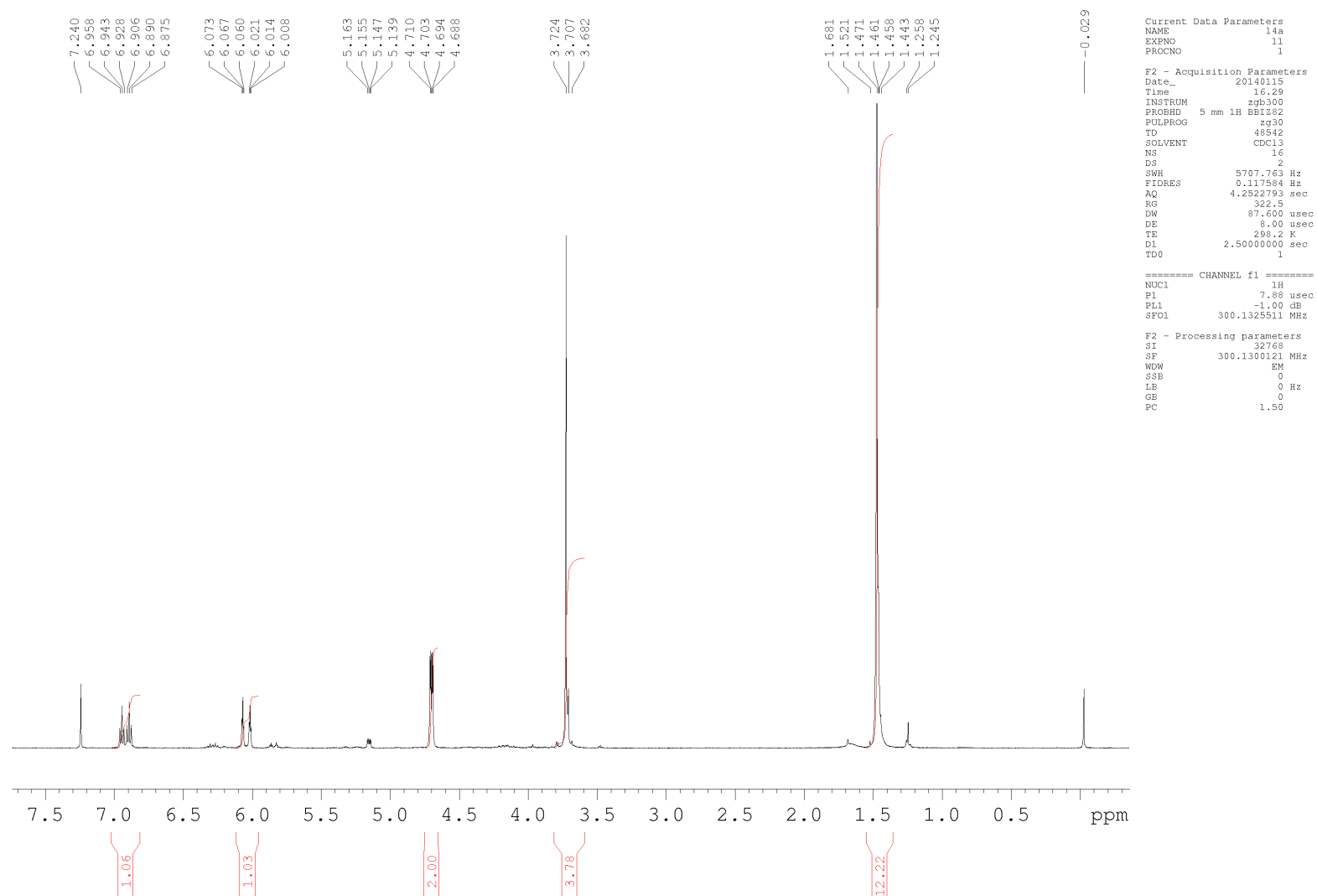
Shift1 (ppm)	C's	Type	Atom1	Multiplet1	Shift1 (ppm)	C's	Type	Atom1	Multiplet1	Shift1 (ppm)	C's	Type	Atom1	Multiplet1	Shift1 (ppm)	C's	Type	Atom1	Multiplet1
5.576	1.000	s	20	M01	39.013	1.000	s	4	M16	74.106	1.000	s	22	M32	150.383	1.000	s	q2	M46
9.905	1.000	s	17	M02	40.722	2.000	s	3'NMe, 3'NMe	M18	77.333	1.000	s	13	M48	175.677	1.000	s	1	M47
10.873	1.000	s	15	M03	41.439	1.000	s	7	M19	79.030	1.000	s	11	M33					
15.289	1.000	s	21	M04	44.714	1.000	s	2	M20	81.258	1.000	s	3	M34					
16.764	1.000	s	16	M05	49.439	1.000	s	3''OMe	M21	85.873	1.000	s		M35					
18.651	1.000	s	6''	M06	49.574	1.000	s	23	M22	96.823	1.000	s	1''	M36					
20.300	1.000	s	14	M07	56.694	1.000	s	10	M23	103.023	1.000	s	1'	M37					
21.252	1.000	s	6'	M08	64.132	1.000	s	9	M24	121.974	1.000	s	q3	M38					
21.482	1.000	s	19	M09	65.836	1.000	s	3'	M25	123.837	1.000	s	q5	M39					
21.648	1.000	s	3''Me	M10	66.899	1.000	s	5''	M26	126.240	1.000	s	q6	M40					
26.572	1.000	s	18	M11	68.413	1.000	s	5'	M27	128.642	1.000	s	q4a	M41					
26.762	1.000	s	8	M12	71.173	1.000	s	2'	M28	128.959	1.000	s	q7	M42					
31.145	1.000	m	4'	M13	73.209	1.000	s		M29	130.299	1.000	s	q8	M43					
34.890	1.000	s	L1	M14	73.209	2.000	s	3'', 12	M30	143.668	1.000	s	q4	M44					
35.579	1.000	s	2''	M15	73.924	1.000	s	6	M31	148.179	1.000	s	q8a	M45					

Solvent **CHLOROFORM-d**  
 Pulse Sequence **jmod**  
 Temperature (degree C) **25.160**  
 Acquisition Time (sec) **1.6712**  
 Number of Transients **2068**  
 Points Count **32768**  
 Sweep Width (Hz) **19607.25**  
 Spectrum Offset (Hz) **9037.8359**  
 Original Points Count **32768**  
 Nucleus **13C**  
 Frequency (MHz) **75.4677**

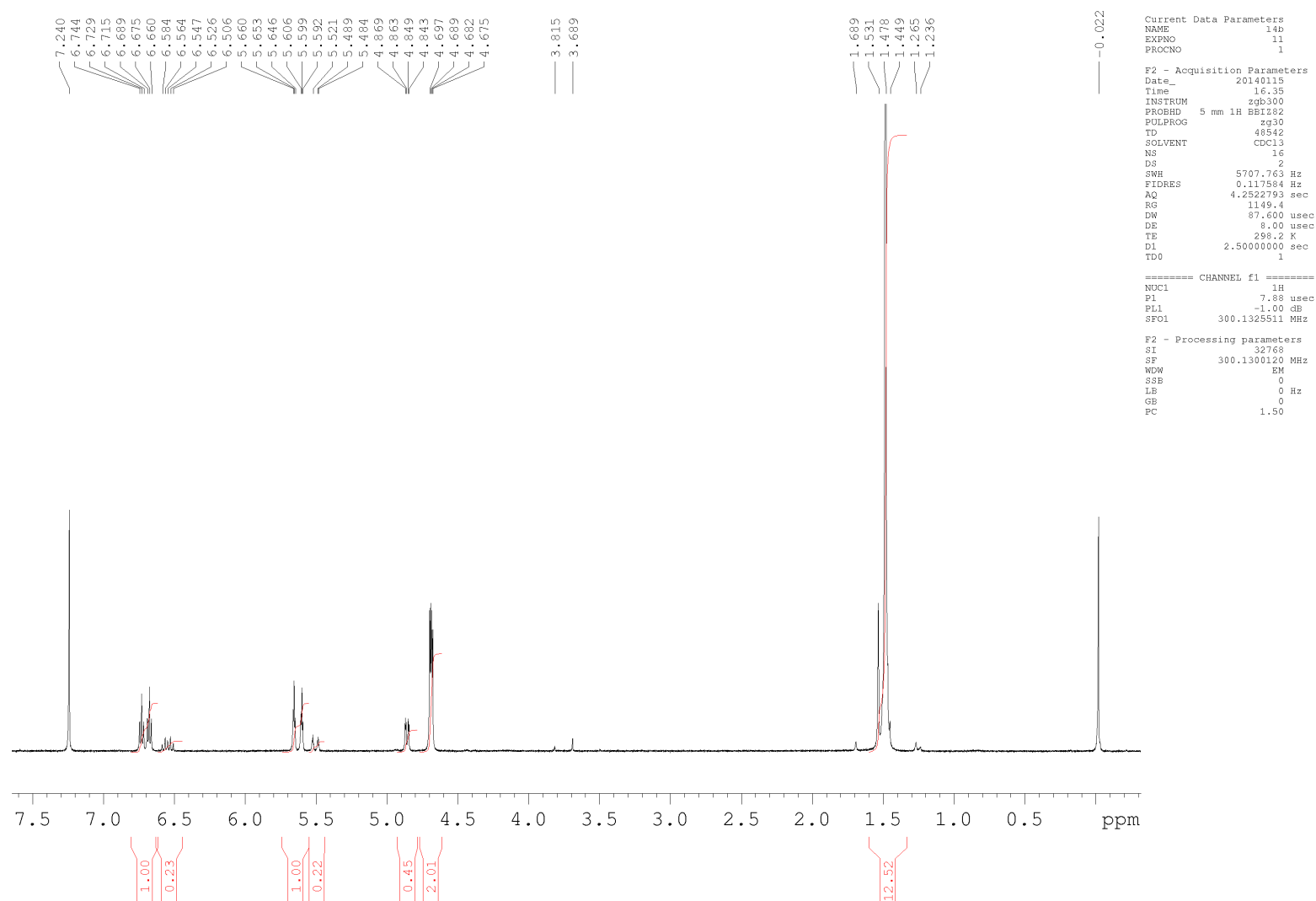
**Figure S11.** <sup>13</sup>C- DEPTq spectrum, structure, numbering and assignment of **10b** in CDCl<sub>3</sub> at 25 °C



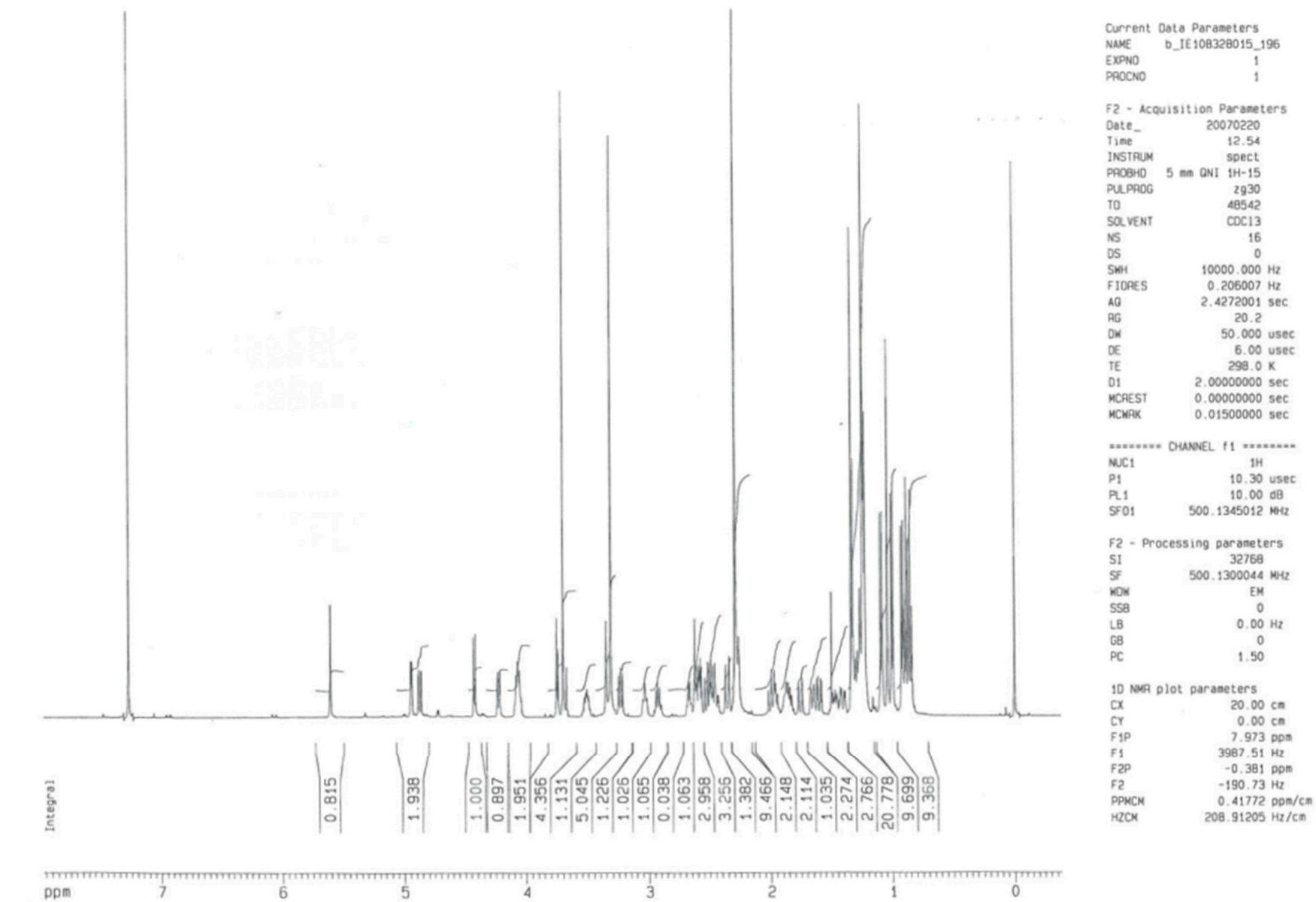
**Figure S12.** NOESY spectrum of **10b** in  $\text{CDCl}_3$  at 25 °C



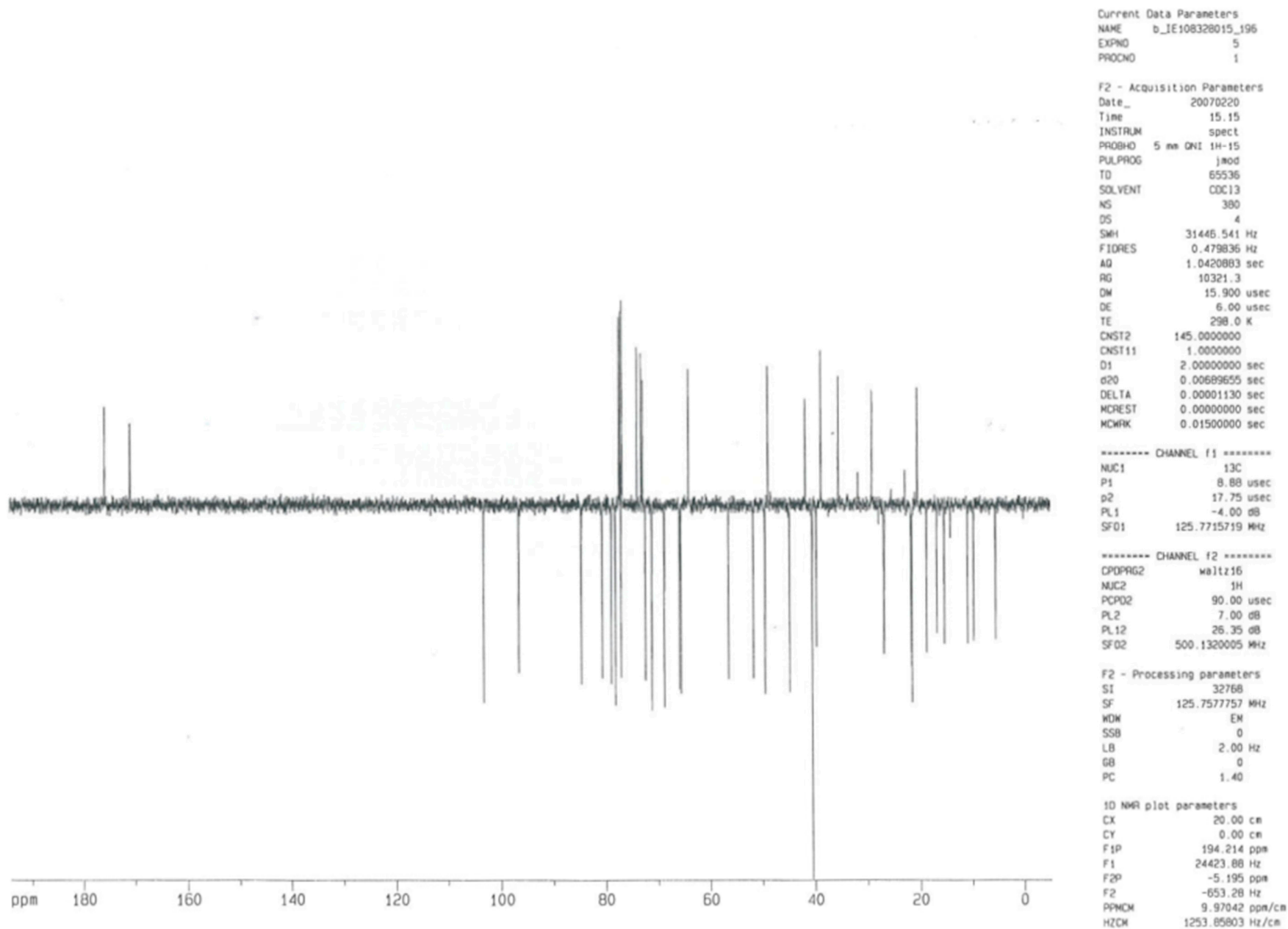
**Figure S13.**  $^1\text{H}$  spectrum of **14a** in  $\text{CDCl}_3$  at 25 °C



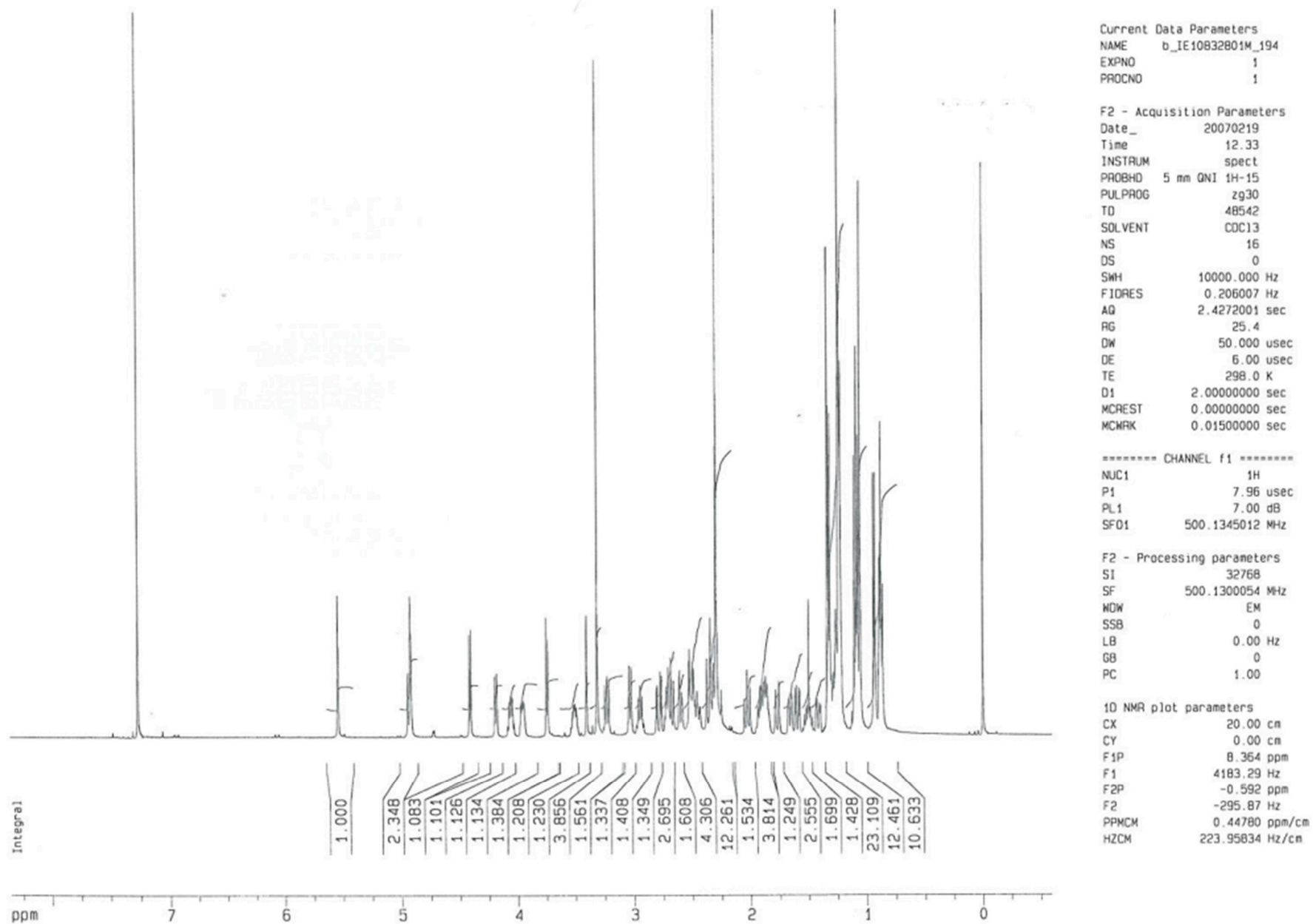
**Figure S14.**  $^1\text{H}$  spectrum of **14b** in  $\text{CDCl}_3$  at 25 °C



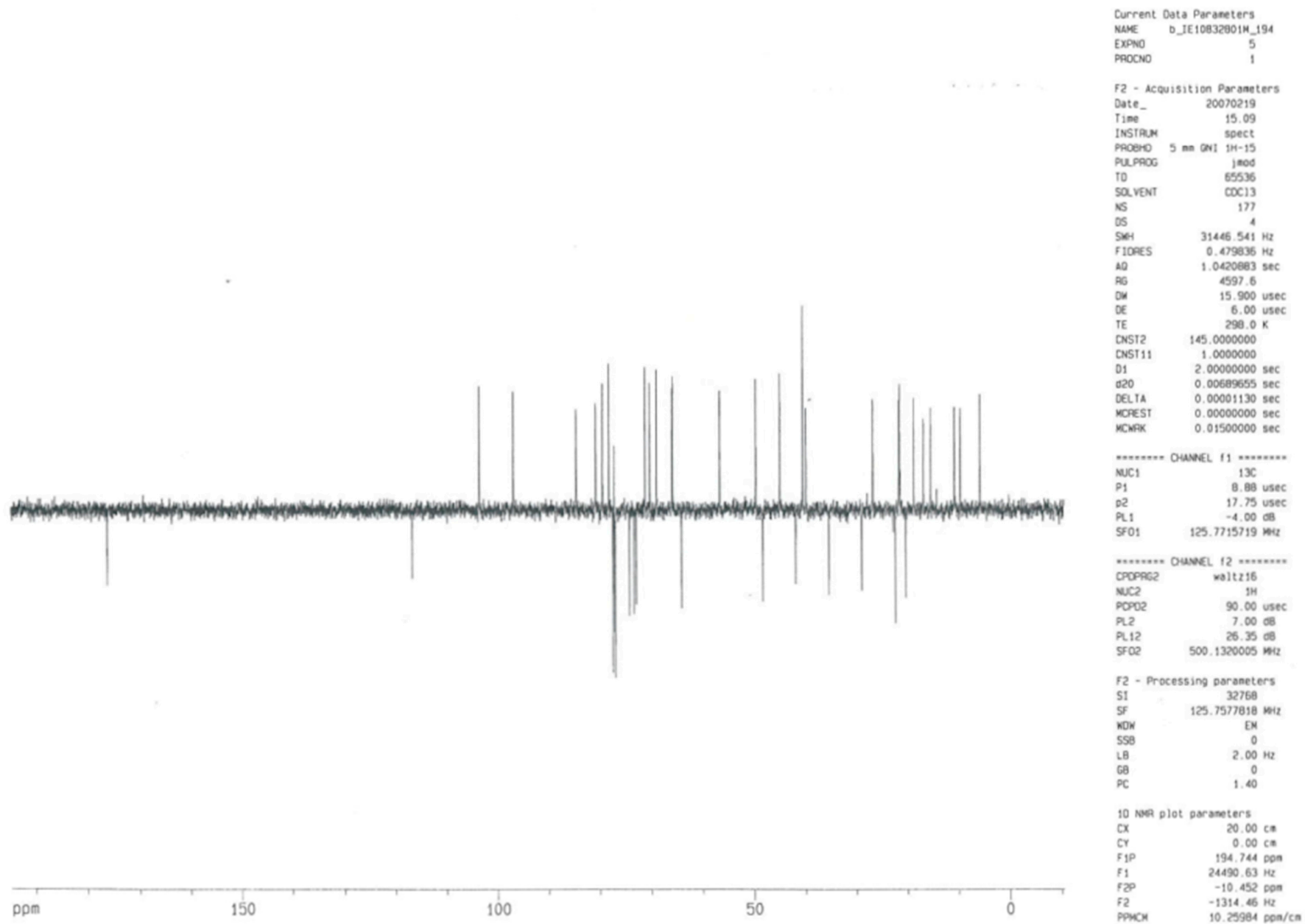
**Figure S15.**  $^1\text{H}$  spectrum of **15a** in  $\text{CDCl}_3$  at 25  $^\circ\text{C}$



**Figure S16.**  $^{13}\text{C}$ -APT spectrum of **15a** in  $\text{CDCl}_3$  at 25 °C



**Figure S17.**  $^1\text{H}$  spectrum of **15b** in  $\text{CDCl}_3$  at 25  $^\circ\text{C}$



**Figure S18.**  $^{13}\text{C}$ -APT spectrum of **15b** in  $\text{CDCl}_3$  at 25 °C

**Table S1.** Comparison of  $^3J_{H,H}$  coupling constants for azithromycin, **3**, **10a** and **10b** in  $CDCl_3$  at 25 °C;  
\*overlapped, the largest difference marked in red

Atom	azithromycin	3	10a	10b	Atom	azithromycin	3	10a	10b
<b>2, 3</b>	<b>4.0</b>	<b>4.1</b>	<b>8.7</b>	<b>9.0</b>	<b>2, 16</b>	7.5	6.9	7.2	7.3
<b>3, 4</b>	2.0	1.6	1.8	1.7	<b>4, 17</b>	7.5	7.3	7.3	7.3
<b>4, 5</b>	7.5	6.7	7.0	6.1	<b>8, 19</b>	6.5	7.1	7.0	7.0
<b>7a, 8</b>	< 1	< 1	< 1	1.1	<b>10, 20</b>	6.8	6.9	6.4	6.4
<b>7b, 8</b>	*	7.4	*	*	<b>14, 15</b>	7.5	7.4	7.5	7.3
<b>8, 9a</b>	< 1	*	< 1	3.5	<b>5', 6'</b>	6.1	6.0	6.4	6.1
<b>8, 9b</b>	11.0	11.0	12.1	12.5	<b>5'', 6''</b>	6.3	6.0	6.2	6.4
<b>10, 11</b>	1.2	1.2	1.8	1.8					
<b>13, 14a</b>	2.8	1.6	2.0	2.0	<b>11, 11 OH</b>	6.6	< 1	-	-
<b>13, 14b</b>	9.8	9.4	11.1	11.1	<b>2', 2' OH</b>	+	n/a	n/a	n/a
<b>22, 23eq</b>	-	4.9	2.9	3.7	<b>4'', 4'' OH</b>	10.5	10.2	n/a	n/a
<b>22, 23ax</b>	-	7.4	10.7	10.8					
<b>22, L1a</b>	-	16.0	4.9	7.2	<b>7a, 7b</b>	14.7	14.2	14.5	14.7
<b>22, L1b</b>	-	-	8.2	4.0	<b>9a, 9b</b>	11.0	10.5	12.1	12.5
<b>q2, q3</b>	-	-		4.6	<b>14a, 14b</b>	14.4	14.5	14.4	14.4
<b>q3, q4</b>	-	-	8.5	-	<b>4'a, 4'b</b>	12.6	13.0	12.5	*
<b>q5, q6</b>	-	8.2	8.2	7.0	<b>2''a, 2''b</b>	15.2	15.0	14.7	14.7
<b>q6, q7</b>	-	7.6	6.9	8.2	<b>23a, 23b</b>	-	14.5	11.6	8.5
<b>q7, q8</b>	-	8.3	8.2	7.6	<b>L1a, L1b</b>	-	-	15.0	15.6
<b>1', 2'</b>	7.3	7.2	7.3	7.3					
<b>2', 3'</b>	10.3	9.8	10.4	9.9					
<b>3', 4'a</b>	3.8	3.6	4.0	*					
<b>3', 4'b</b>	12.2	13.0	12.0	*					
<b>4'a, 5'</b>	1.9	< 1	2.1	2.0					
<b>4'b, 5'</b>	11.0	10.4	10.2	10.2					
<b>1'', 2''a</b>	< 1	< 1	1.4	2.4					
<b>1'', 2''b</b>	5.0	4.6	4.6	4.3					
<b>4'', 5''</b>	9.6	9.6	8.8	8.8					