

## Supplementary information

### Curtachalasans, immunosuppressive agents from endophytic fungus

#### *Xylaria cf. curta*

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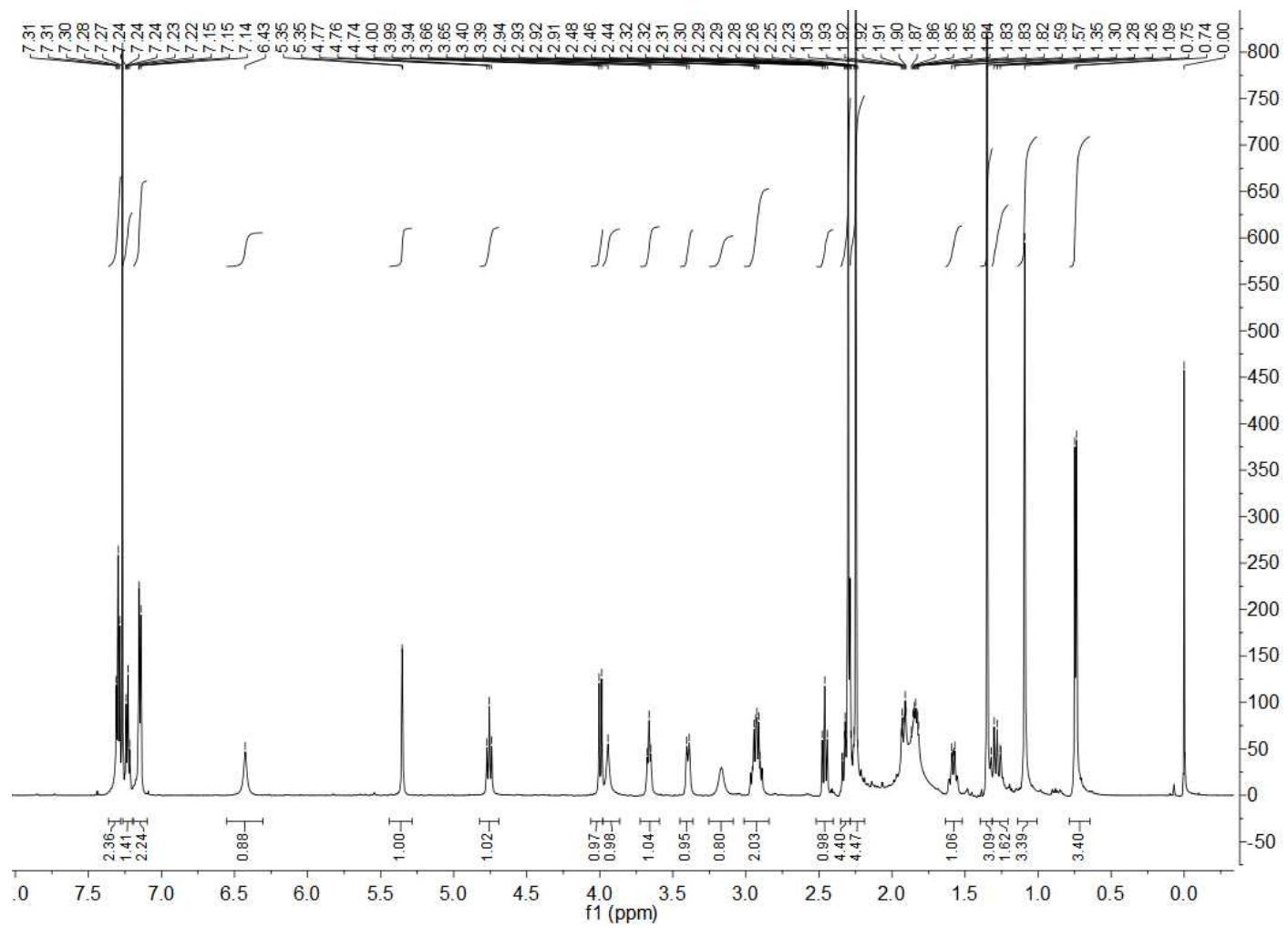
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**Table S1.**  $^1\text{H}$  NMR data of **1–11** ( $\text{CDCl}_3$ , 600 MHz,  $J$  in Hz).

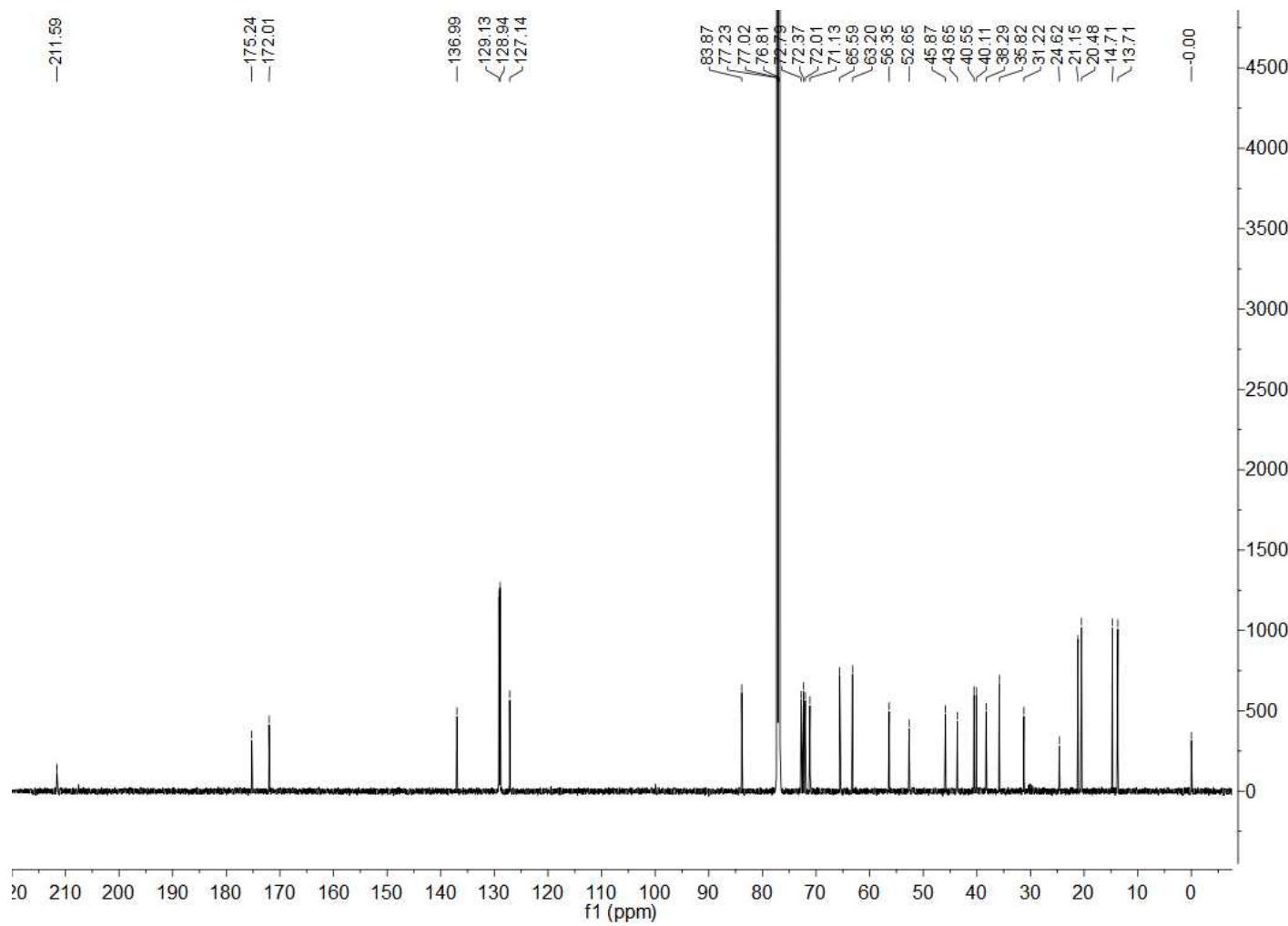
No	1	2	3	4	5
3	3.66 (dd, 7.5, 7.5)	3.38 m	3.32 (br dd, 8.2, 6.9)	4.97 m	3.33 overlapped
4	2.29 overlapped	2.10 br s	2.44 br s	2.28 overlapped	2.34 br s
5		2.50 m			
7	4.00 (d, 10.1)	4.46 br s	3.92 br s	4.20 (d, 3.0)	4.24 (d, 9.6)
8	2.46 (dd, 10.1, 10.1)				1.88 (dd, 9.6, 9.6)
10	2.93 m	2.62 (dd, 13.8, 9.0); 2.91 (dd, 13.8, 3.8)	3.00 (dd, 13.2, 8.2); 3.06 (dd, 13.2, 6.9)	3.08 (dd, 12.9, 4.7); 2.49 (dd, 12.9, 10.5)	2.94 (dd, 13.6, 6.7); 2.86 (dd, 13.6, 7.9)
11	1.09 s	1.04 (d, 6.3)	1.26 s	0.80 s	1.20 s
12	1.34 s	5.07 br s; 5.48 br s	1.64 s	1.47 s	1.61 s
13	4.76 (dd, 10.1, 10.1)	5.96 br s	5.86 br s	2.09 (dd, 15.4, 3.6); 1.59 (dd, 15.4, 12.6)	4.70 (dd, 9.6, 9.6)
14	1.58 (dddd, 12.3, 10.1, 10.1, 3.4)	2.07 overlapped		1.81 (dddd, 12.0, 12.0, 12.0, 3.9, 3.9)	1.61 overlapped
15	1.92 m; 1.29 (ddd, 12.3, 12.3, 12.3)	1.59 (ddd, 12.9, 4.0, 4.0); 1.35 (ddd, 12.9, 12.9, 12.9)	2.0 (br dd, 13.4, 4.0); 2.29 br dd, 13.4, 13.4	1.30 m; 1.49 overlapped	1.93 (br dd, 11.0, 4.0); 1.27 (ddd, 11.0, 11.0, 11.0)
16	1.84 m	1.93 m	1.89 m	1.87 m	1.85 m
19	3.40 (d, 10.1)	3.46 (d, 10.0)	3.64 (dd, 9.6, 8.6)	3.47 (dd, 10.4, 10.4)	3.41 (dd, 10.5, 8.1)
20	2.31 (ddd, 10.1, 10.1, 2.0)	2.74 (dd, 10.0, 10.0)	2.84 (dd, 9.6, 3.8)	2.29 overlapped	2.36 overlapped
21	5.35 (d, 2.0)	5.30 br s	5.69 (d, 3.8)	5.56 (d, 1.8)	5.50 br s
22	2.25 s	2.26 s	2.25 s	2.27 s	2.24 s
23	0.74 (d, 6.7)	0.75 (d, 6.6)	0.71 (d, 6.6)	0.74 (d, 6.6)	0.72 (d, 6.5)
25/29	7.15 (d, 7.6)	7.13 (d, 7.5)	7.20 (d, 7.5)	7.17 (d, 7.4)	7.13 (d, 7.0)
26/28	7.30 (dd, 7.6, 7.6)	7.27 (dd, 7.5, 7.5)	7.29 (dd, 7.5, 7.5)	7.31 (dd, 7.4, 7.4)	7.26 (dd, 7.0, 7.0)
27	7.23 (t, 7.6)	7.20 (t, 7.5)	7.22 overlapped	7.22 (dd, 7.4)	7.18 (t, 7.0)
OAc	2.30 s	2.11 s	2.20 s	2.19 s	2.24 s

**Table S1.** Continued.

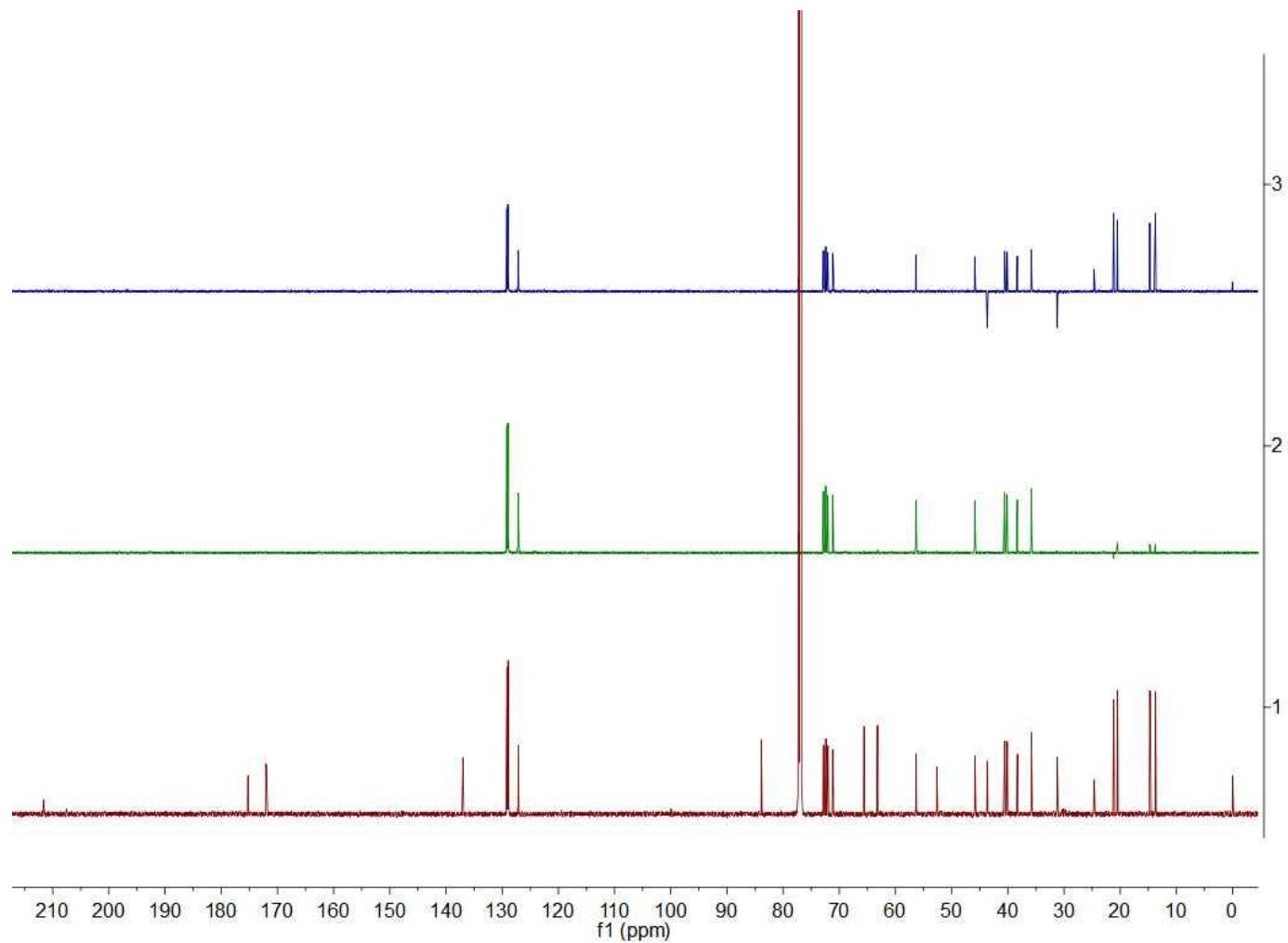
No	6	7	8	9	10	11
3	3.40 (dd, 7.2, 7.2)	3.34 m	3.49 m	3.55 (dd, 7.3, 7.3)	3.64 overlapped	3.33 m
4	2.31 br s	2.78 br s	2.94 br s	3.68 br s	2.87 br s	3.00 br s
7	4.36 br s	5.82 br s	5.97 br s			3.78 overlapped
8	2.11 overlapped	2.59 overlapped				1.74 (ddd, 13.5, 9.8, 4.4)
10	2.97 (dd, 13.6, 6.7); 2.82 (dd, 13.6, 7.6)	2.95 (dd, 13.3, 7.0); 2.82 (dd, 13.3, 8.3)	2.92, overlapped; 2.84 overlapped	3.04 (dd, 13.3, 6.4); 2.91 (dd, 13.3, 8.4)	3.16 (dd, 13.2, 5.9); 2.89 (dd, 13.2, 9.3)	2.80 m
11	1.17 s	4.82 m; 4.26 m	4.83 br s; 4.22 br s	1.44 s	1.43 s	1.38 s
12	1.72 s	1.82 s	1.75 s	1.68 s	1.73 s	1.62 s
13	4.99 (dd, 11.0, 11.0)	4.81 (dd, 11.0, 11.0)	5.60 s	6.75 s	6.84 (d, 2.0)	3.05 (dd, 13.5, 13.5); 2.33 (dd, 13.5, 4.4)
14	1.81 (dddd, 11.0, 11.0, 11.0, 3.3)	1.80 (dddd, 11.0, 11.0, 11.0, 3.0)	2.06 m	2.38 m	2.23 overlapped	
15	2.10 overlapped; 1.40 (ddd, 11.0, 11.0, 11.0)	2.09 (ddd, 11.0, 3.0, 3.0); 1.39 (ddd, 11.0, 11.0, 11.0)	1.47 (ddd, 12.0, 12.0, 12.0); 1.53 m	1.66 m; 1.44 m	1.68 (ddd, 12.9, 3.7, 3.7); 1.52 (ddd, 12.9, 12.9, 12.9)	5.20 br s
16	1.87 m	1.87 m	1.85 m	1.95 m	1.95 m	3.89 m
19	3.43 (dd, 11.0, 8.0)	3.46 (dd, 10.4, 8.0)	3.40 (dd, 10.7)	3.92 (d, 10.0)	3.48 (dd, 10.5, 6.7)	5.93 (d, 11.6)
20	2.69 (ddd, 11.0, 11.0, 2.0)	2.62 overlapped	2.79 (br dd, 10.7, 10.7)	2.40 m	2.75 (br dd, 12.9, 10.5)	3.44 (br d, 11.6)
21	5.46 (d, 2.0)	5.52 (d, 1.9)	5.36 br s	4.29 br s	5.59 br s	3.82 (br d, 6.9)
22	2.27 s	2.26 s	2.20 s	2.29 s	2.28 s	1.28 s
23	0.79 (d, 6.7)	0.78 (d, 6.6)	0.71 (d, 6.6)	0.69 (d, 6.5)	0.76 (d, 6.6)	1.22 (d, 6.7)
25/29	7.17 (d, 7.3)	7.16 (d, 7.2)	7.10 (d, 7.2)	7.28 (d, 7.2)	7.23 (d, 7.3)	7.14 (d, 7.5)
26/28	7.28 (dd, 7.3, 7.3)	7.31 (dd, 7.2)	7.24 (dd, 7.2)	7.33 (dd, 7.2, 7.2)	7.34 (dd, 7.3, 7.3)	7.33 (dd, 7.5, 7.5)
27	7.20 (t, 6.8)	7.23 (t, 7.2)	7.16 (t, 7.2)	7.22 (t, 7.2)	7.25 (t, 7.3)	7.26 (t, 7.5)
OAc	2.22 s	2.24 s	2.10 s		2.23 s	2.26, s



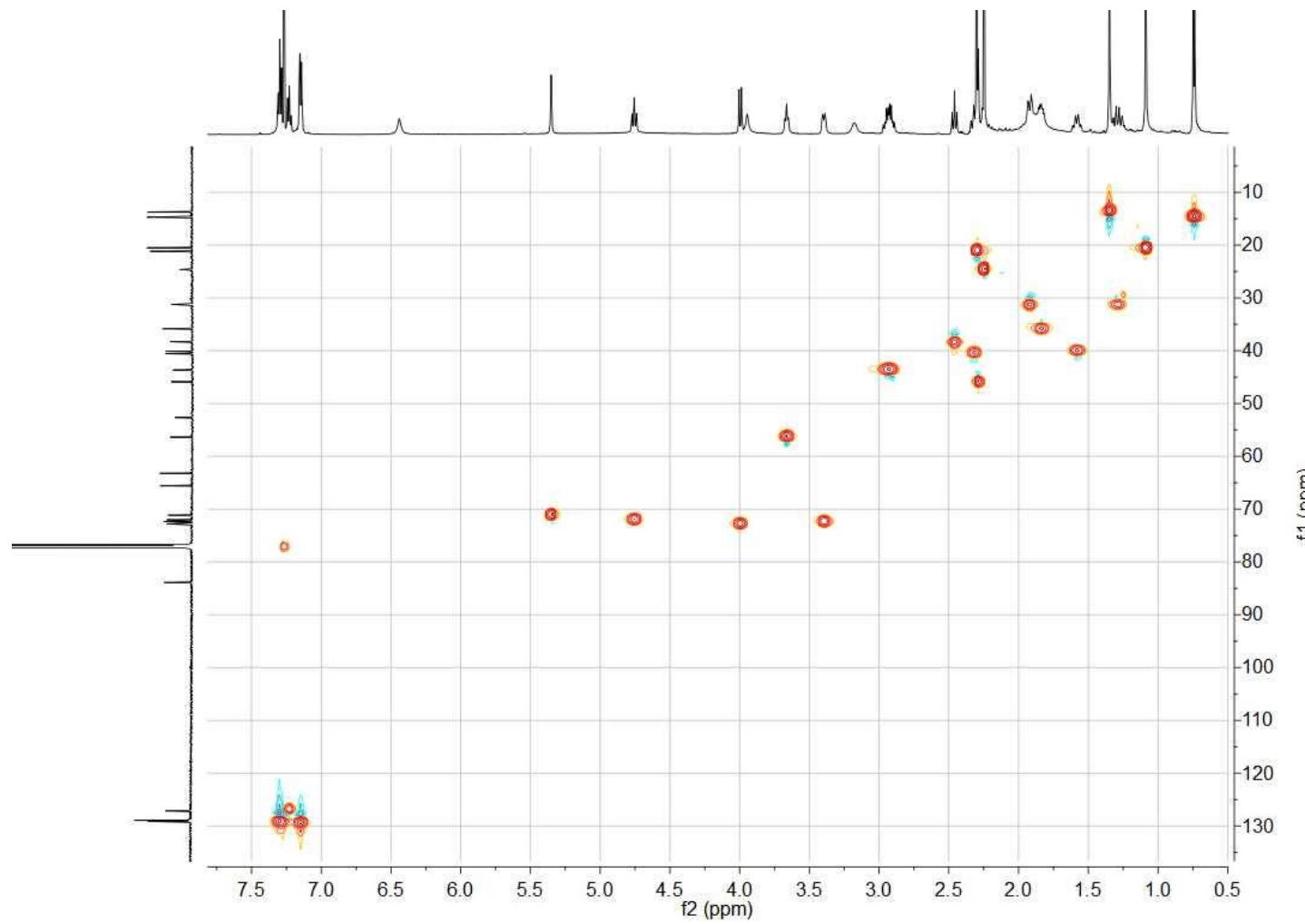
**Fig. S1.**  $^1\text{H}$  NMR of **1** ( $\text{CDCl}_3$ , 600 MHz).



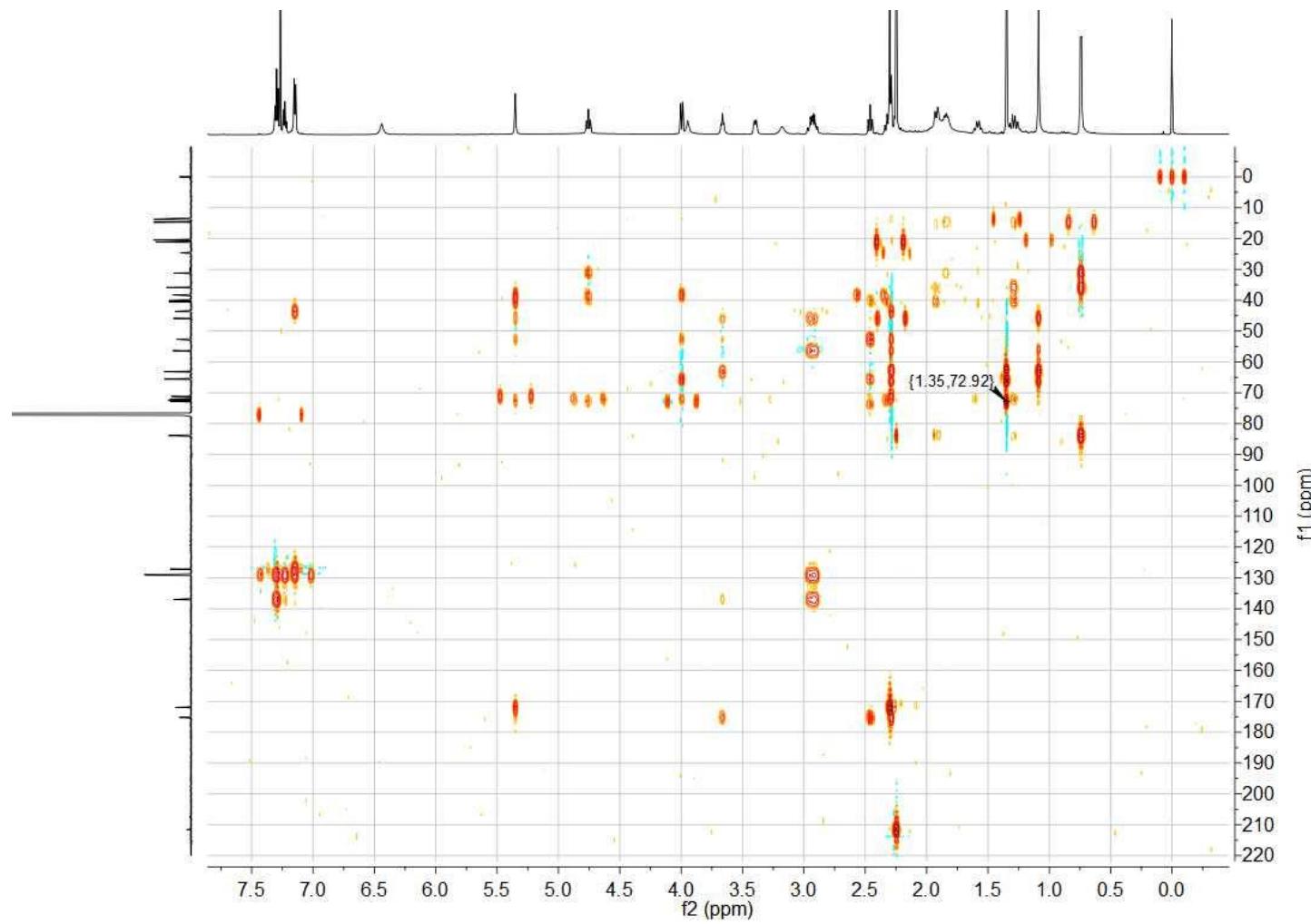
**Fig. S2.**  $^{13}\text{C}$  NMR of **1** ( $\text{CDCl}_3$ , 150 MHz).



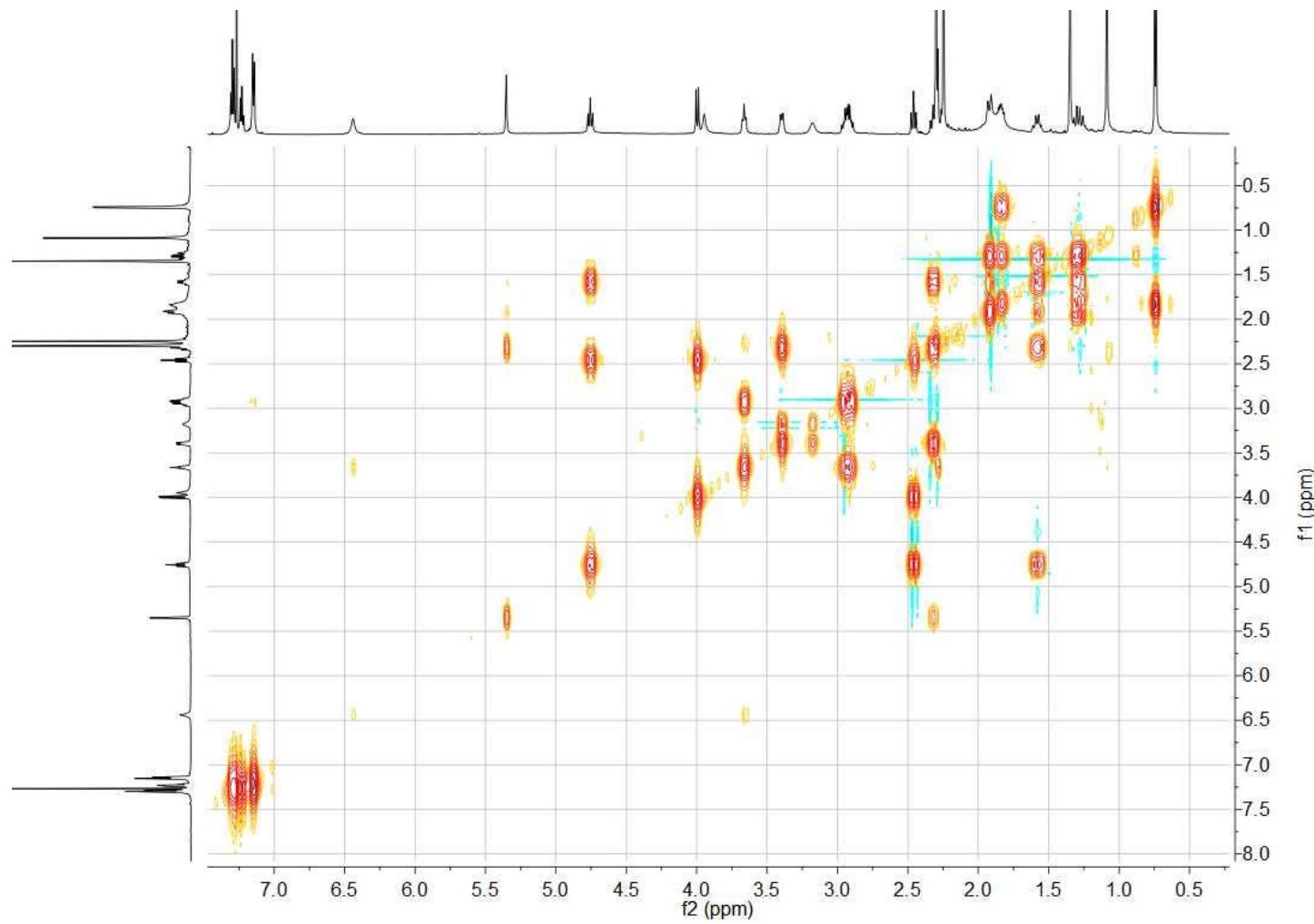
**Fig. S3.** DEPT of **1**.



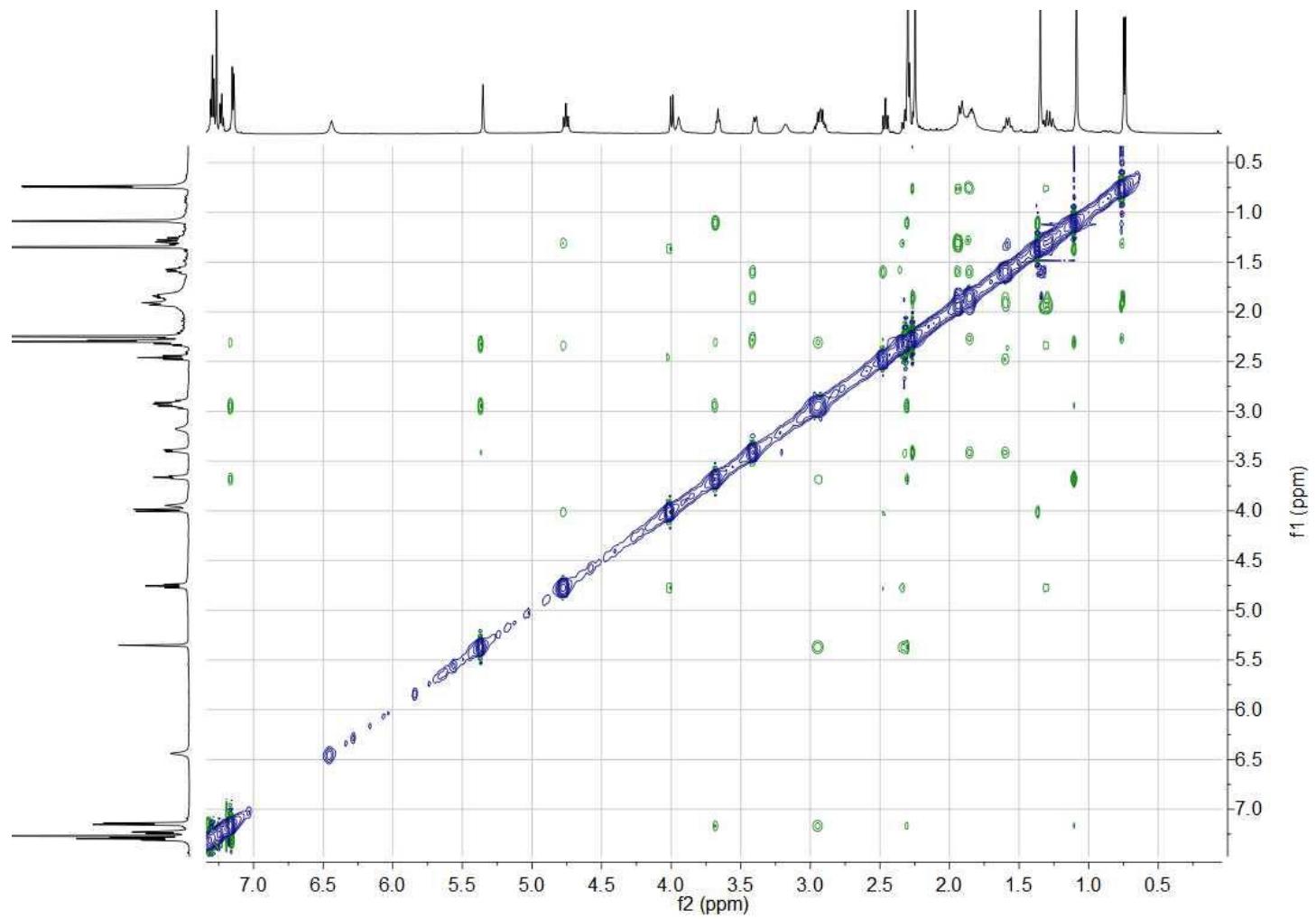
**Fig. S4.** HSQC of **1**.



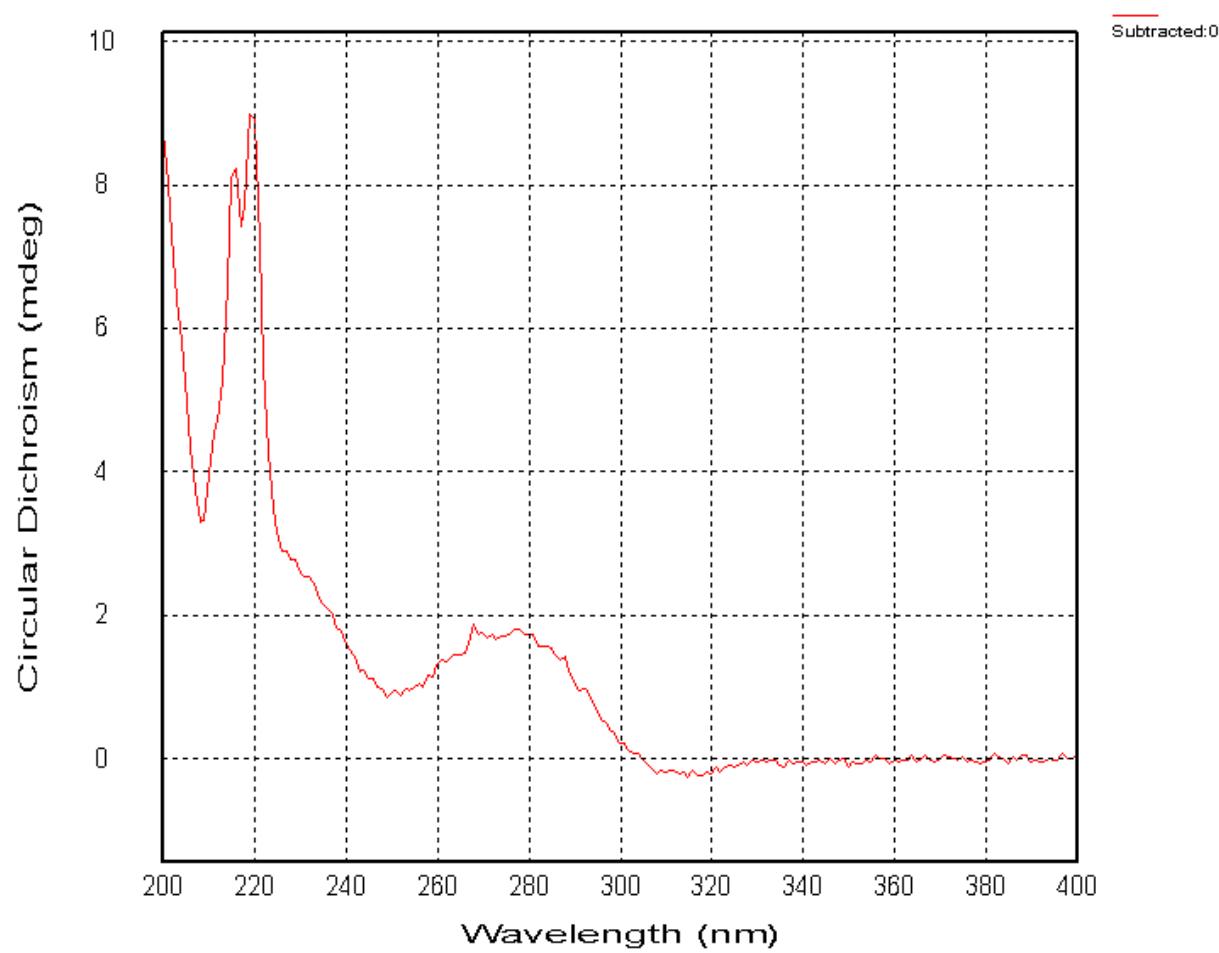
**Fig. S5.** HMBC of **1**.



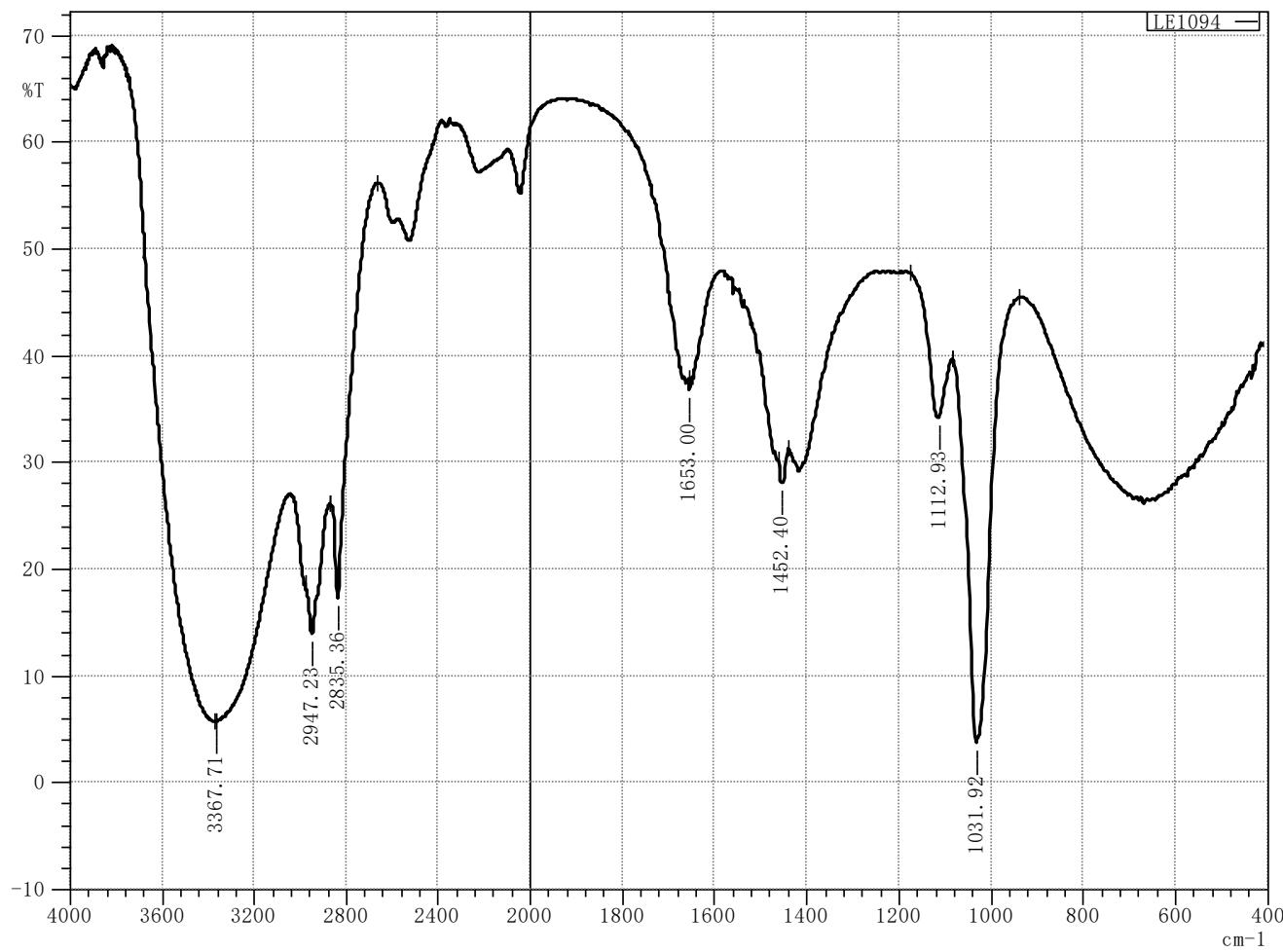
**Fig. S6.**  $^1\text{H}$ - $^1\text{H}$  COSY of **1**.



**Fig. S7.** ROESY of **1**.



**Fig. S8.** Experimental ECD of **1**.

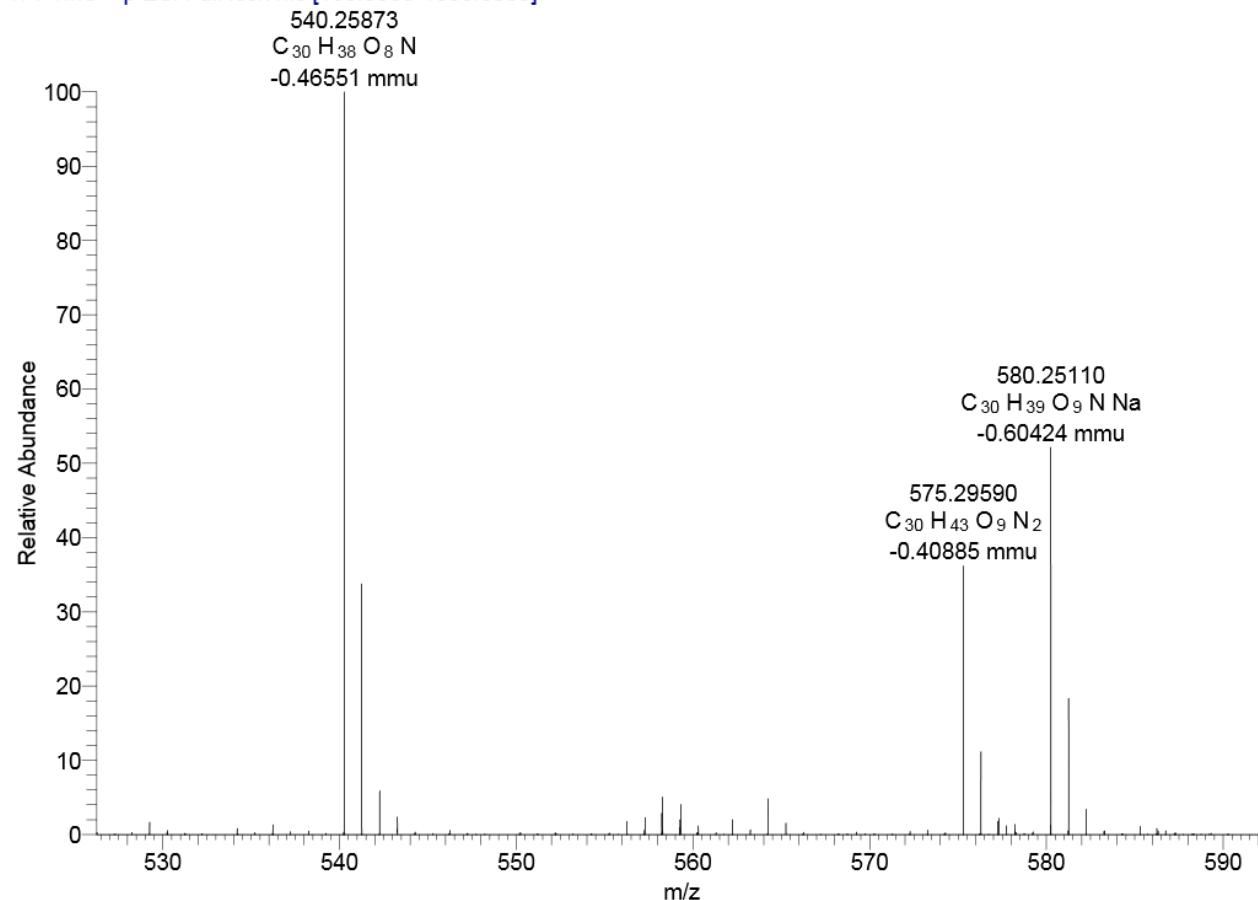


**Fig. S9.** IR spectrum of **1**.

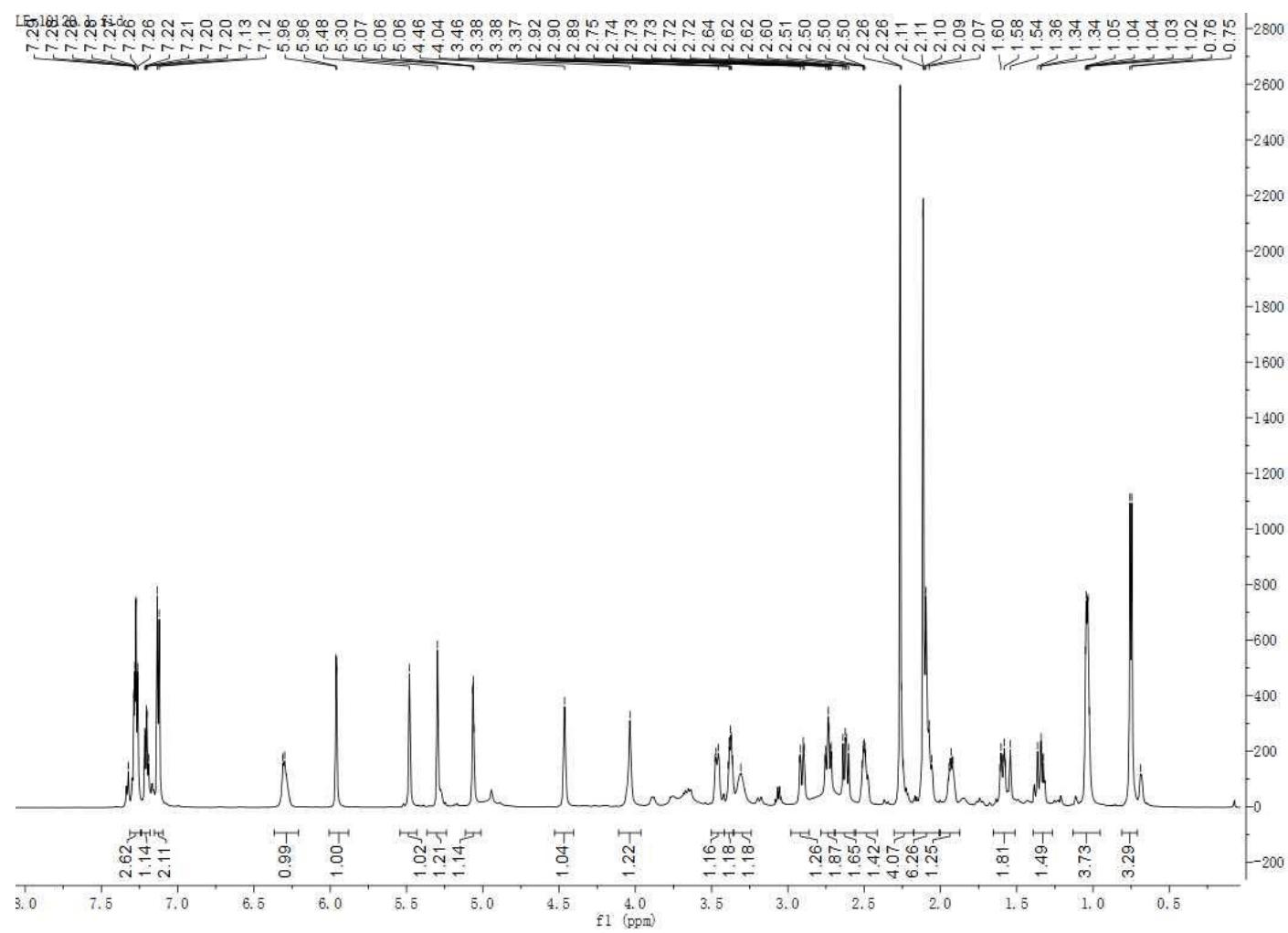
D:\Wen-Xuan\2019\20190604\LE1094

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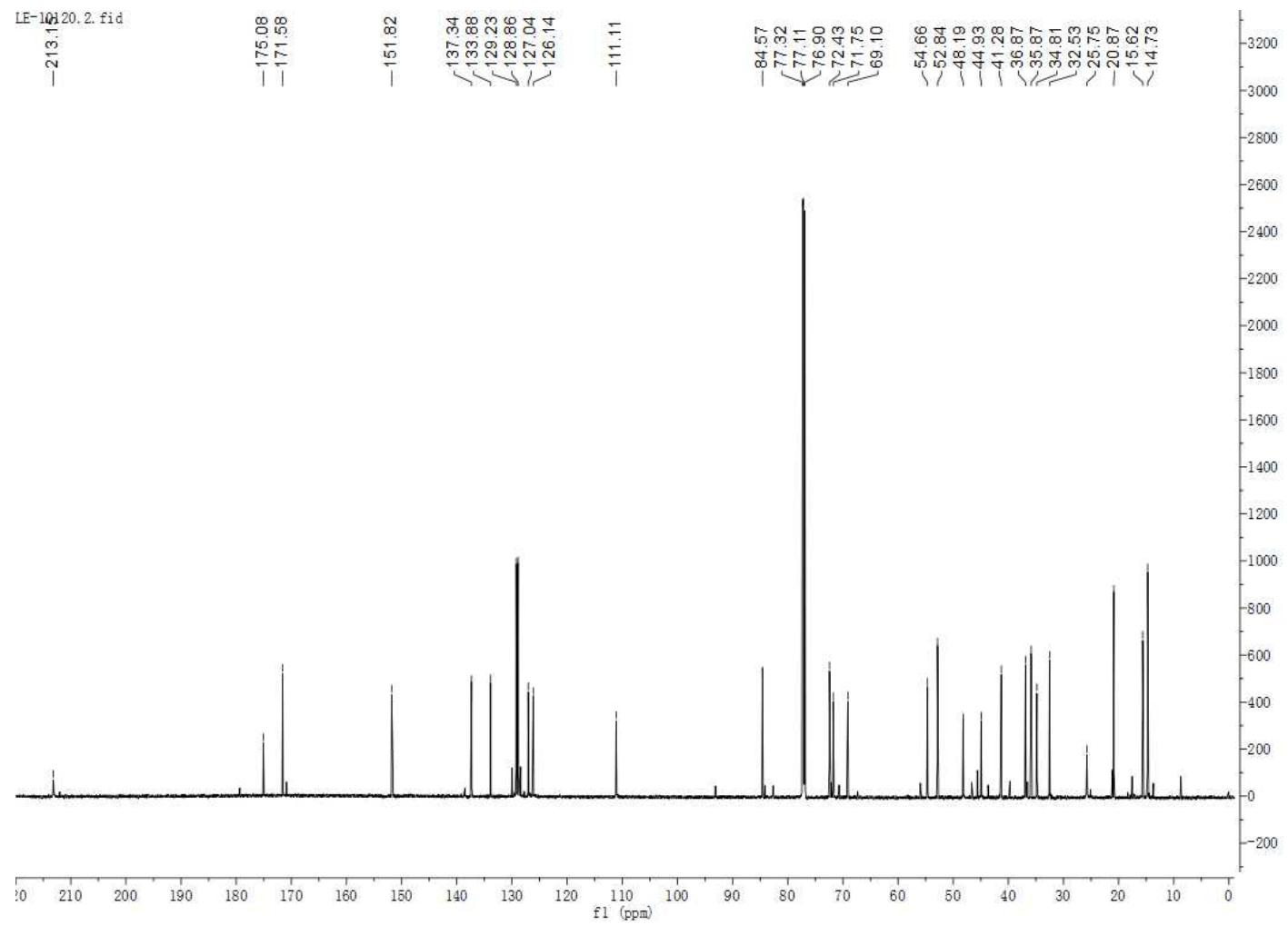
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T: FTMS + p ESI Full lock ms [100.0000-1000.0000]



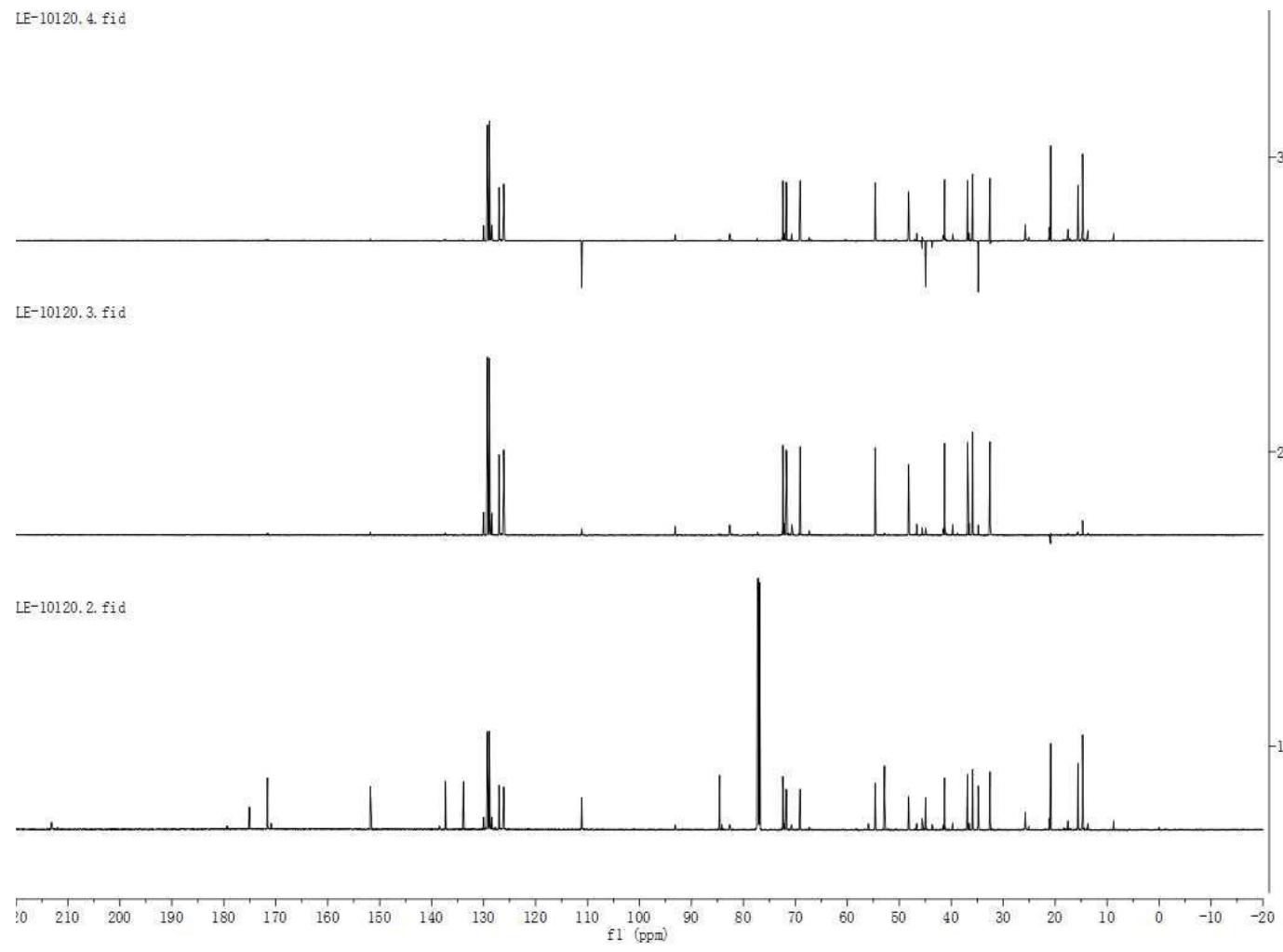
**Fig. S10.** HRESIMS of **1**.



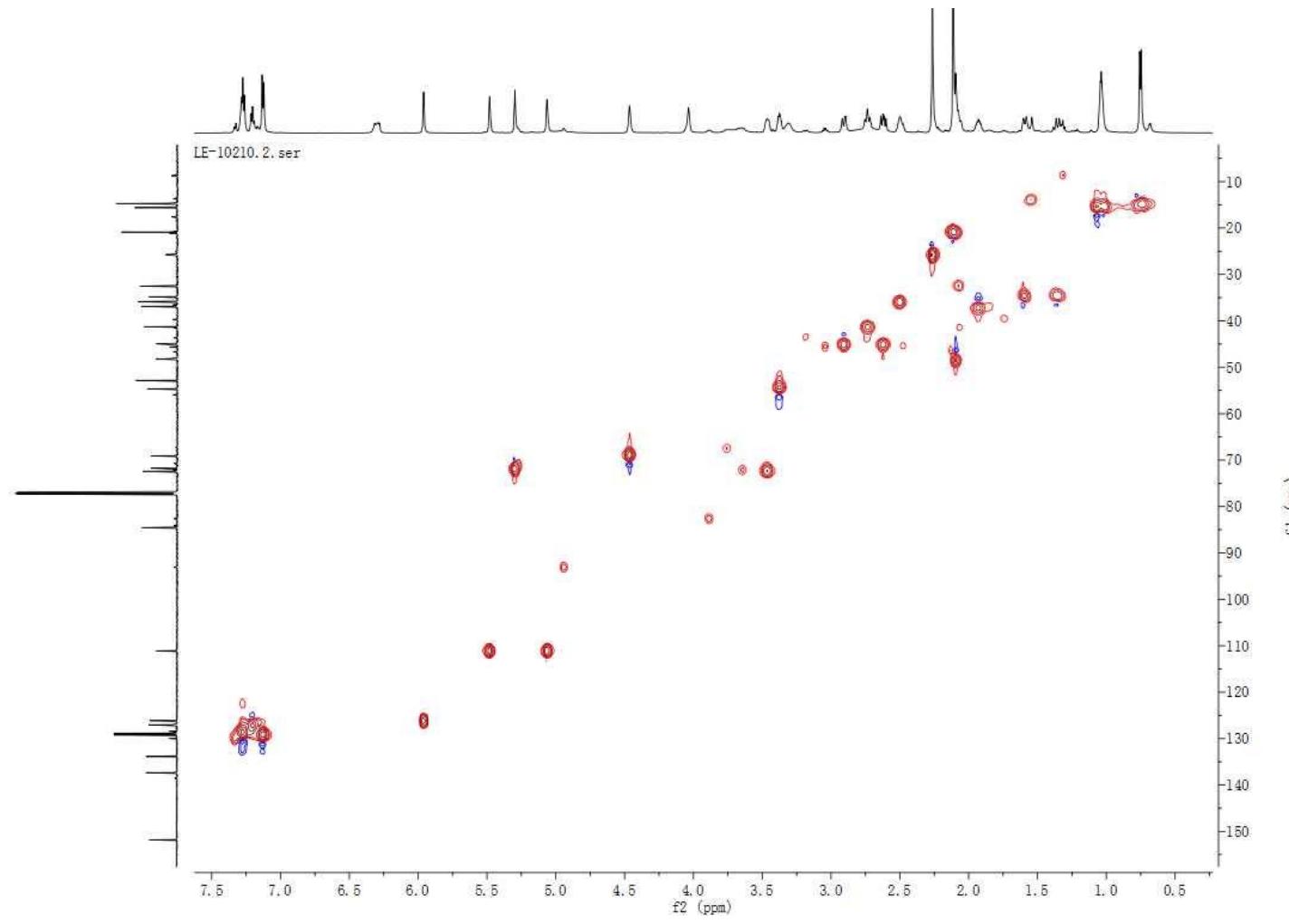
**Fig. S11.**  $^1\text{H}$  NMR of **2** ( $\text{CDCl}_3$ , 600 MHz).



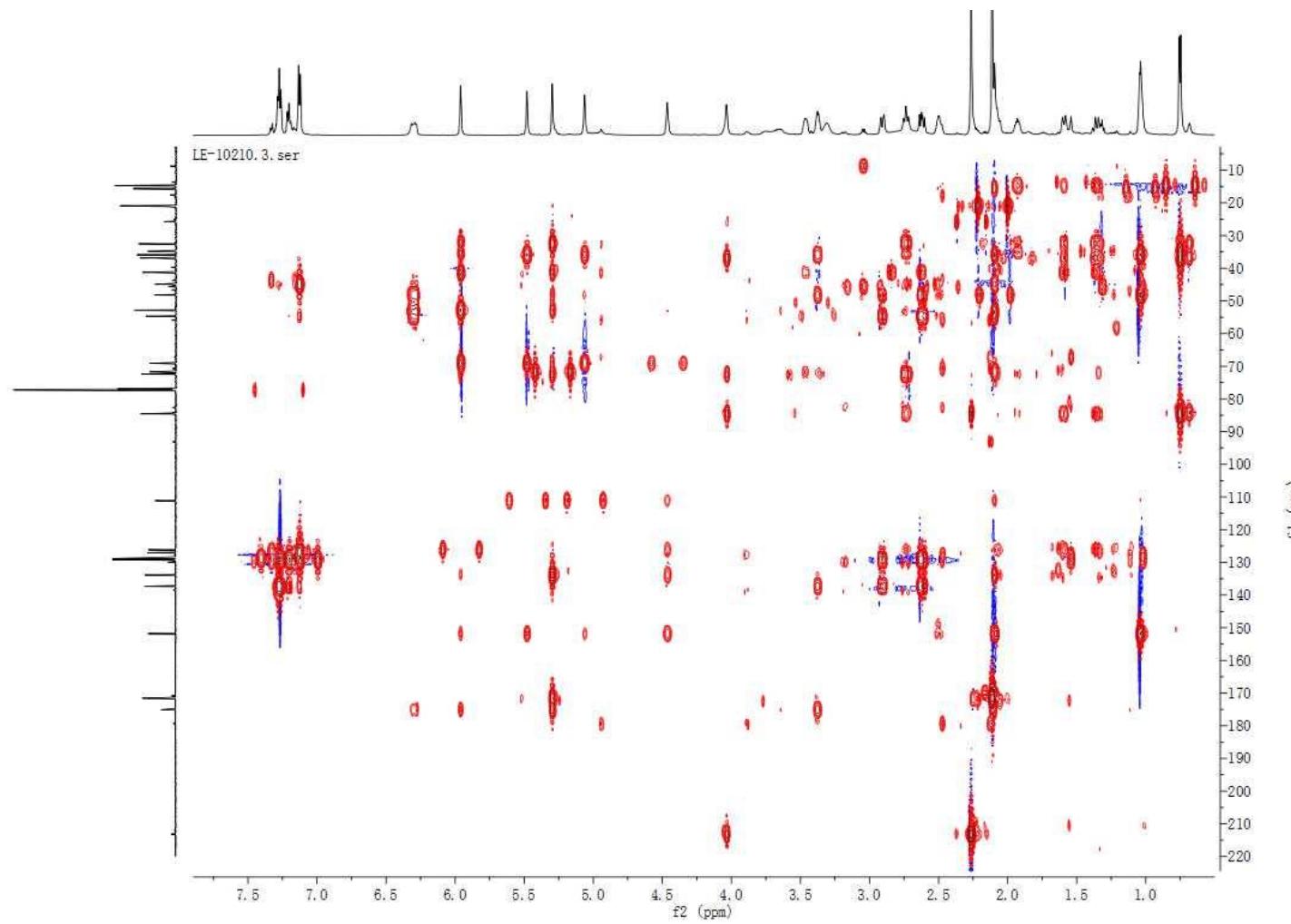
**Fig. S12.**  $^{13}\text{C}$  NMR of **2** ( $\text{CDCl}_3$ , 150 MHz).



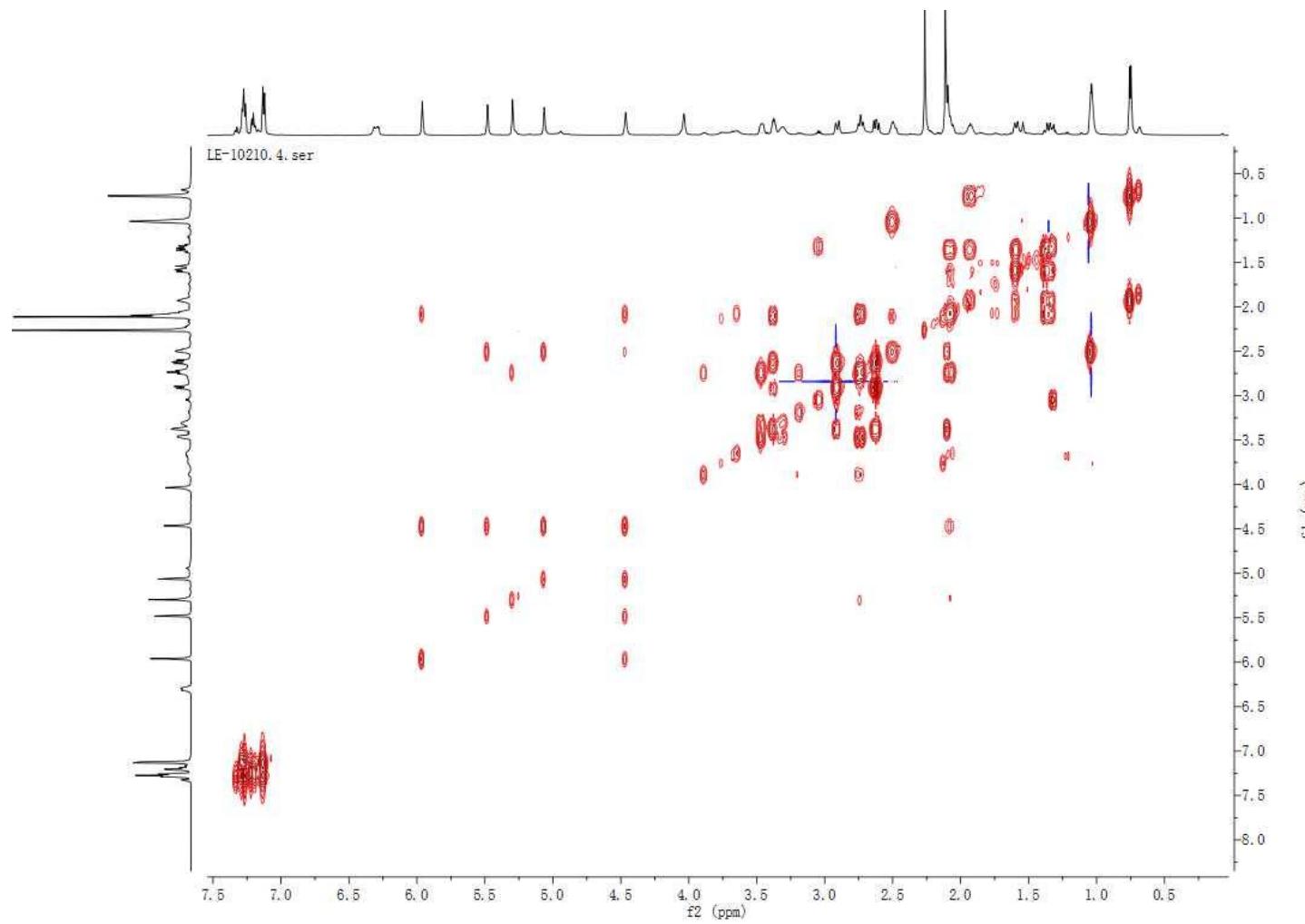
**Fig. S13.** DEPT of 2.



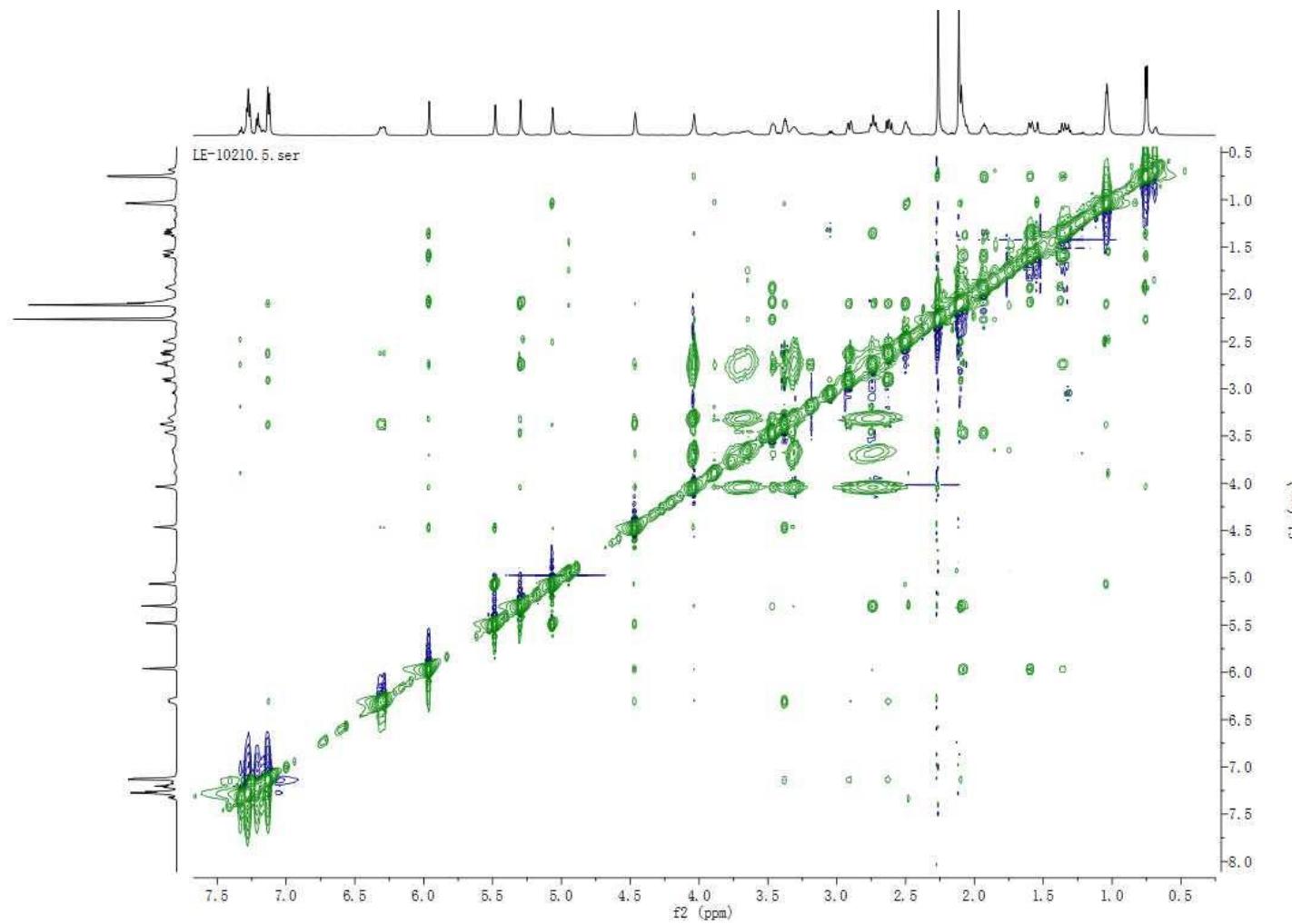
**Fig. S14.** HSQC of **2**.



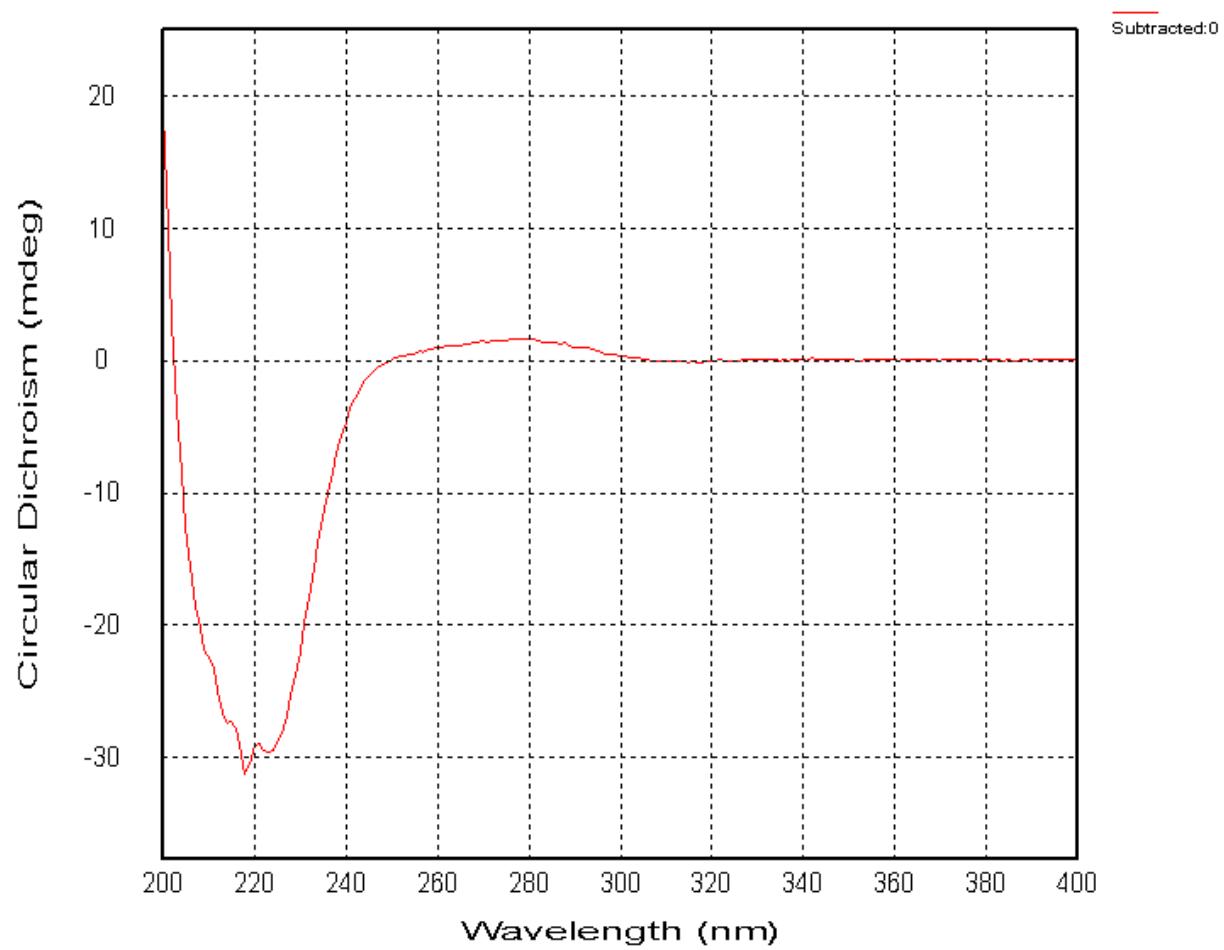
**Fig. S15.** HMBC of **2**.



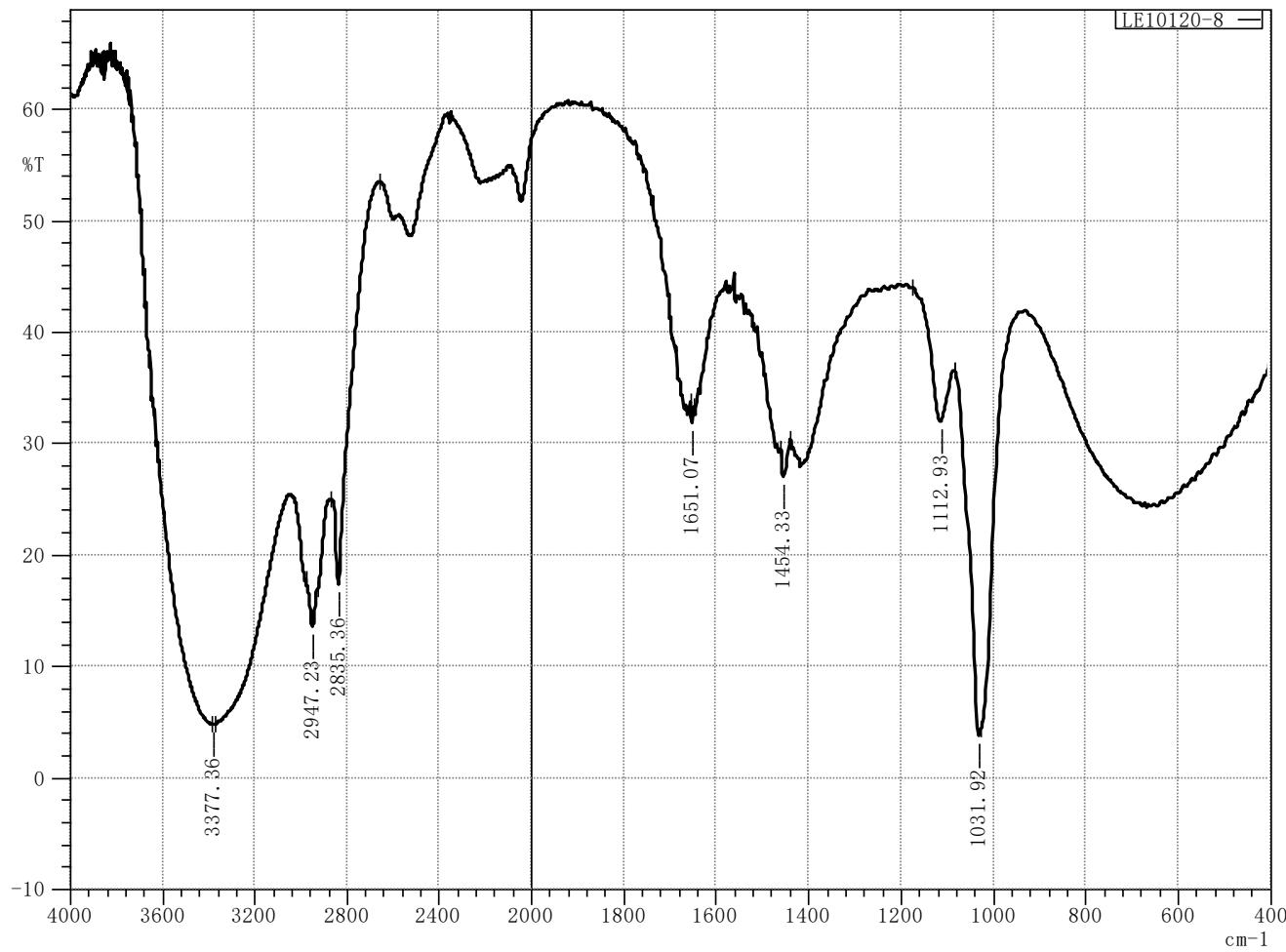
**Fig. S16.**  $^1\text{H}$ - $^1\text{H}$  COSY of **2**.



**Fig. S17.** ROESY of **2**.



**Fig. S18.** Experimental ECD of **2**.

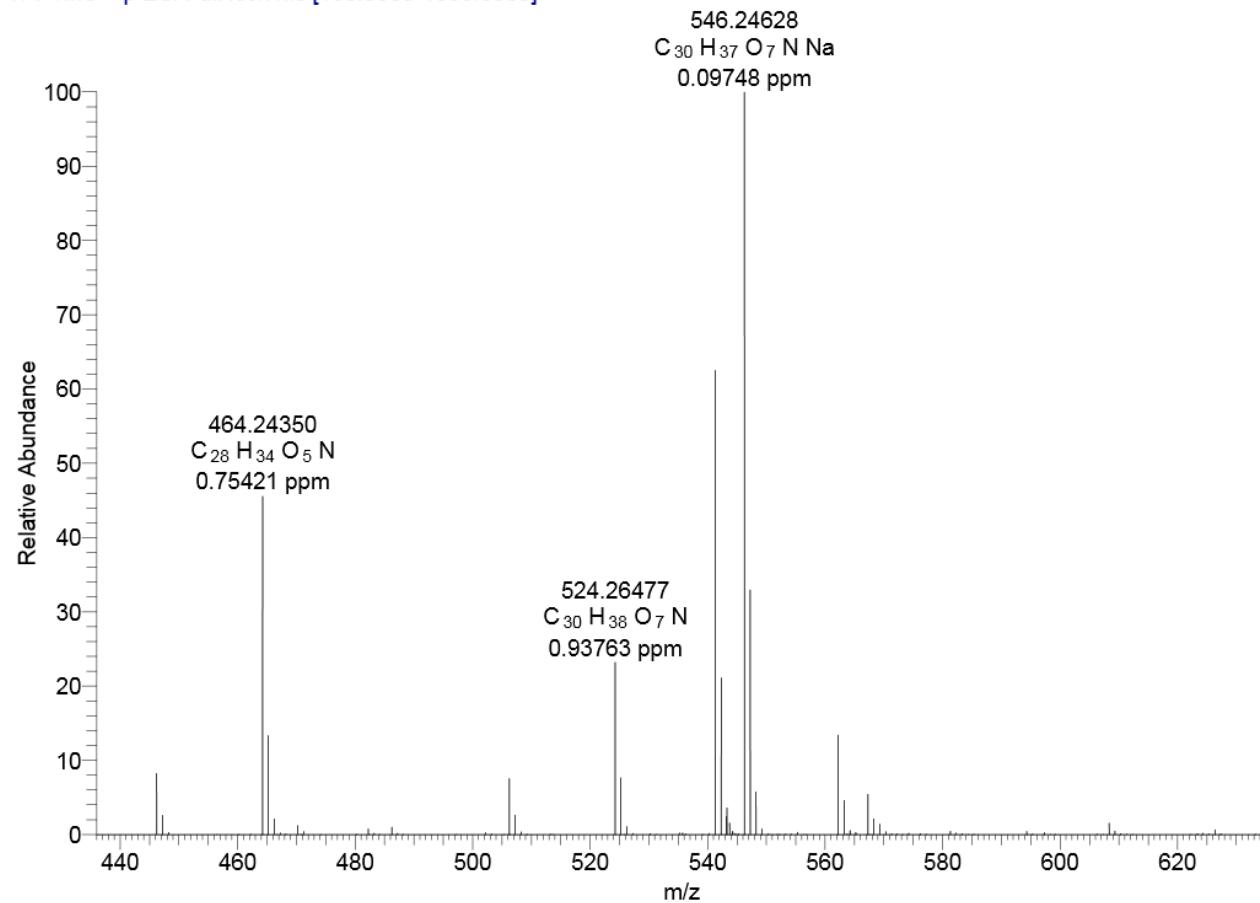


**Fig. S19.** IR of **2**.

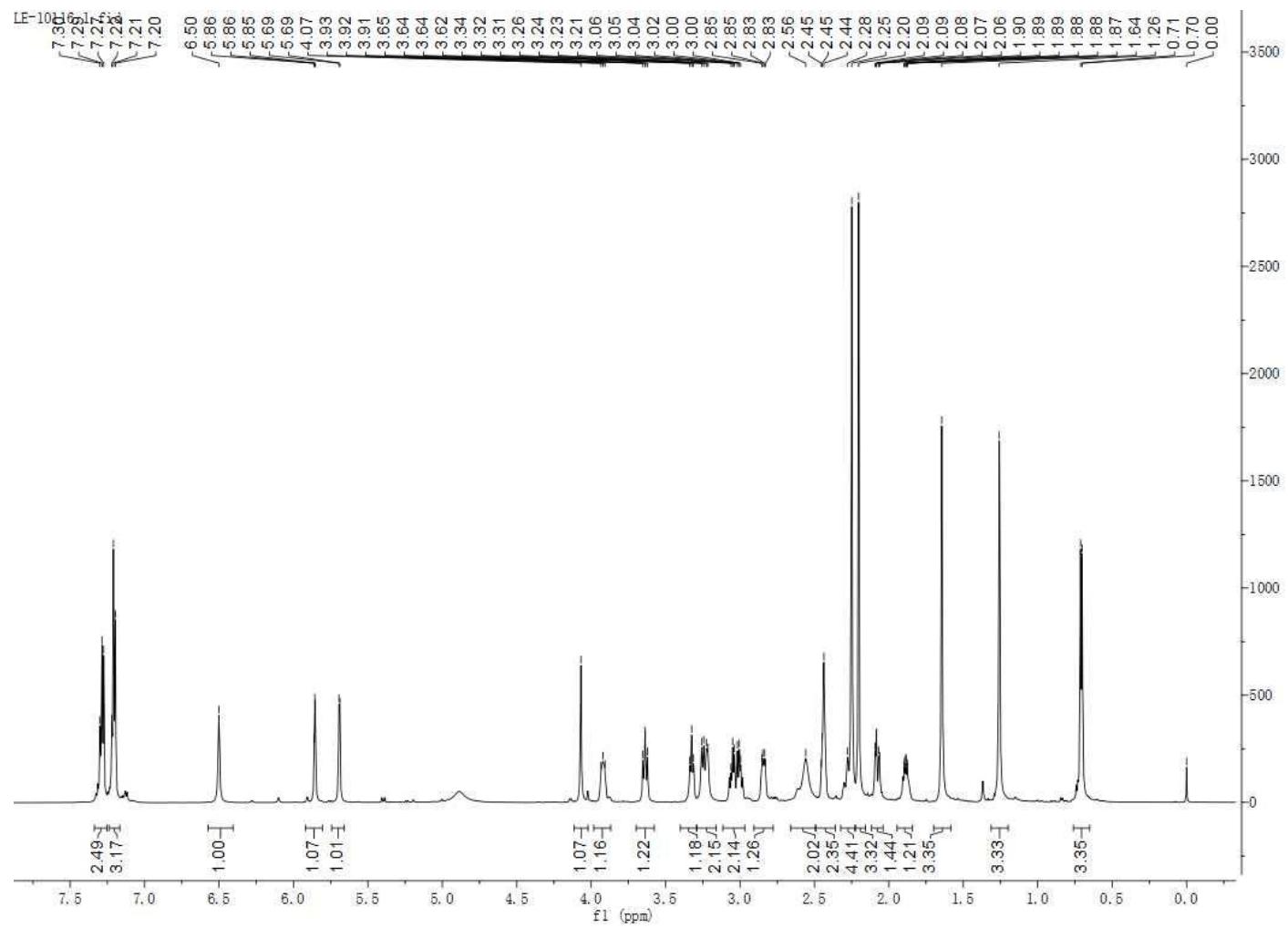
D:\Wen-Xuan\2019\20190604\LE10120

07/02/19 19:19:21

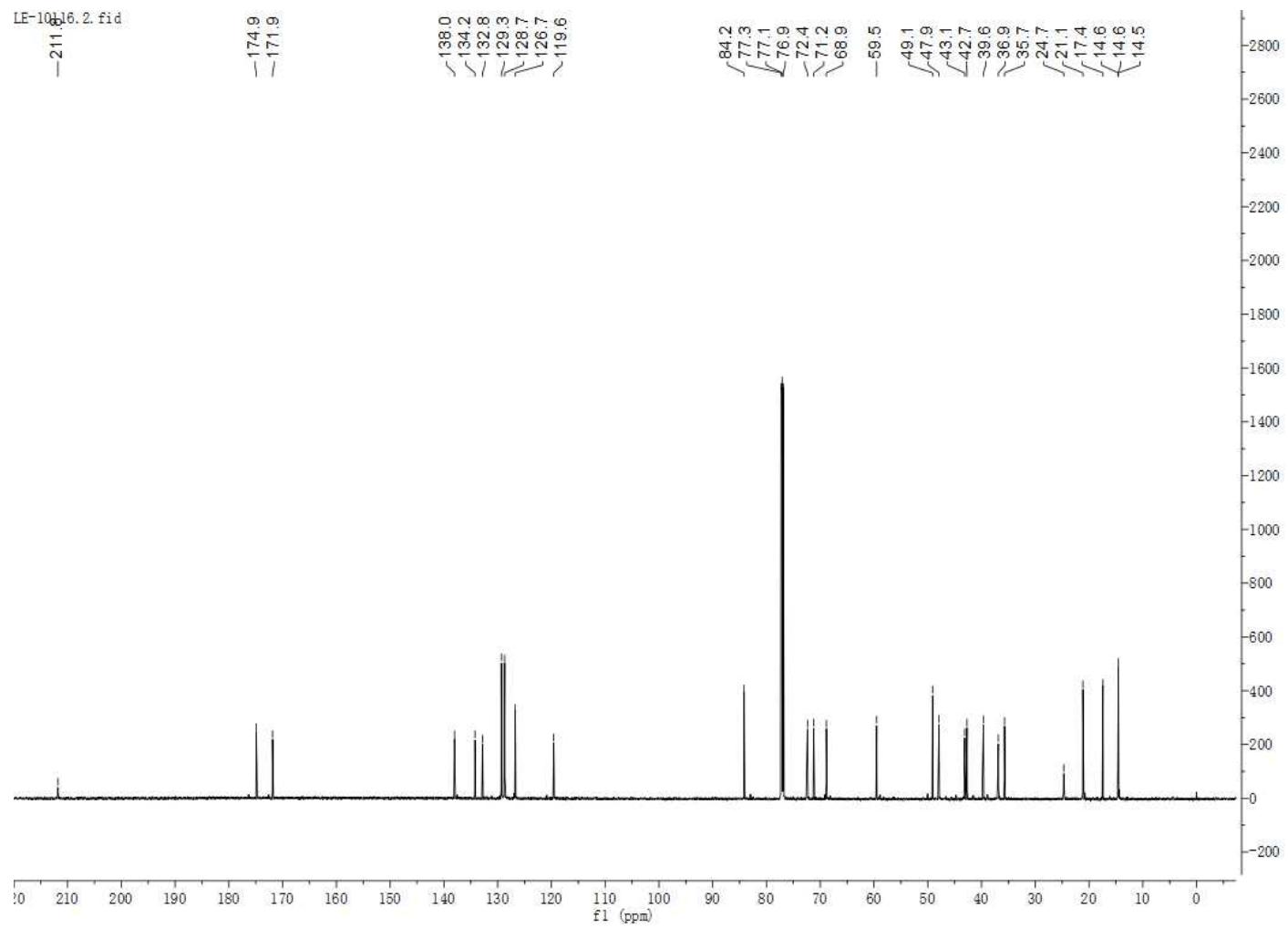
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T: FTMS + p ESI Full lock ms [100.0000-1000.0000]



**Fig. S20.** HRESIMS of **2**.

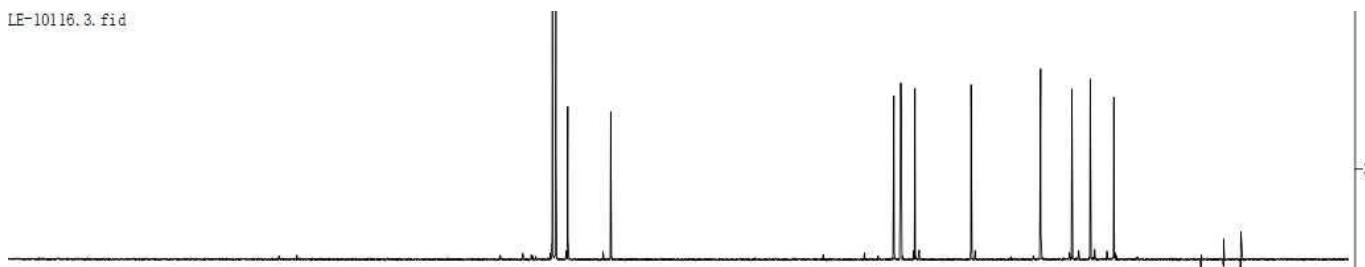


**Fig. S21.** <sup>1</sup>H NMR of **3** (CDCl<sub>3</sub>, 600 MHz).

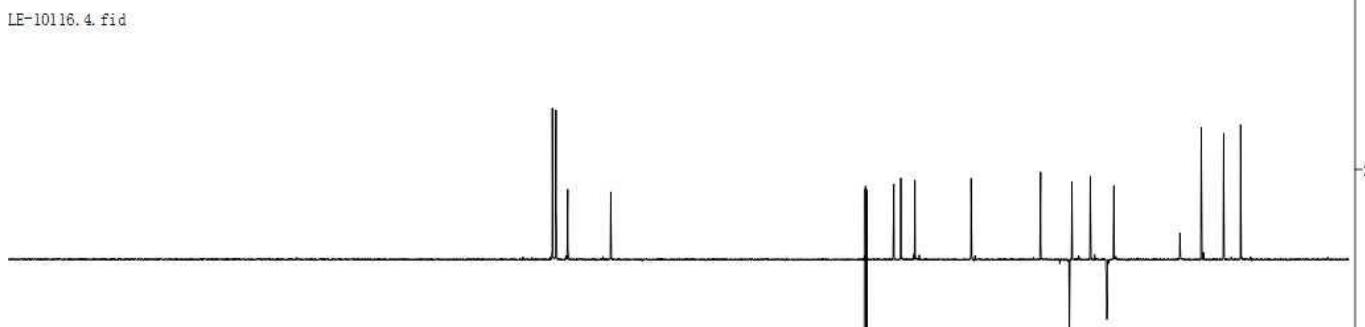


**Fig. S22.**  $^{13}\text{C}$  NMR of **3** ( $\text{CDCl}_3$ , 150 MHz).

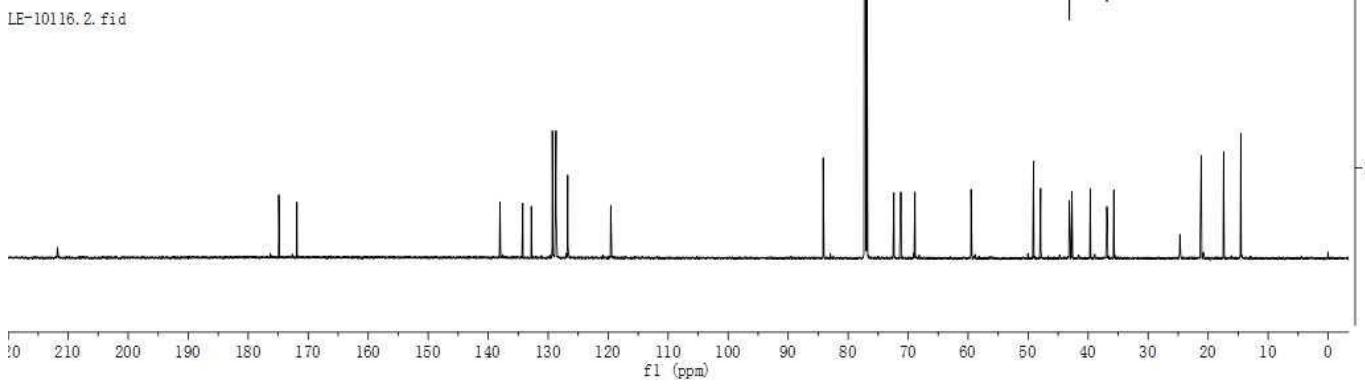
LE-10116.3.fid



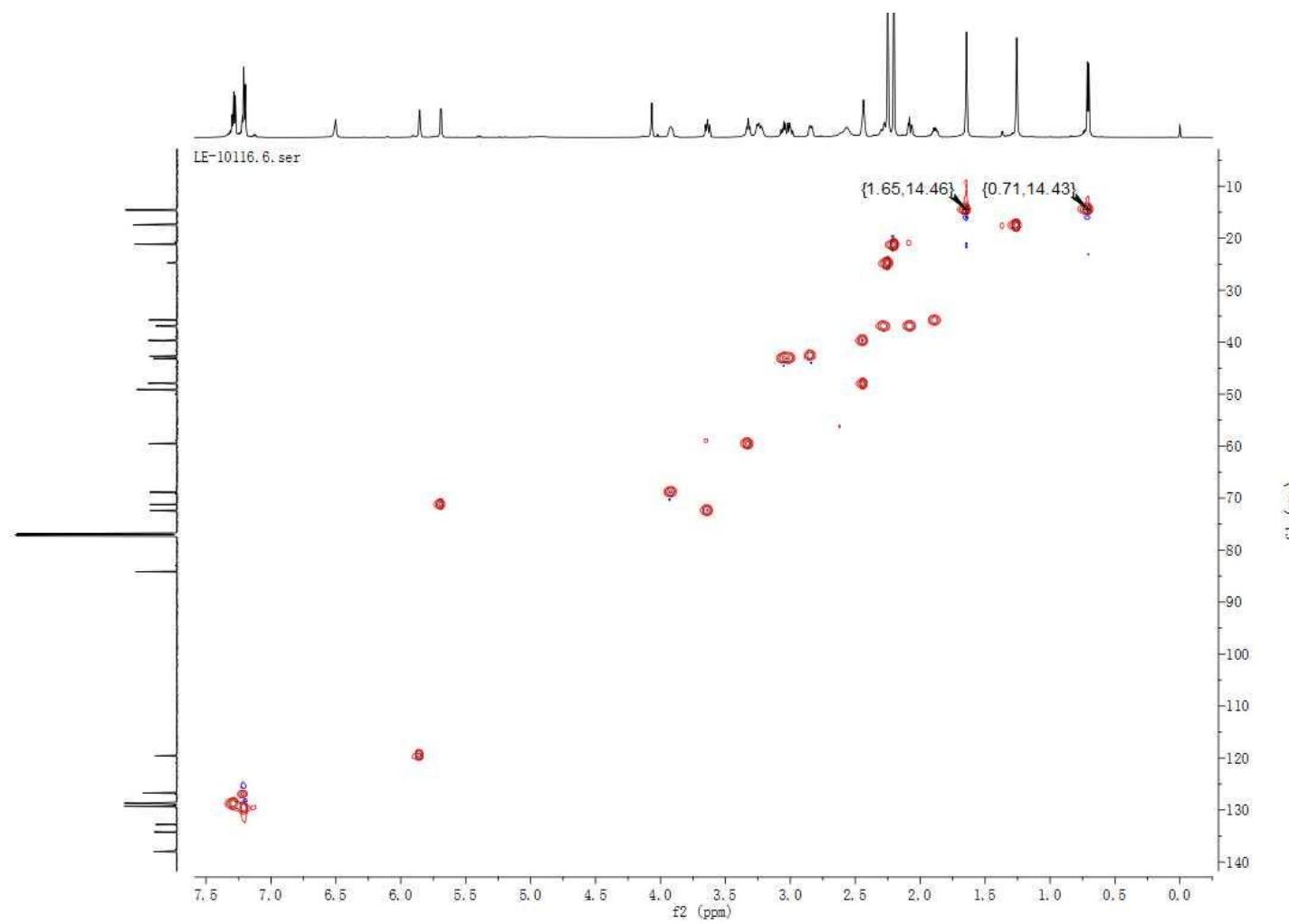
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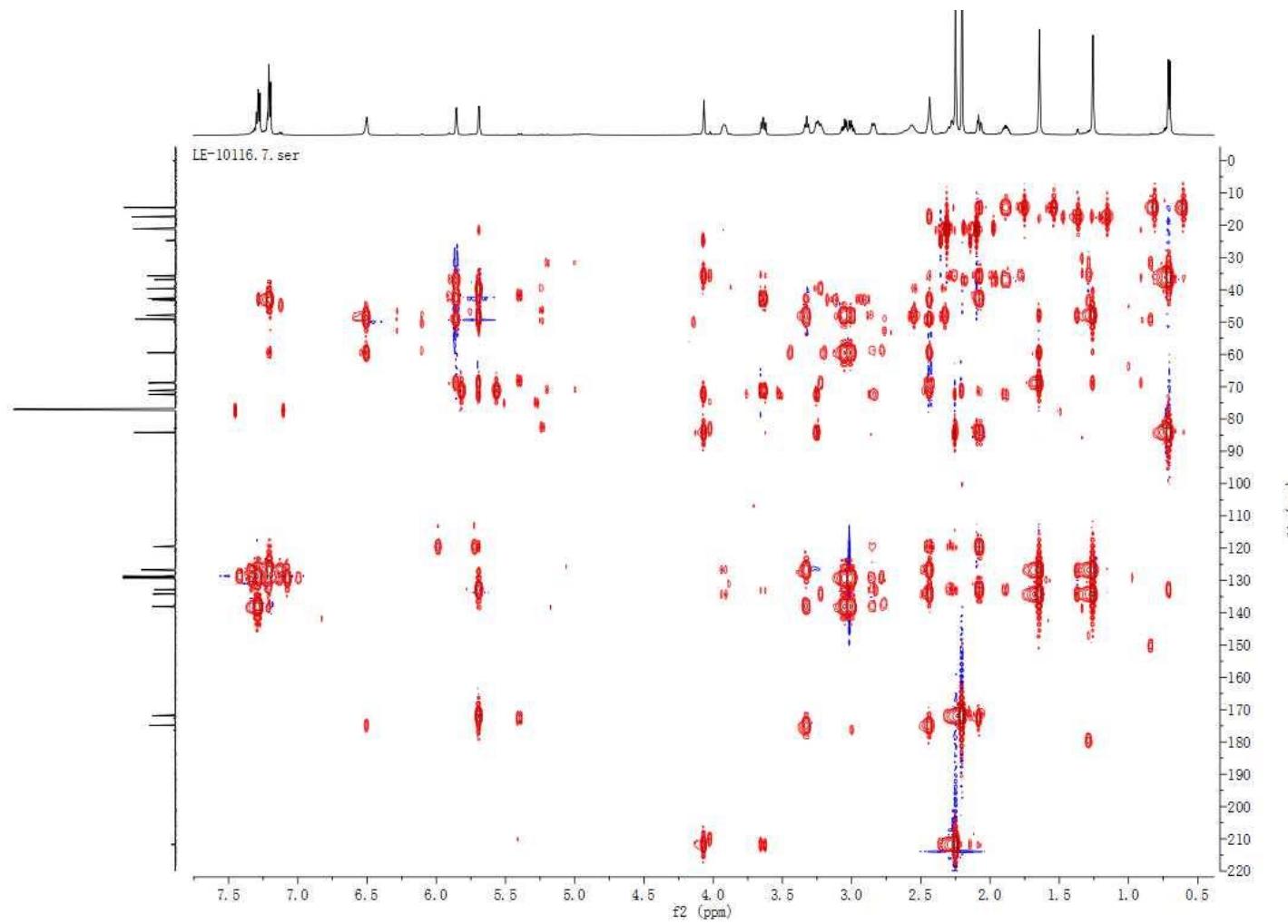
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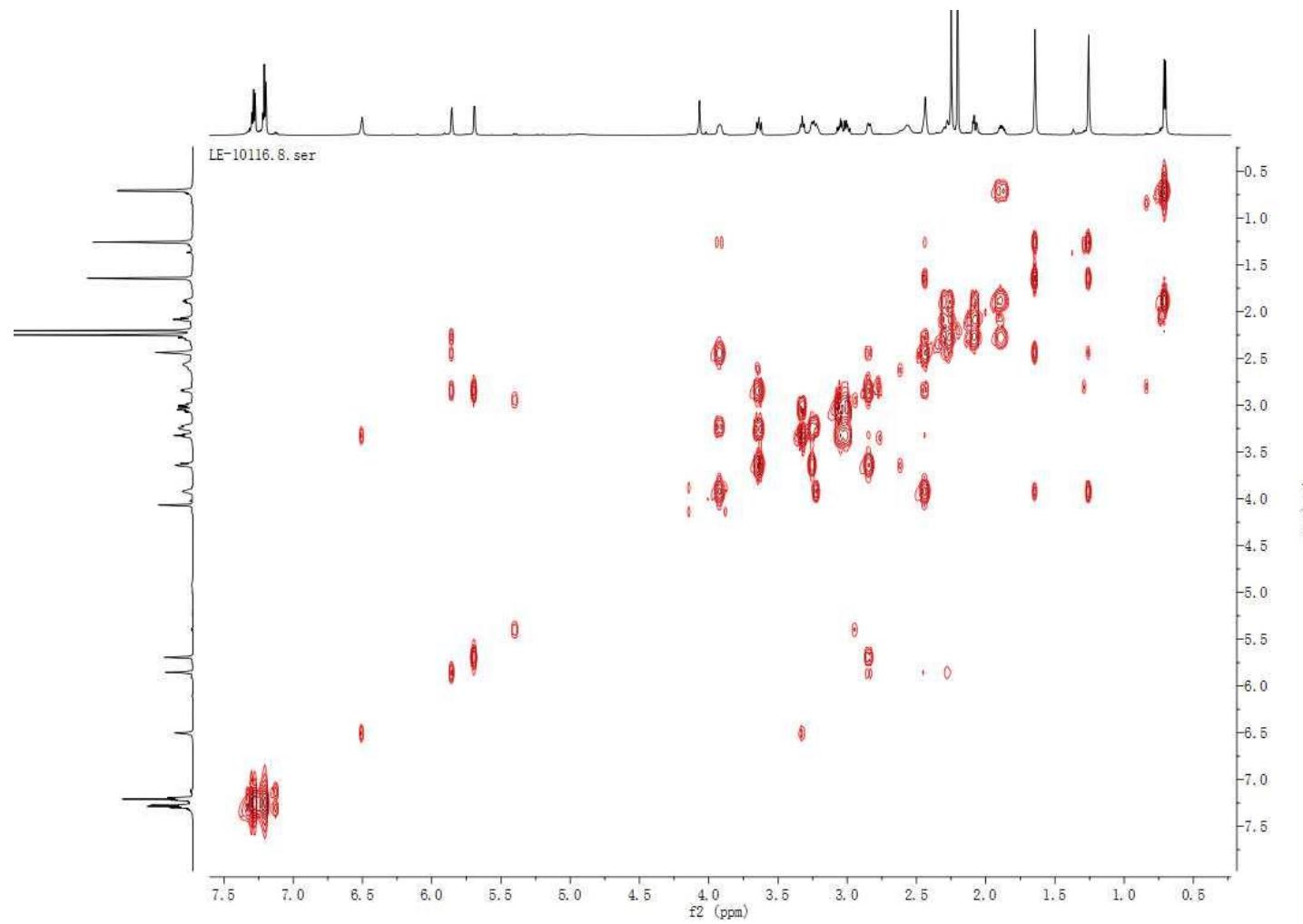
**Fig. S23.** DEPT of 3.



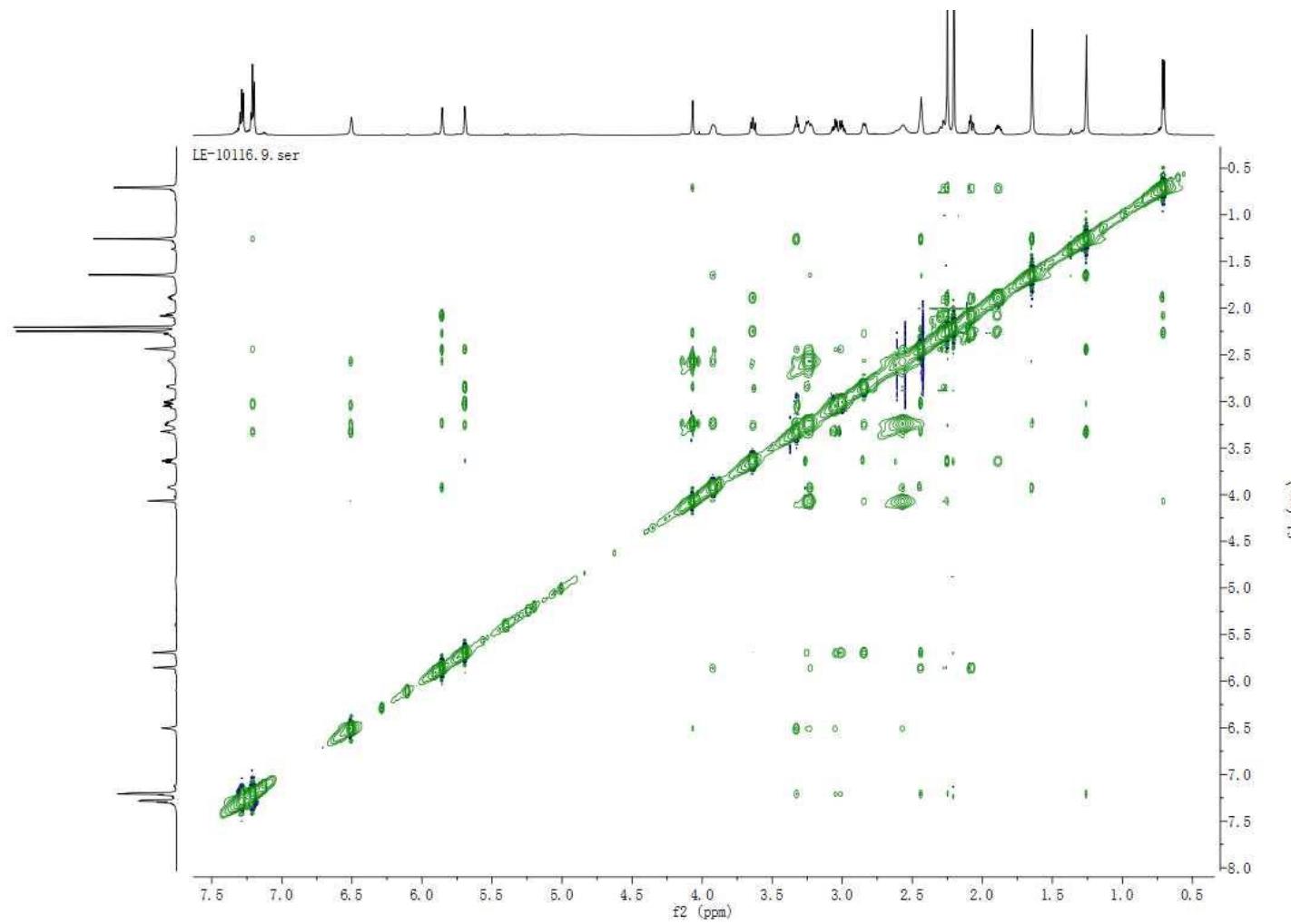
**Fig. S24.** HSQC of **3**.



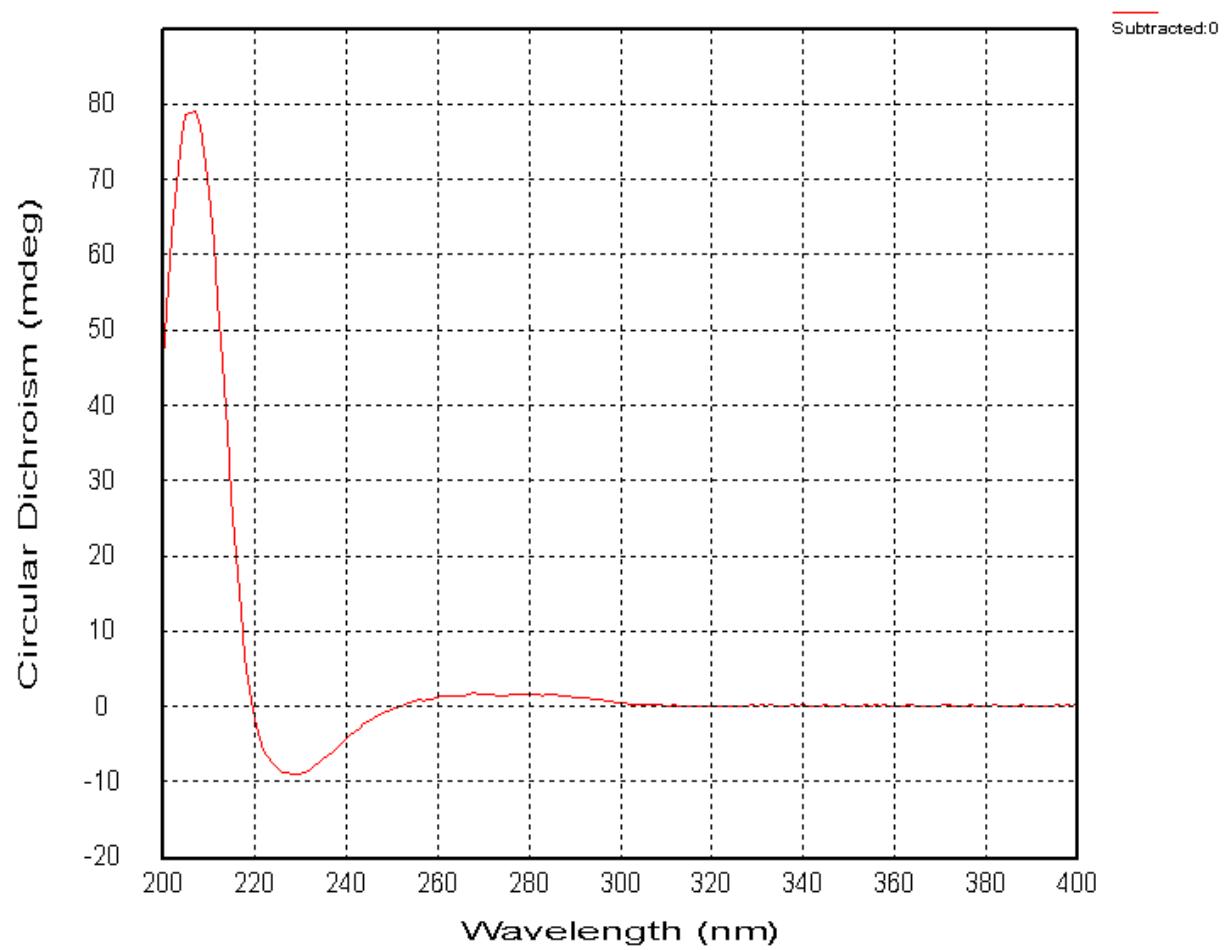
**Fig. S25.** HMBC of **3**.



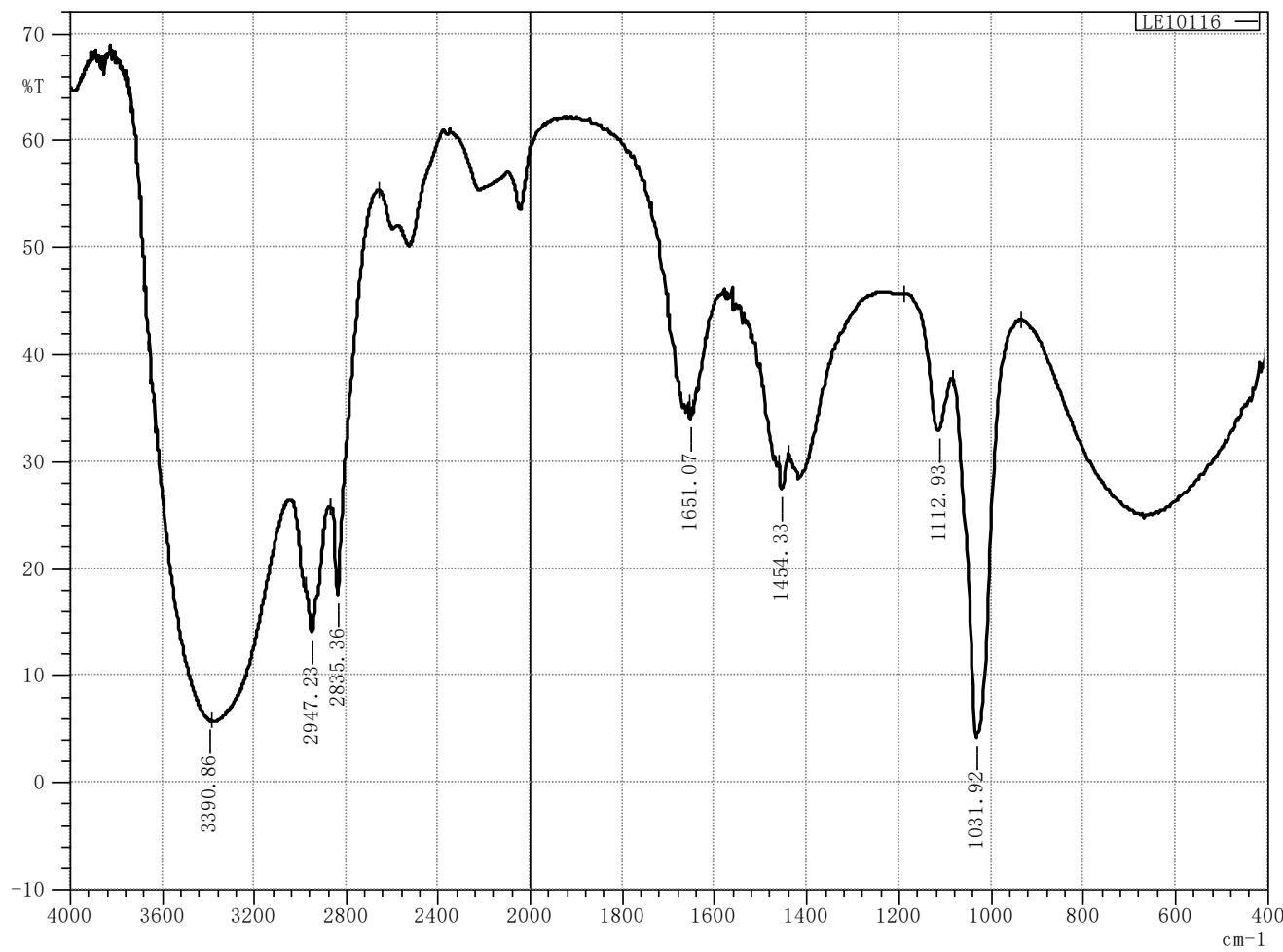
**Fig. S26.**  $^1\text{H}$ - $^1\text{H}$  COSY of **3**.



**Fig. S27.** ROESY of **3**.



**Fig. S28.** Experimental ECD of **3**.



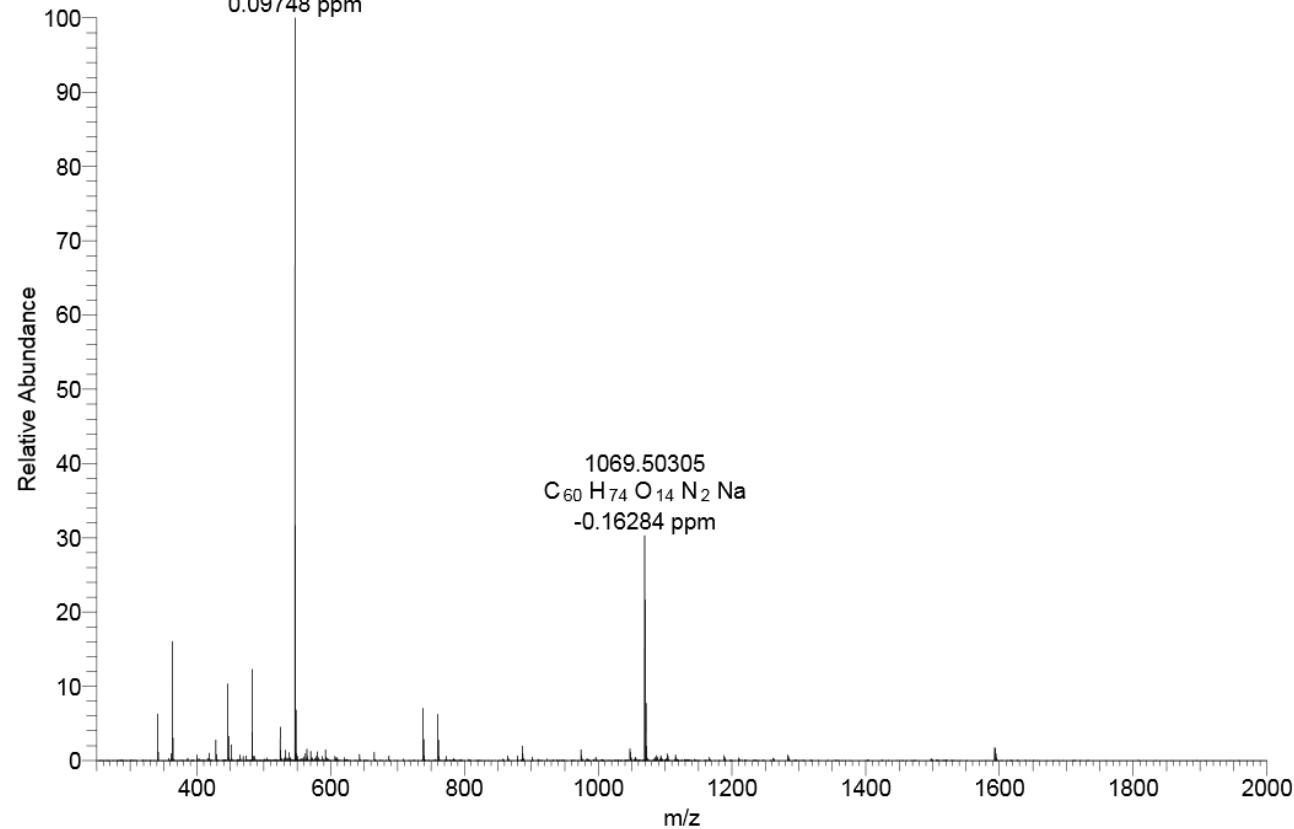
**Fig. S29.** IR of **3**.

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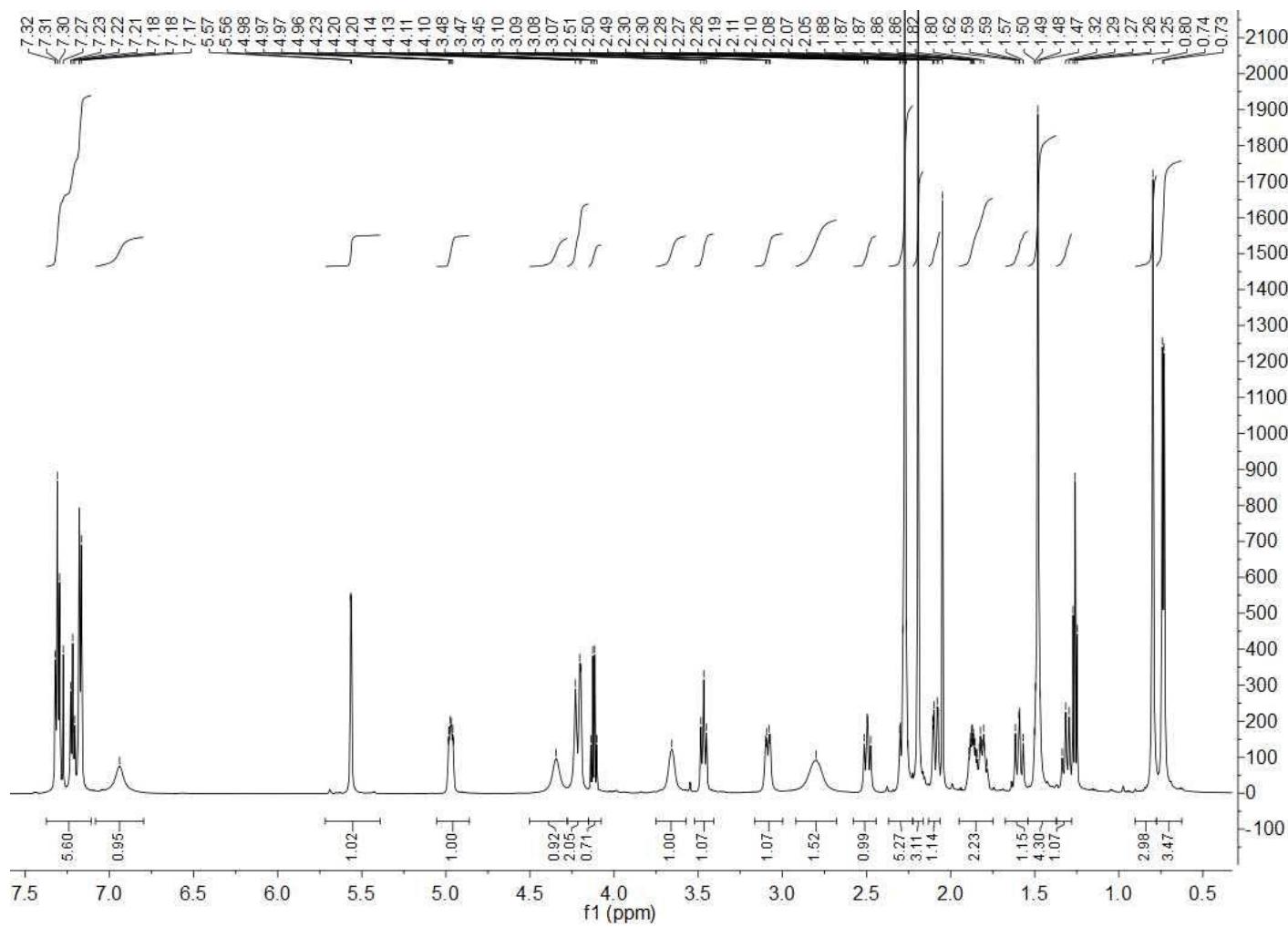
03/20/19 16:48:19

LE10-116 #1557 RT: 13.58 AV: 1 NL: 8.89E8  
T: FTMS + p ESI Full lock ms [250.0000-2000.0000]

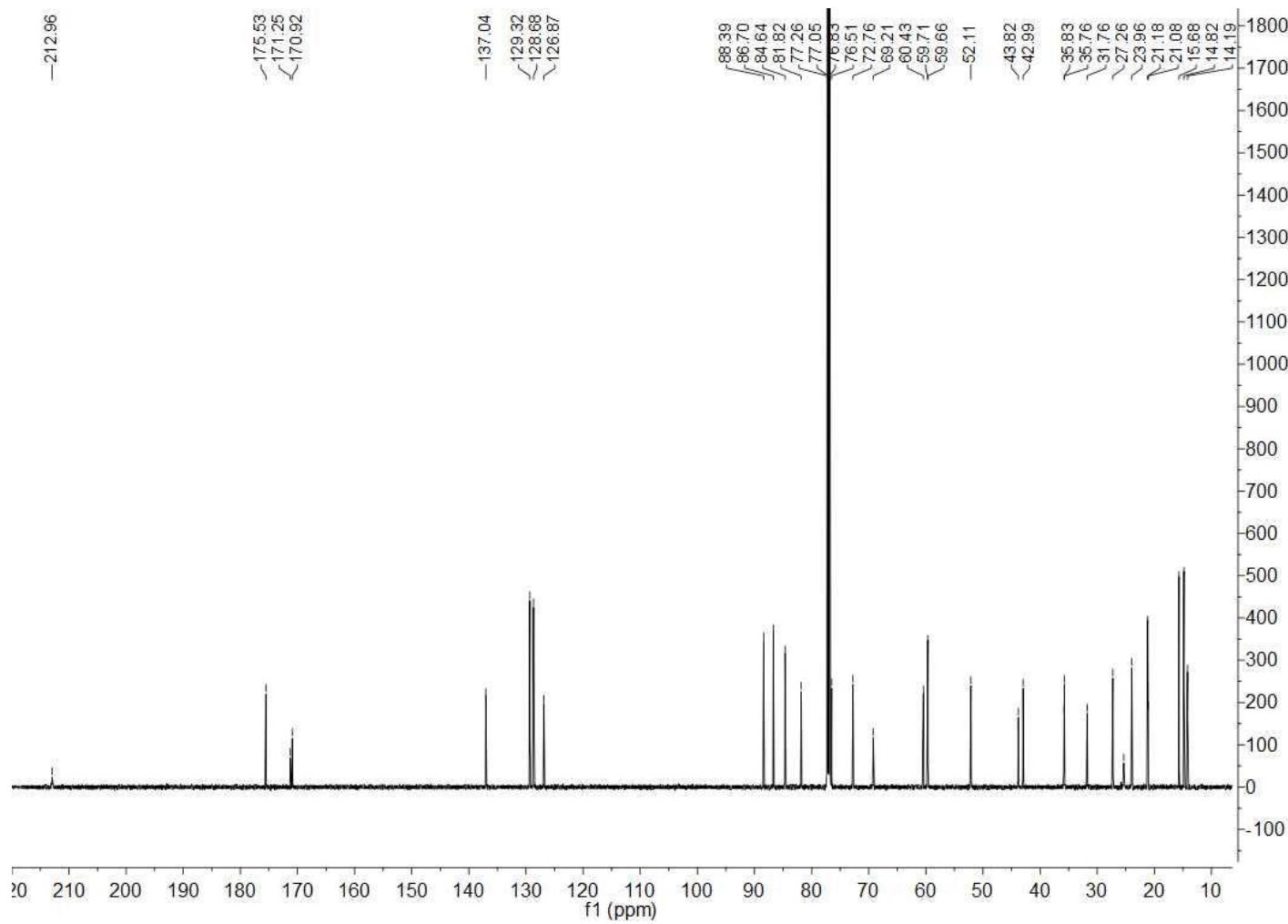
546.24628  
 $C_{30}H_{37}O_7NNa$   
0.09748 ppm



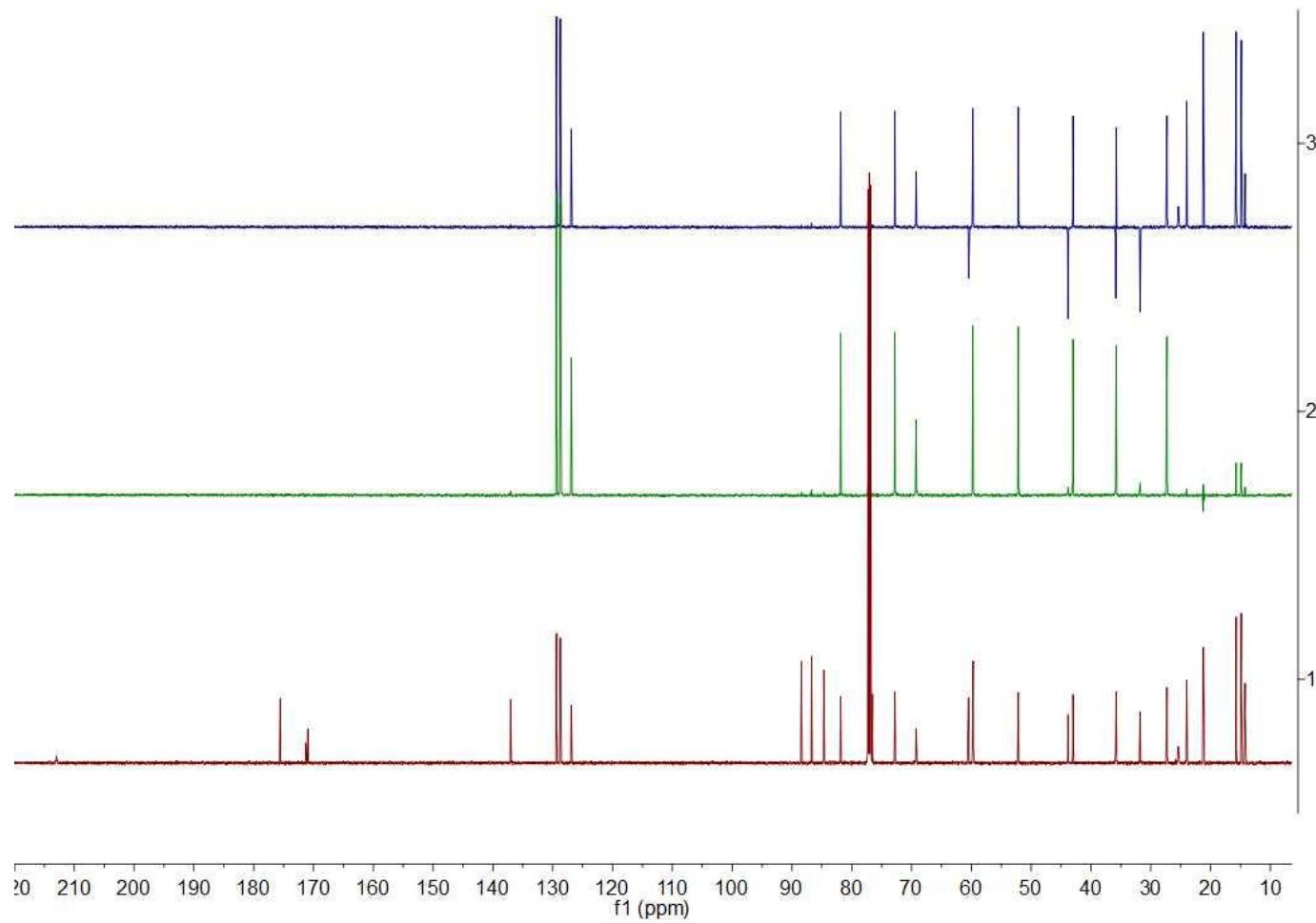
**Fig. S30.** HRESIMS of **3**.



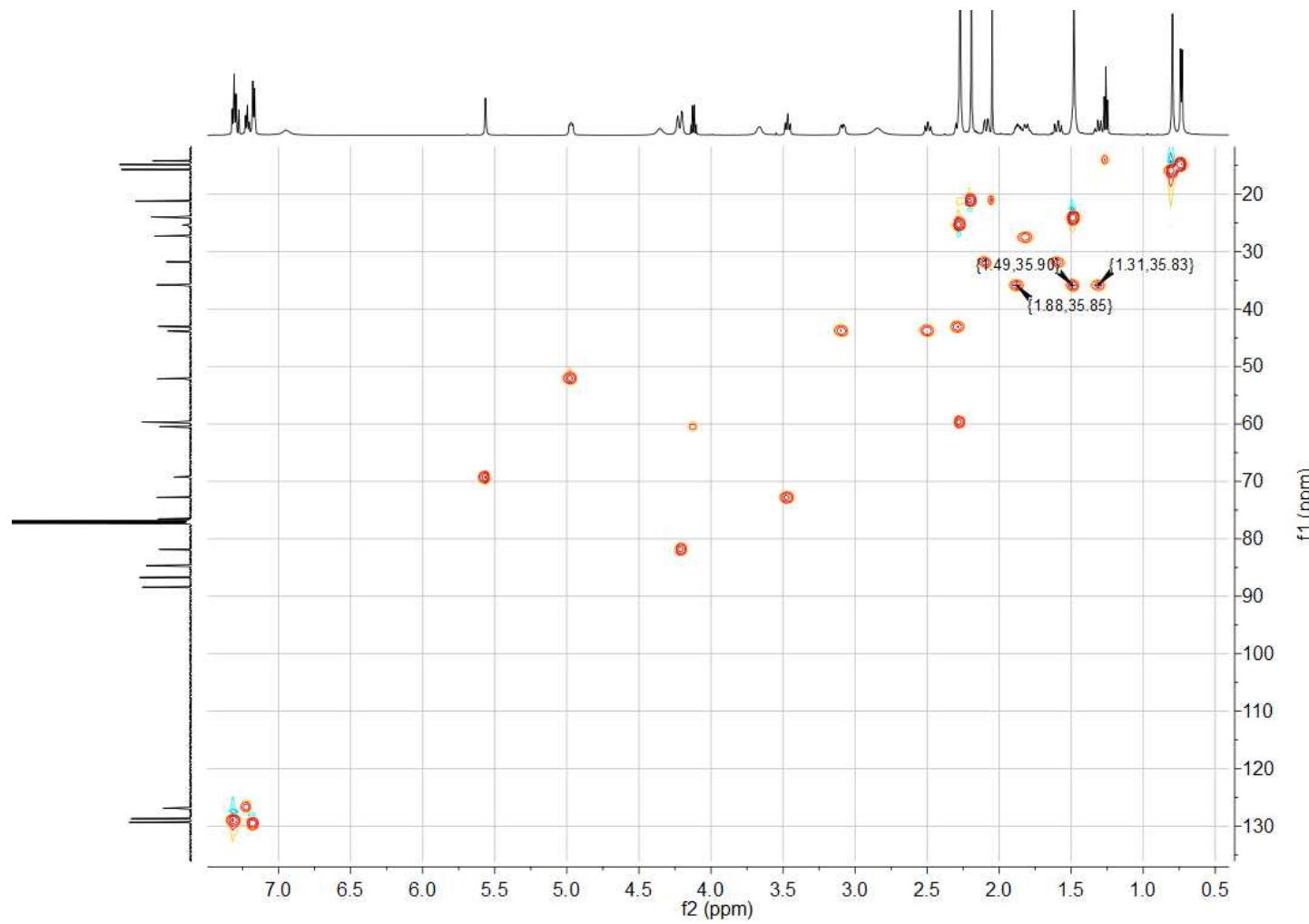
**Fig. S31.**  ${}^1\text{H}$  NMR of **4** ( $\text{CDCl}_3$ , 600 MHz).



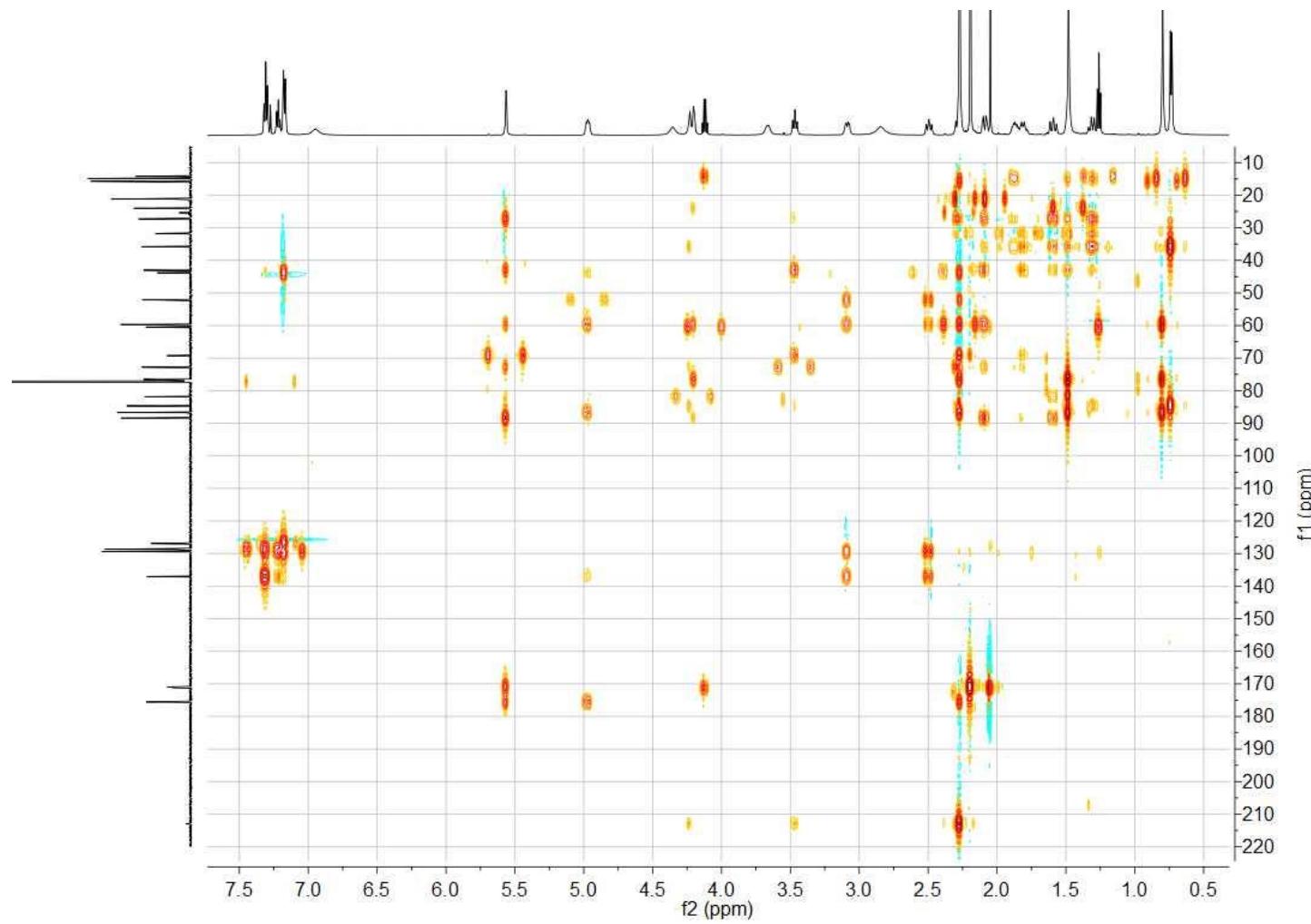
**Fig. S32.**  $^{13}\text{C}$  NMR of **4** ( $\text{CDCl}_3$ , 150 MHz).



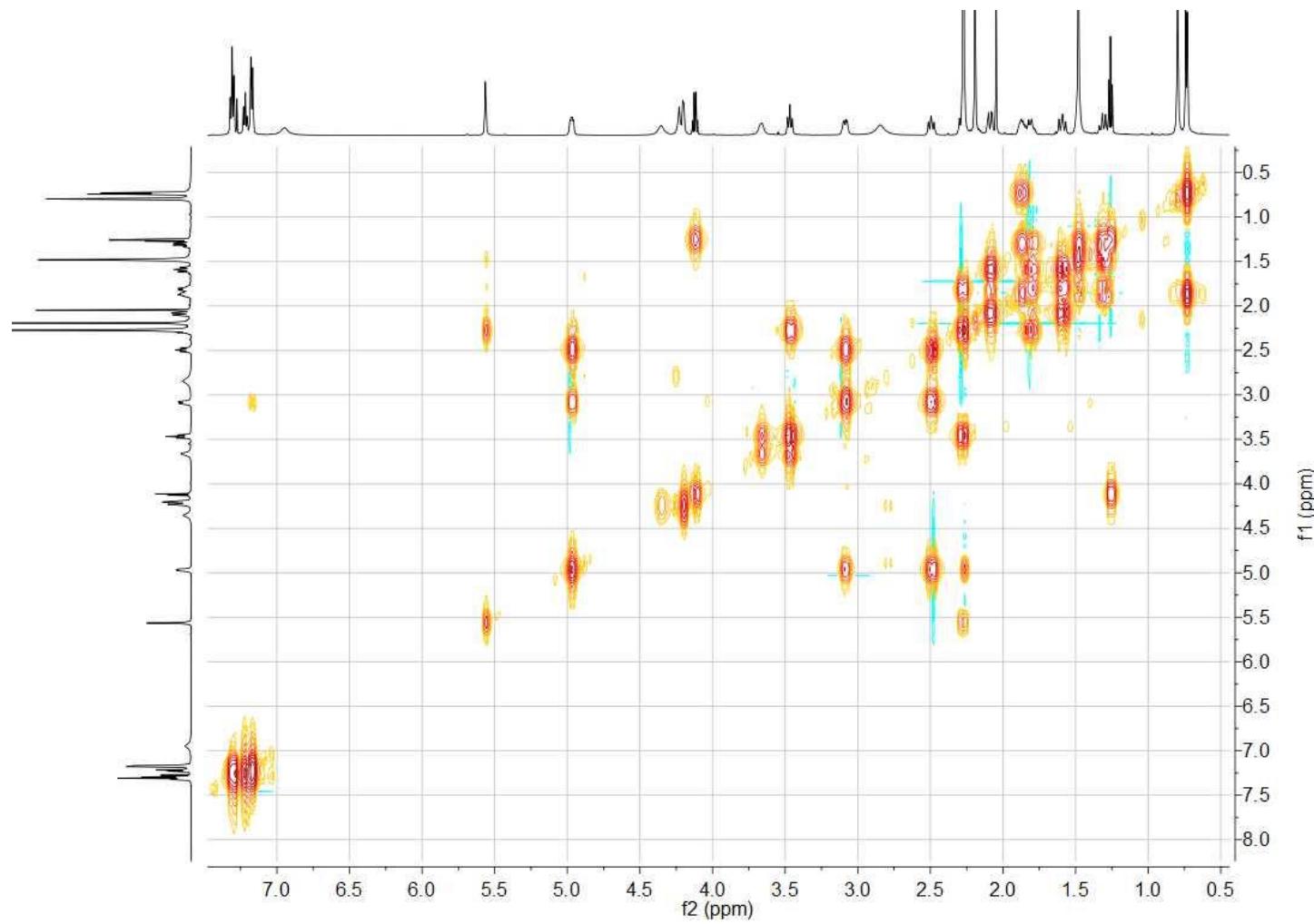
**Fig. S33.** DEPT of 4.



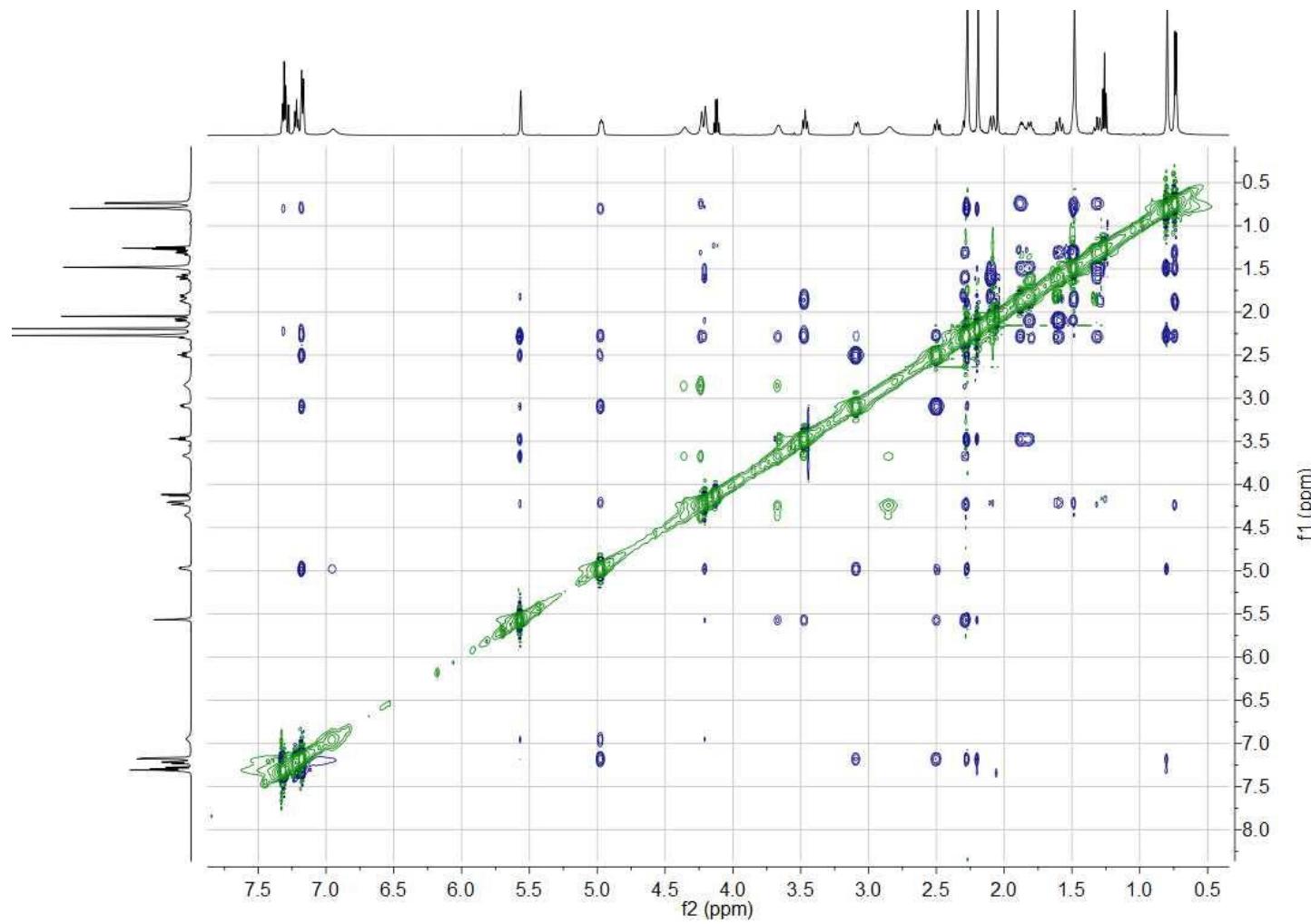
**Fig. S34.** HSQC of 4.



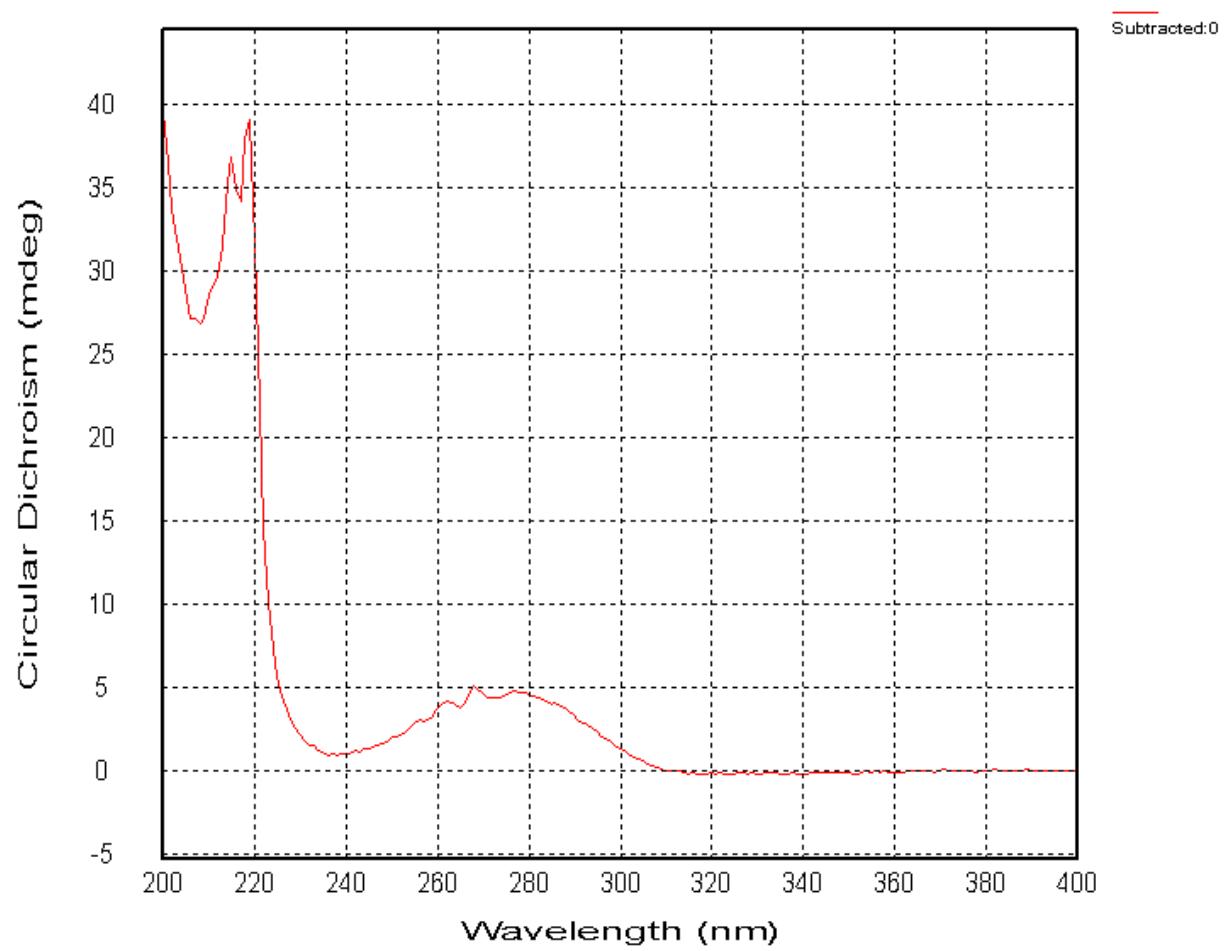
**Fig. S35.** HMBC of **4**.



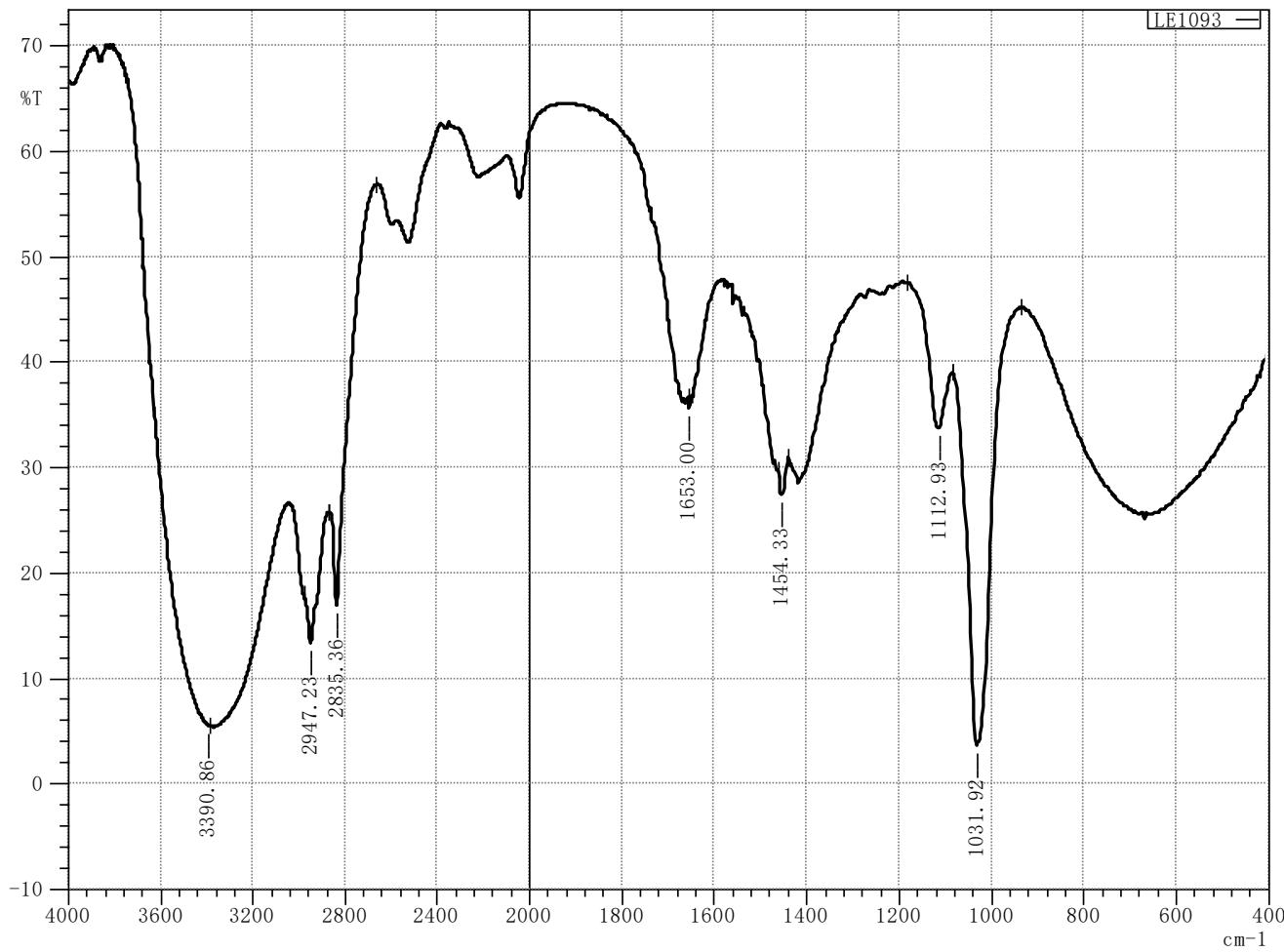
**Fig. S36.**  $^1\text{H}$ - $^1\text{H}$  COSY of 4.



**Fig. S37.** ROESY of 4.



**Fig. S38.** Experimental ECD of **4**.

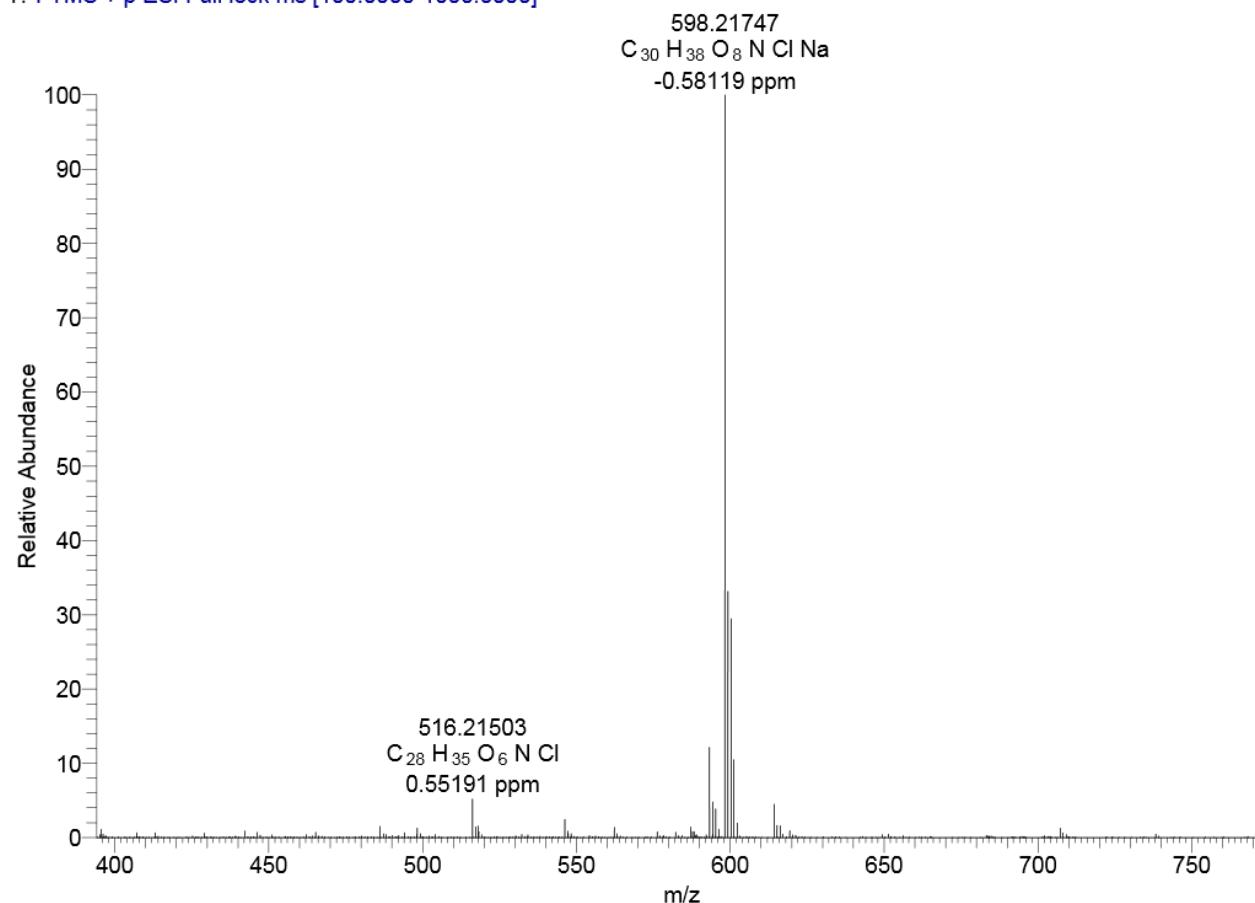


**Fig. S39.** IR of **4**.

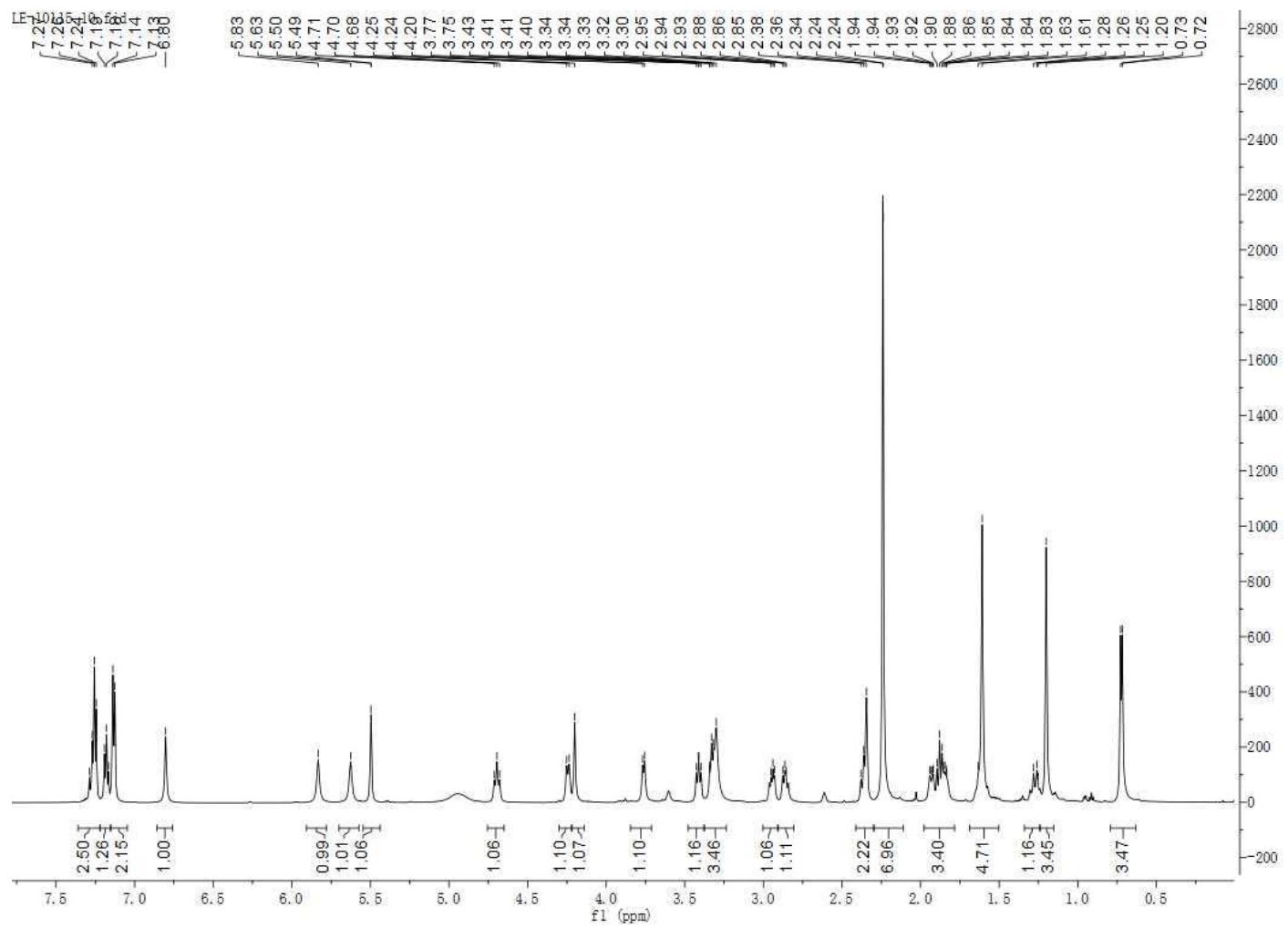
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07/02/19 20:26:48

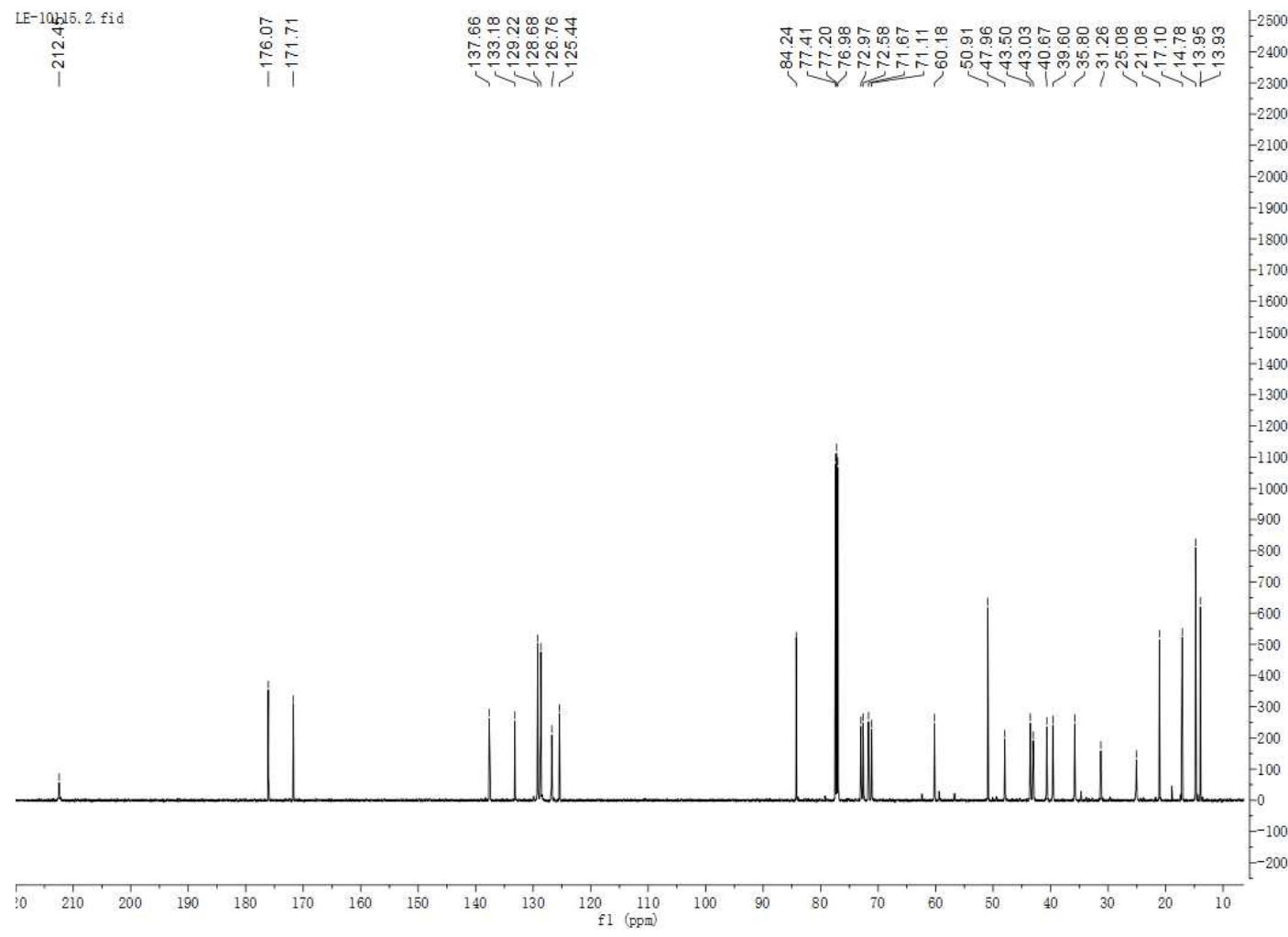
LE1093 #396 RT: 3.45 AV: 1 NL: 5.49E8  
T: FTMS + p ESI Full lock ms [100.0000-1000.0000]



**Fig. S40.** HRESIMS of **4**.

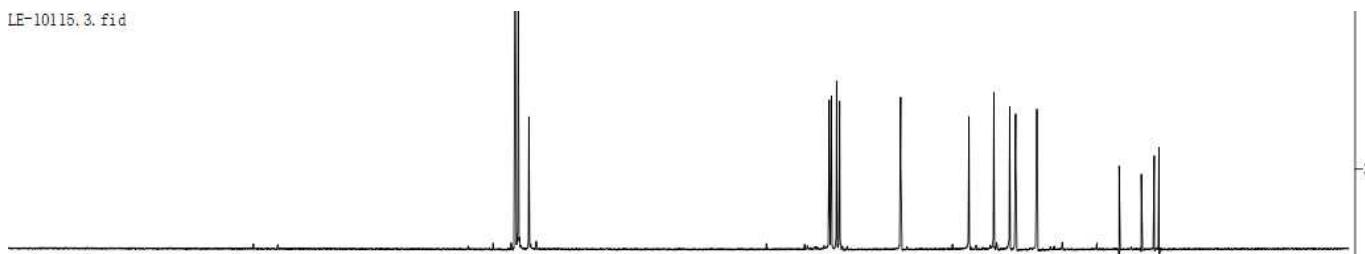


**Fig. S41.** <sup>1</sup>H NMR of **5** ( $\text{CDCl}_3$ , 600 MHz).



**Fig. S42.**  $^{13}\text{C}$  NMR of **5** ( $\text{CDCl}_3$ , 150 MHz).

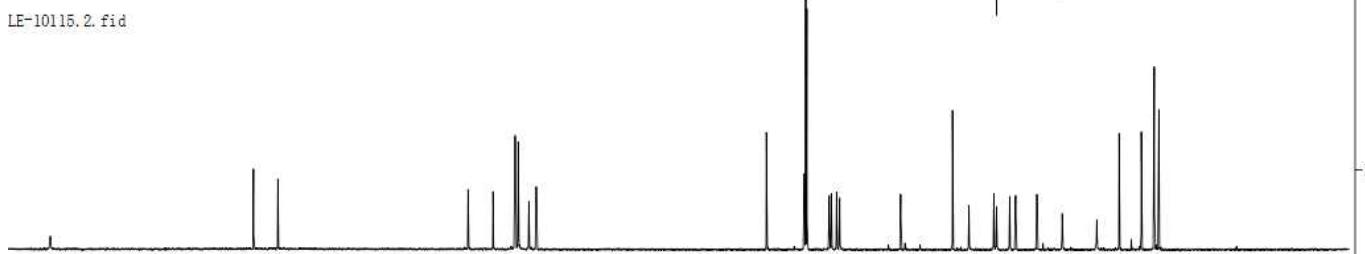
LE-10115.3.fid



LE-10115.4.fid

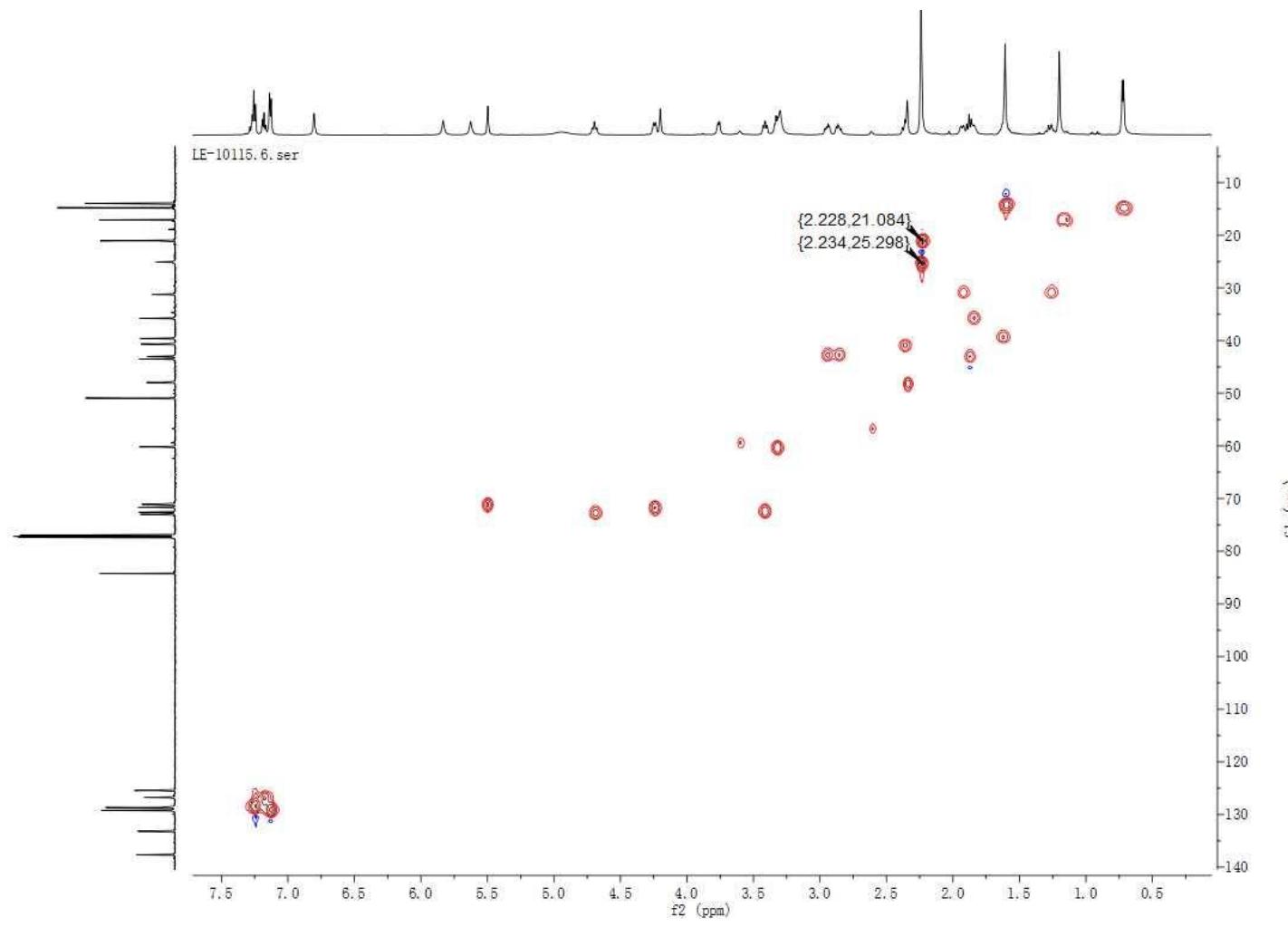


LE-10115.2.fid

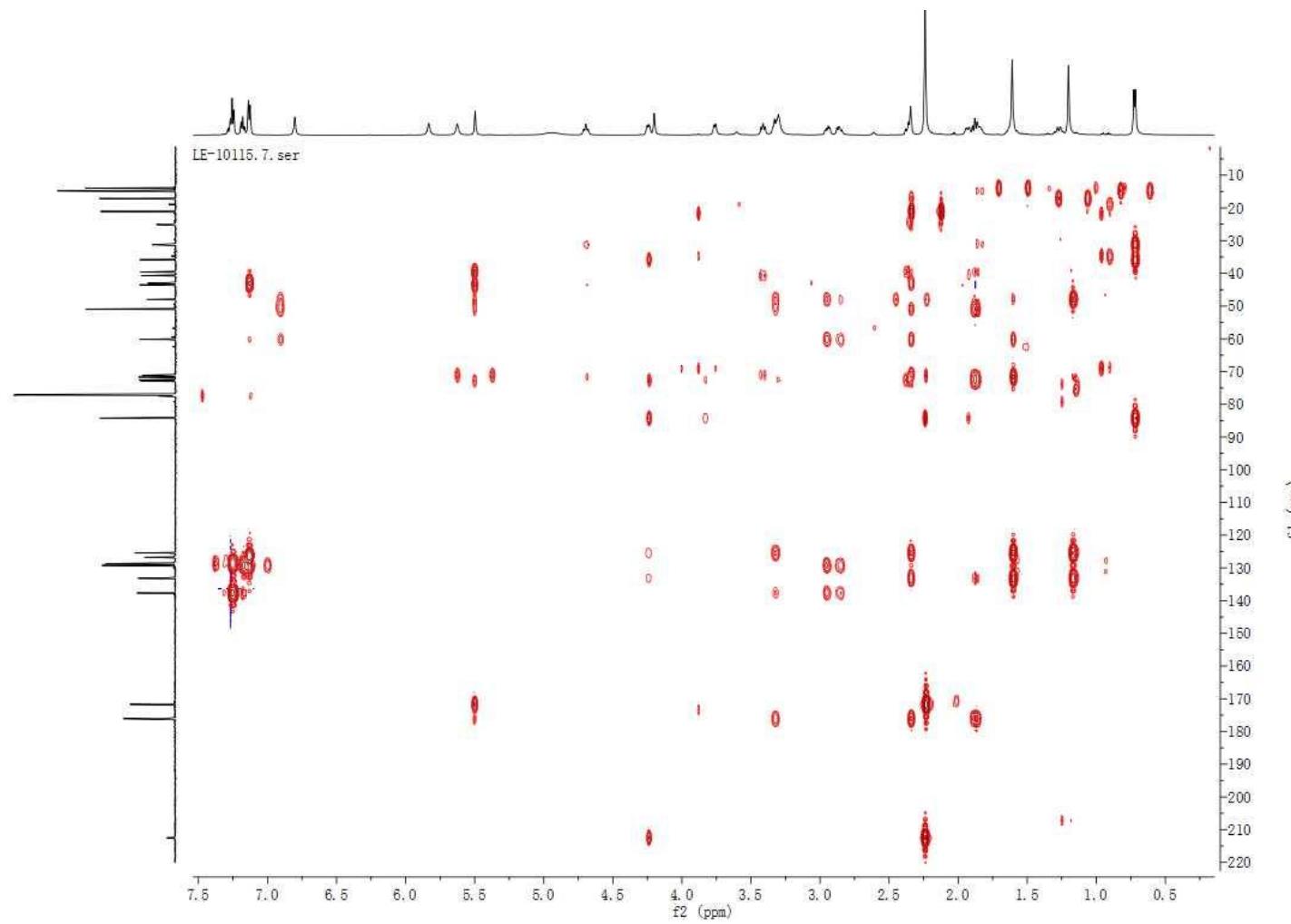


0 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20  
f1 (ppm)

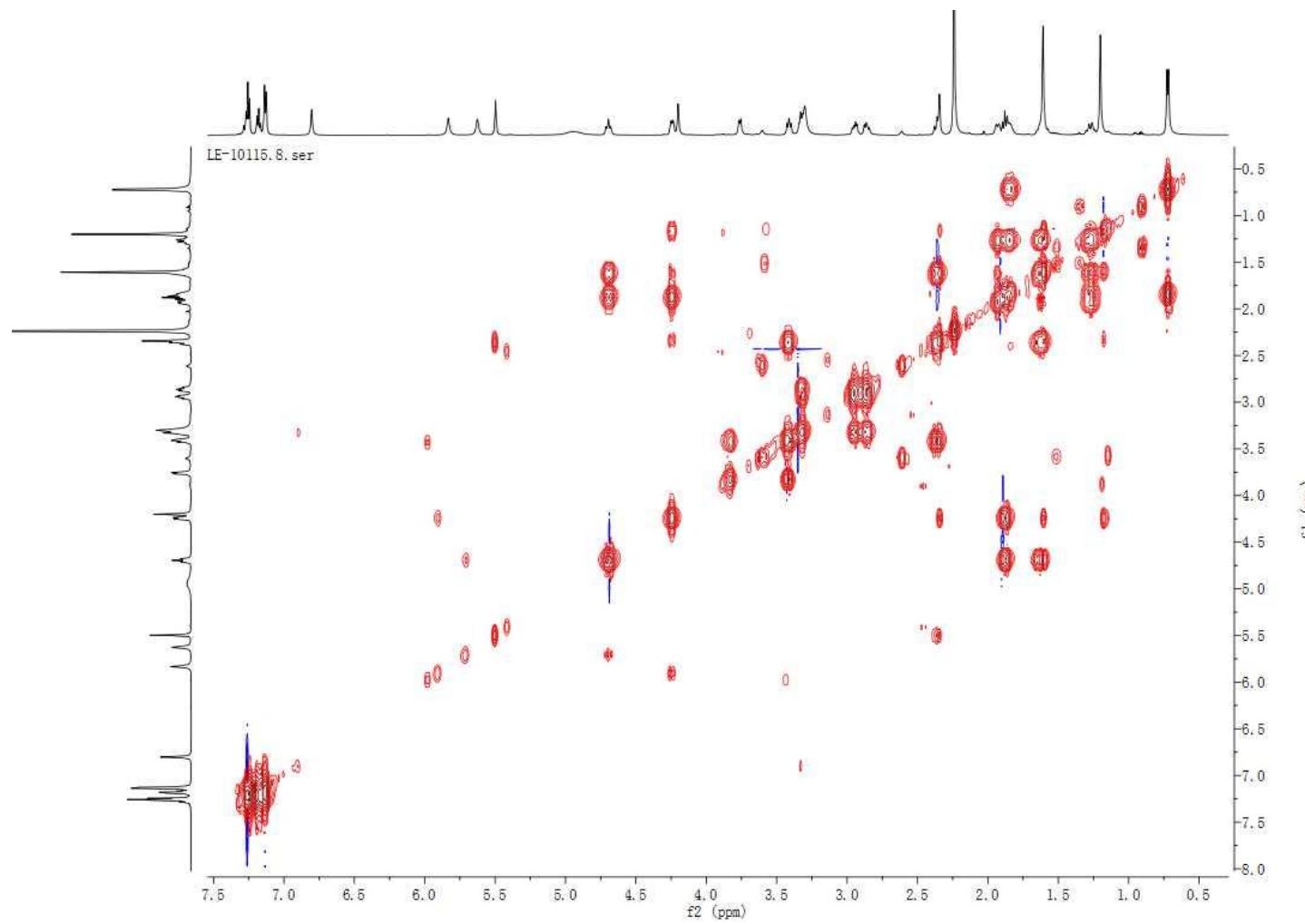
**Fig. S43.** DEPT of 5.



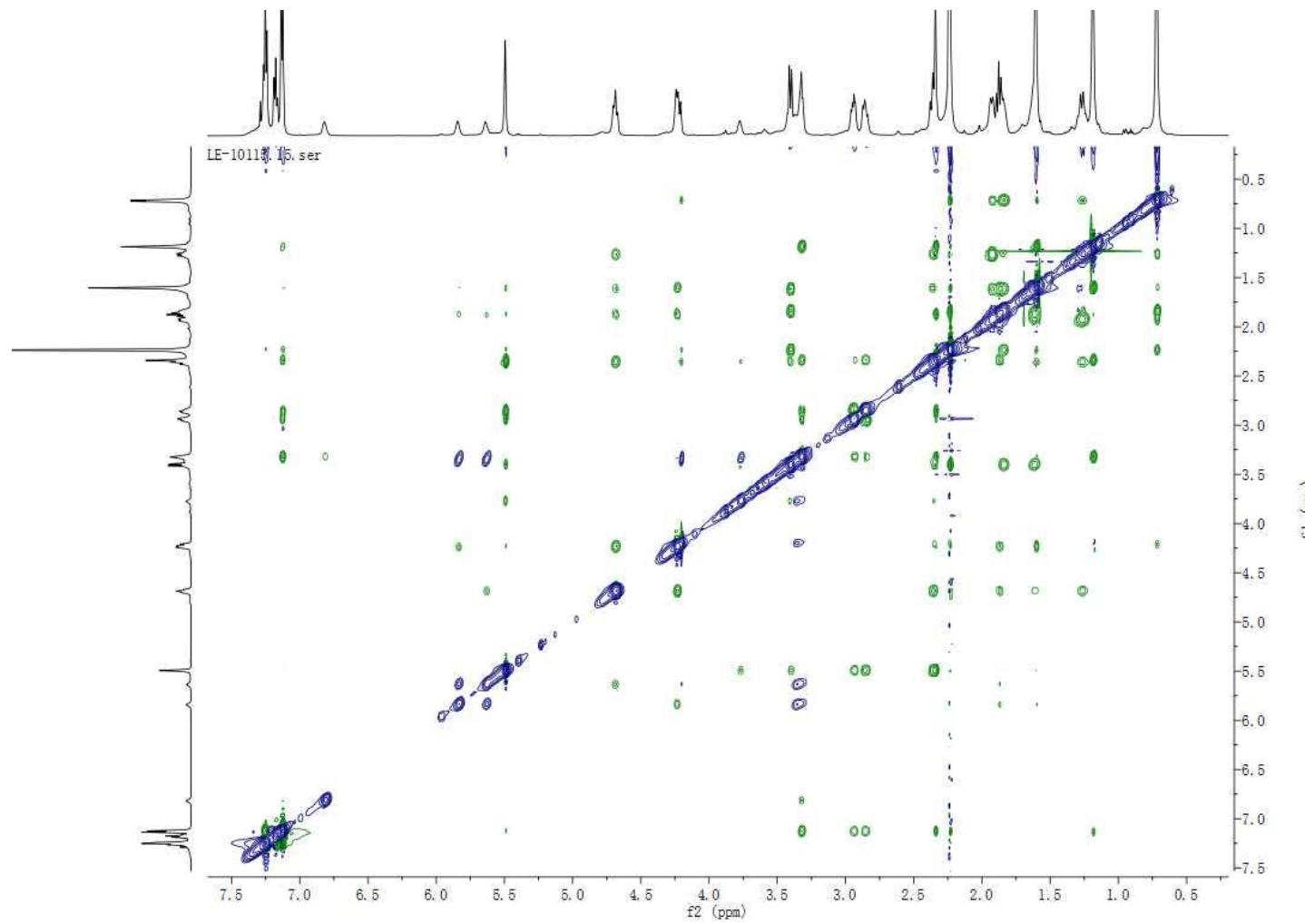
**Fig. S44.** HSQC of 5.



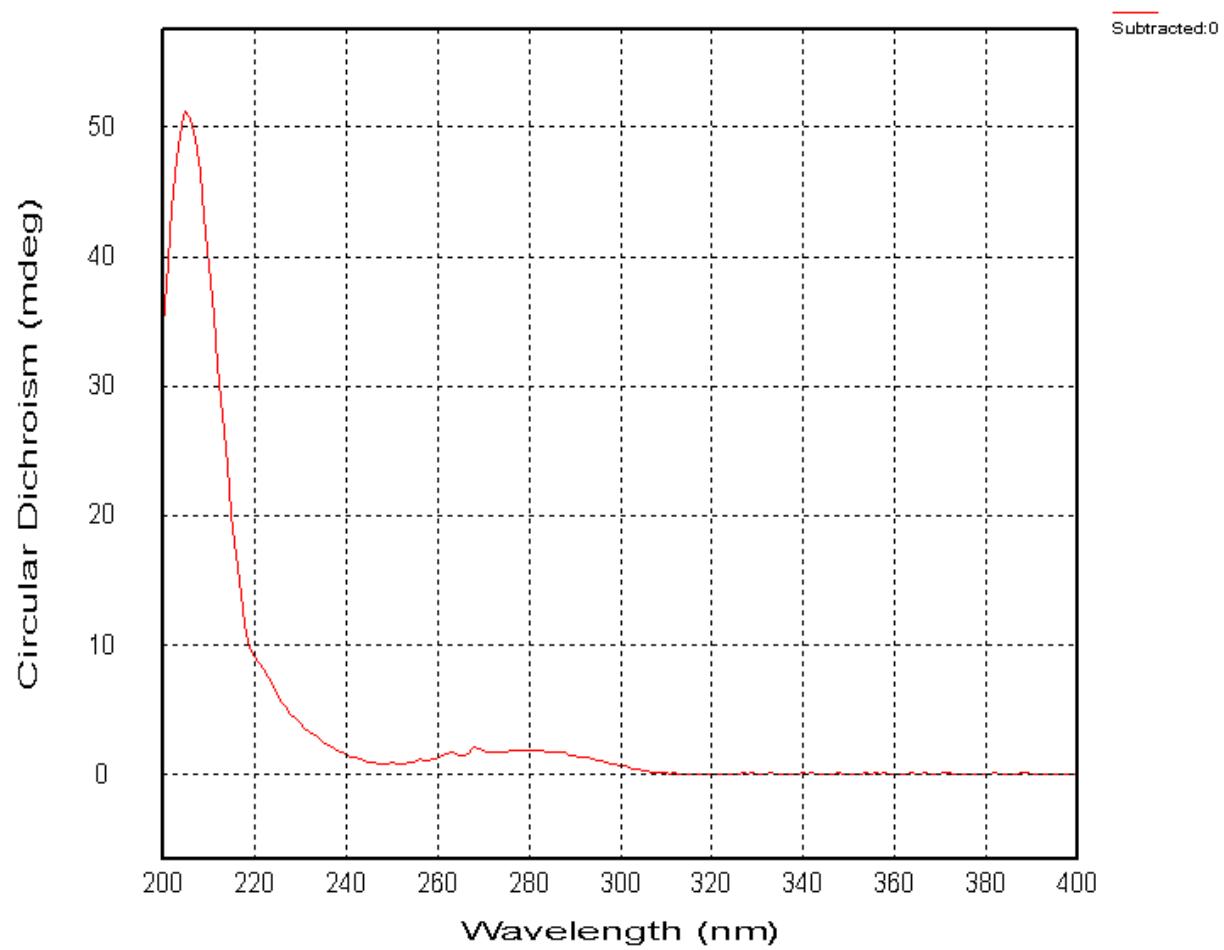
**Fig. S45.** HMBC of **5**.



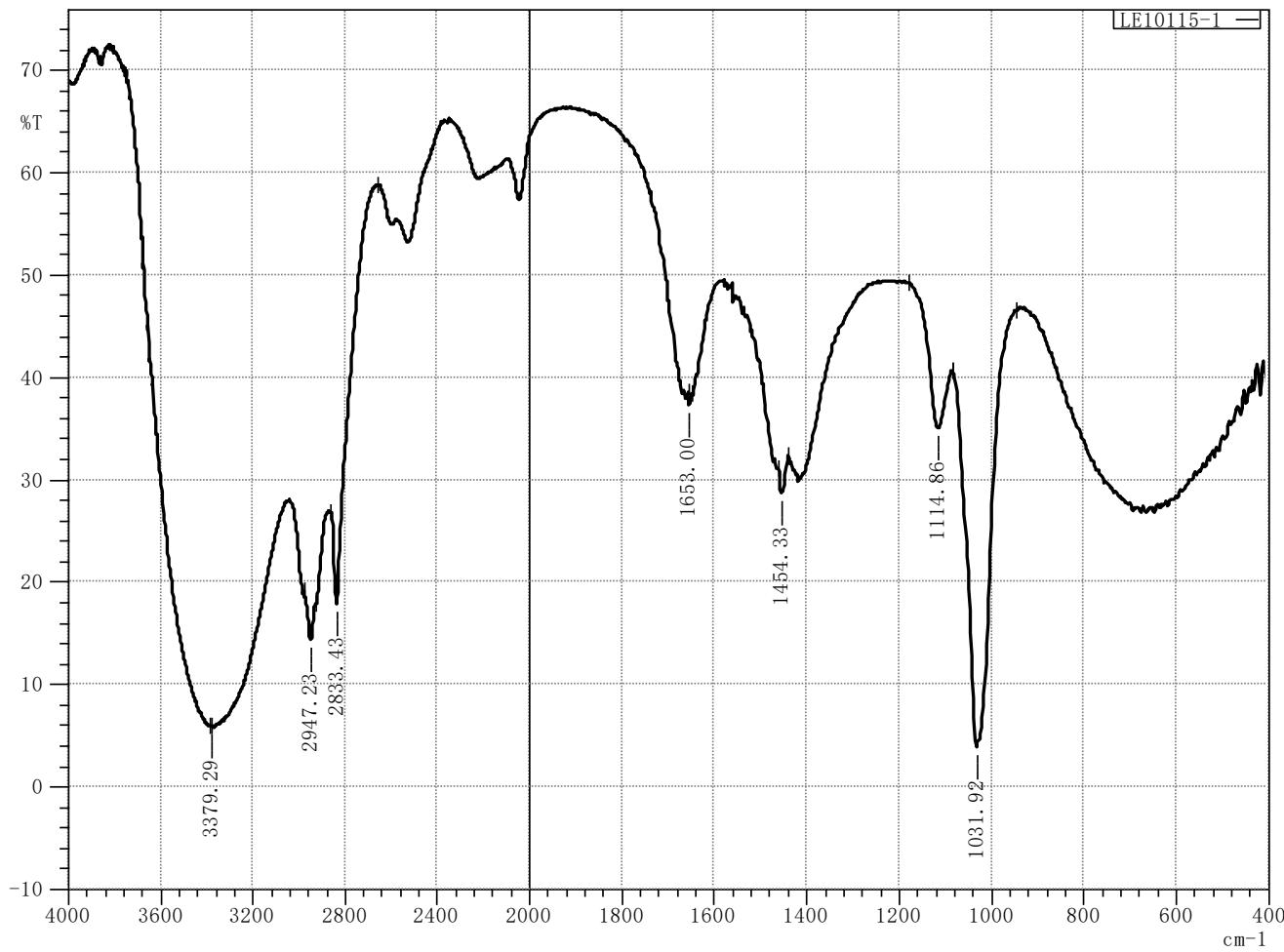
**Fig. S46.**  $^1\text{H}$ - $^1\text{H}$  COSY of 5.



**Fig. S47.** ROESY of 5.



**Fig. S48.** Experimental ECD of **5**.

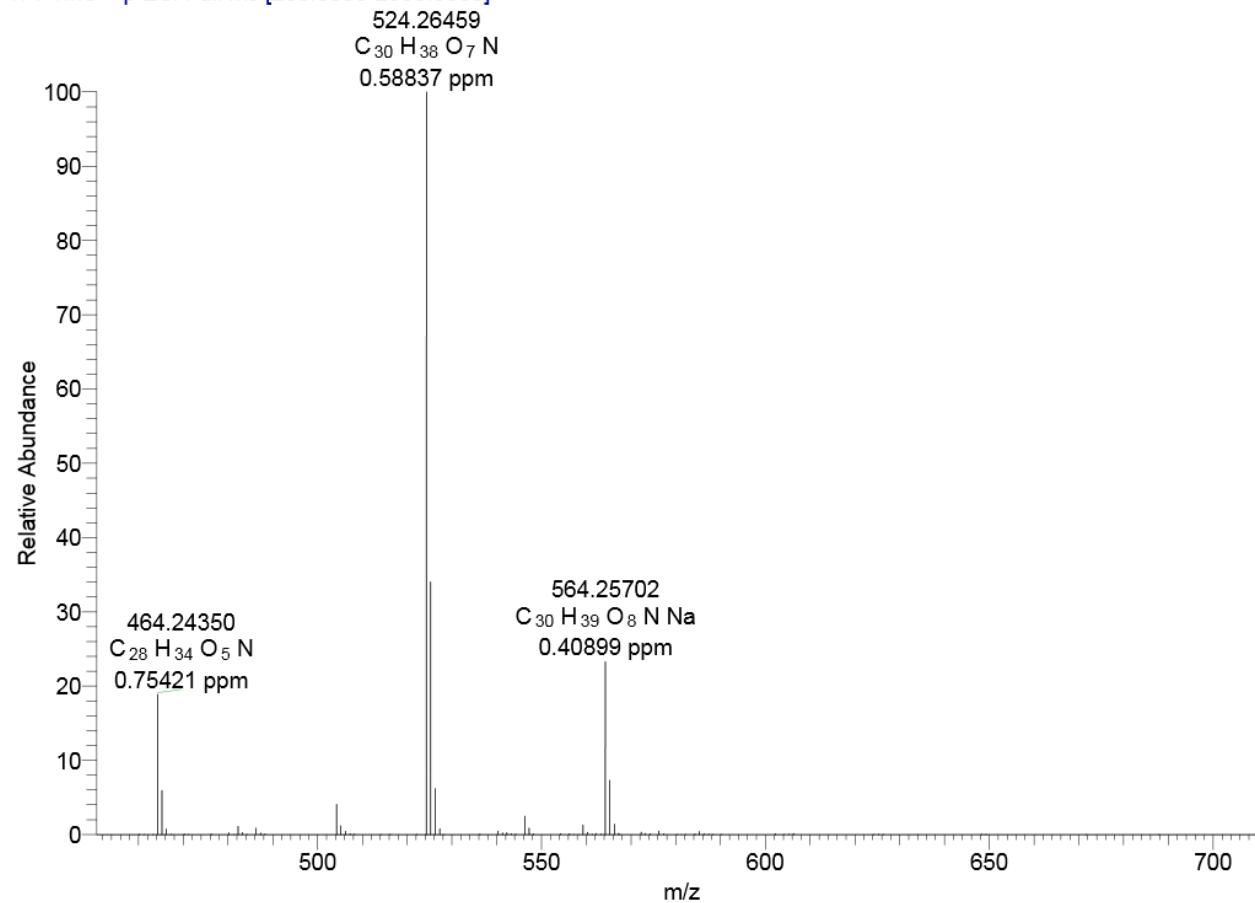


**Fig. S49.** IR of 5.

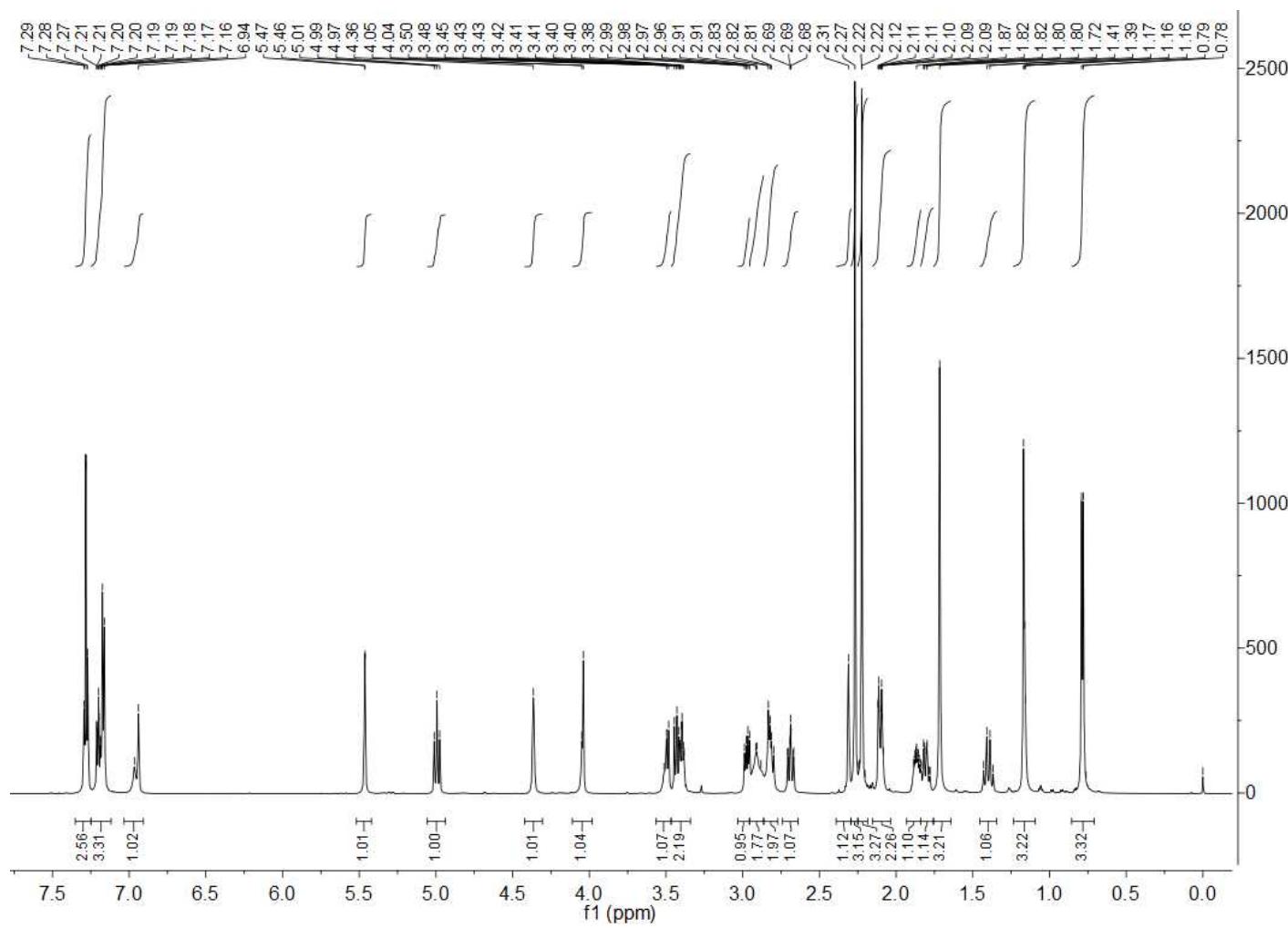
D:\Wen-Xuan\2019\E106R\20190320\LE10-115

03/20/19 17:51:08

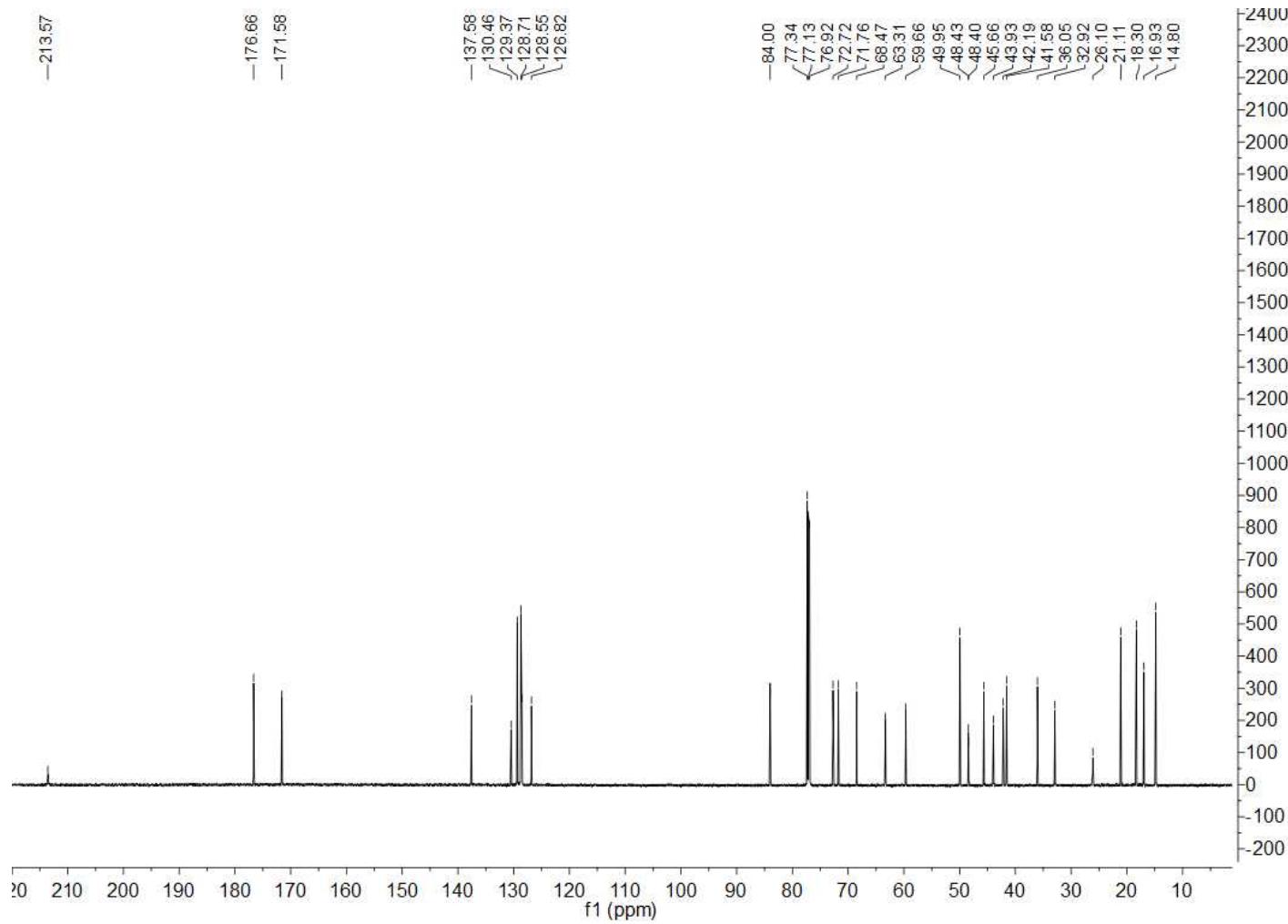
LE10-115 #1586 RT: 13.83 AV: 1 NL: 3.18E9  
T: FTMS + p ESI Full ms [250.0000-2000.0000]



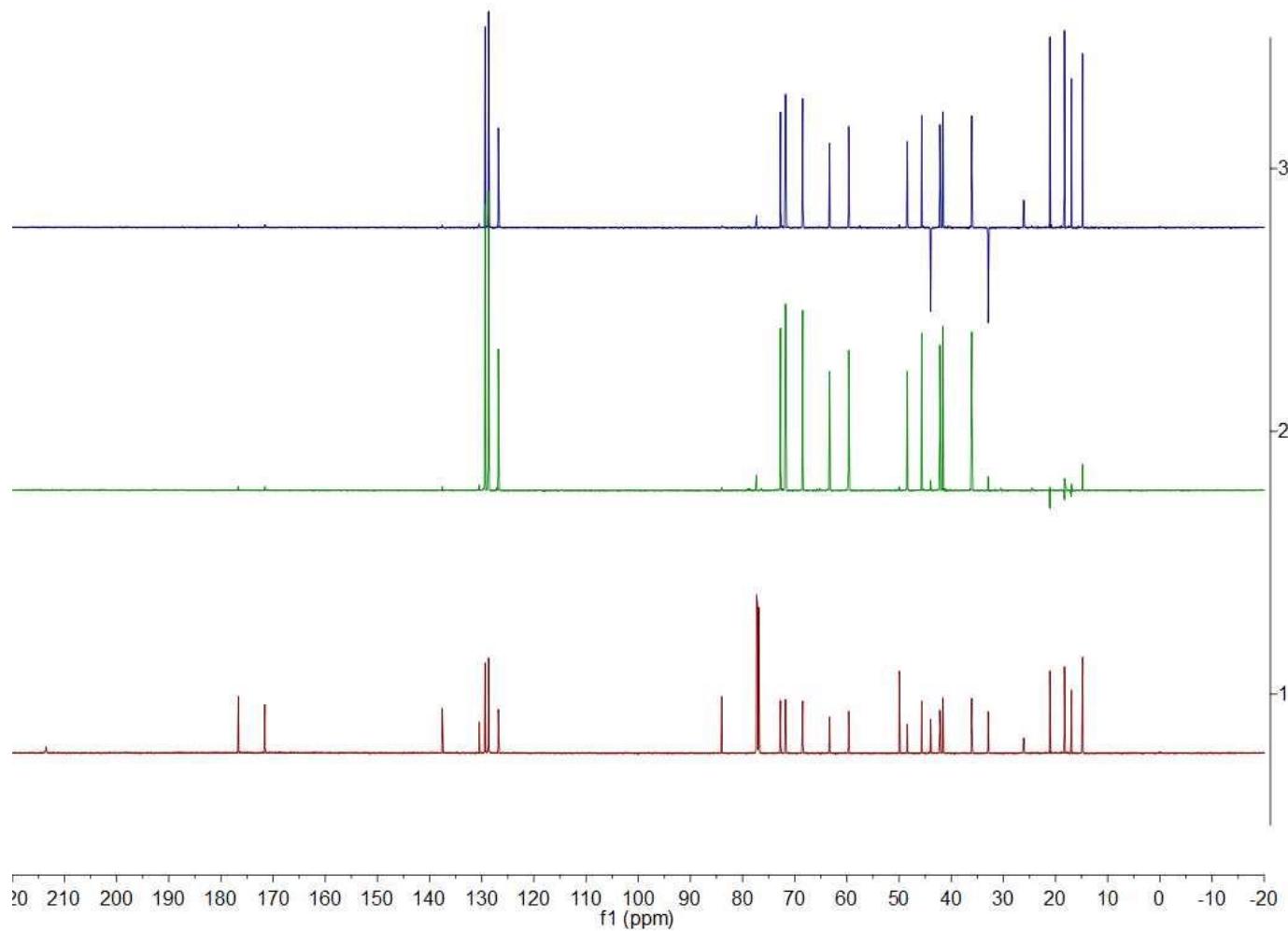
**Fig. S50.** HRESIMS of **5**.



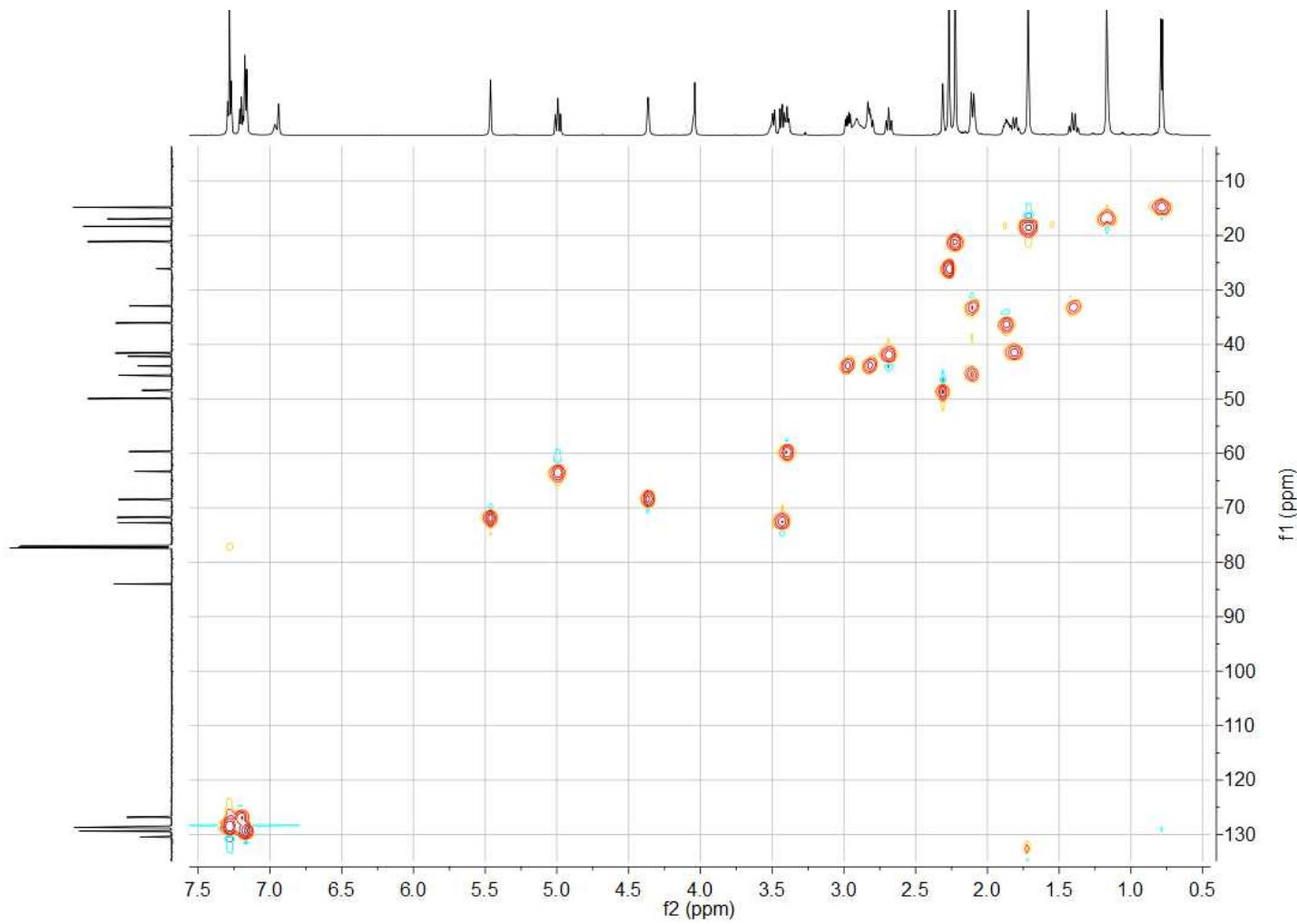
**Fig. S51.**  $^1\text{H}$  NMR of **6** ( $\text{CDCl}_3$ , 600 MHz).



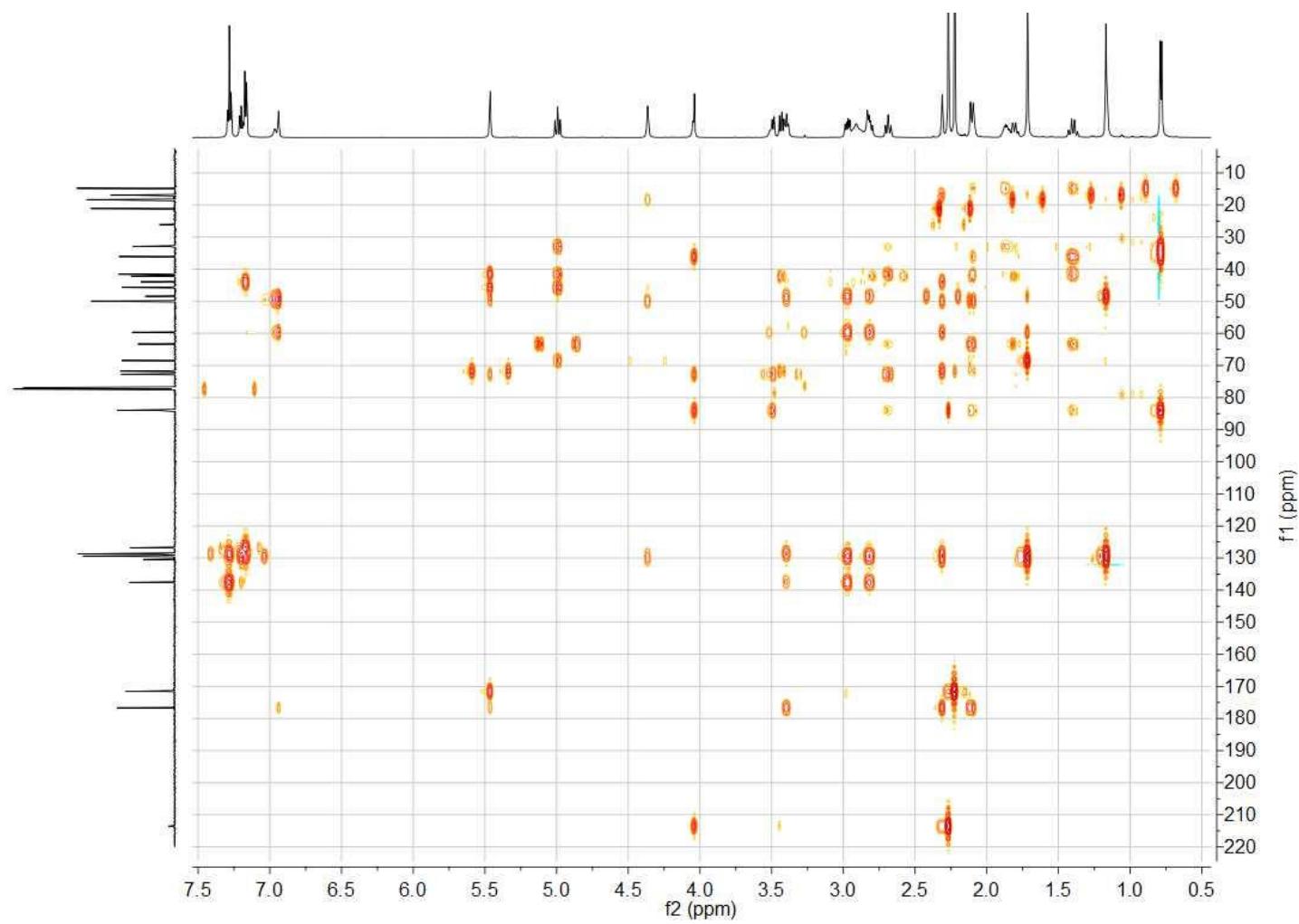
**Fig. S52.**  $^{13}\text{C}$  NMR of **6** ( $\text{CDCl}_3$ , 150 MHz).



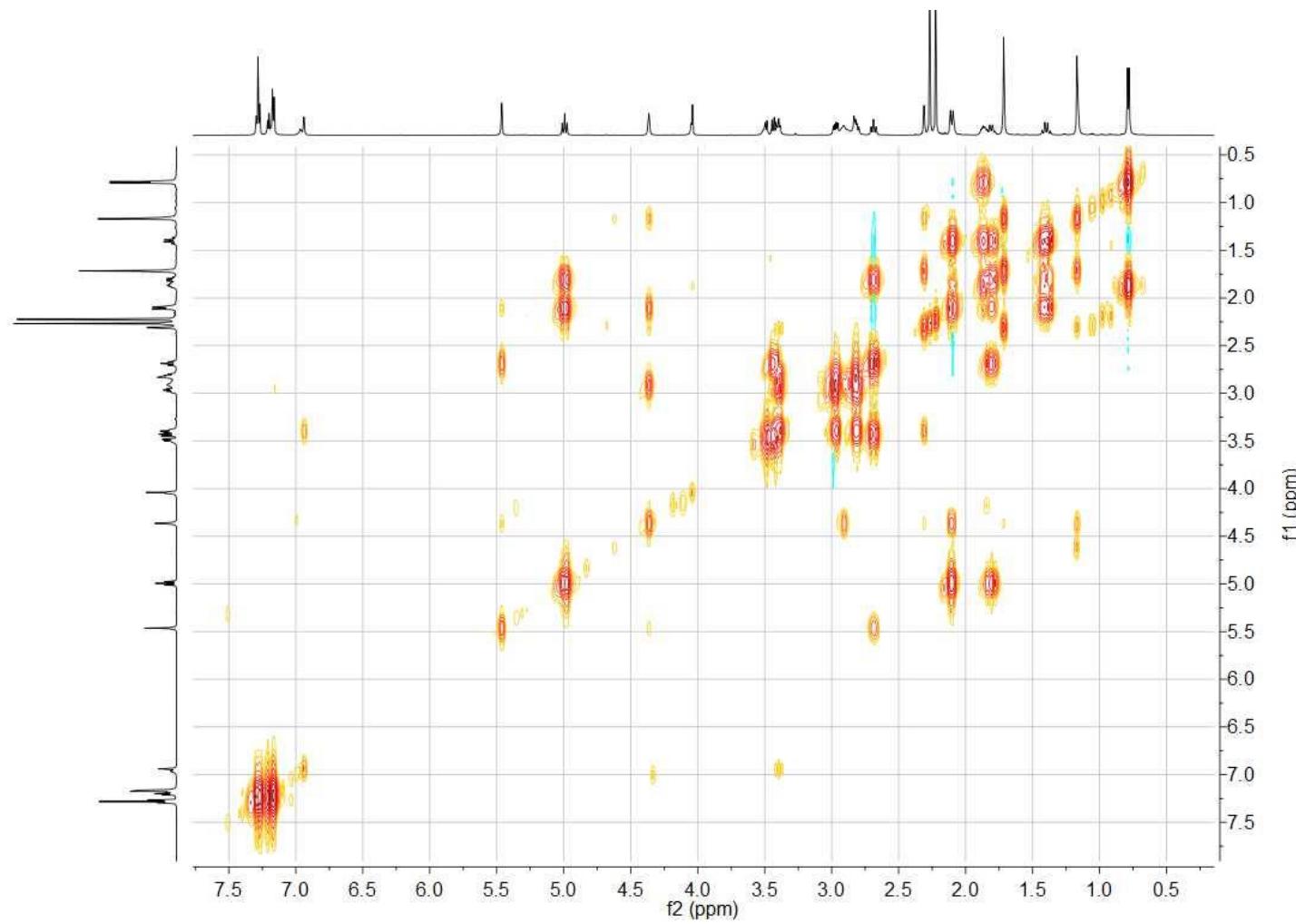
**Fig. S53.** DEPT of 6.



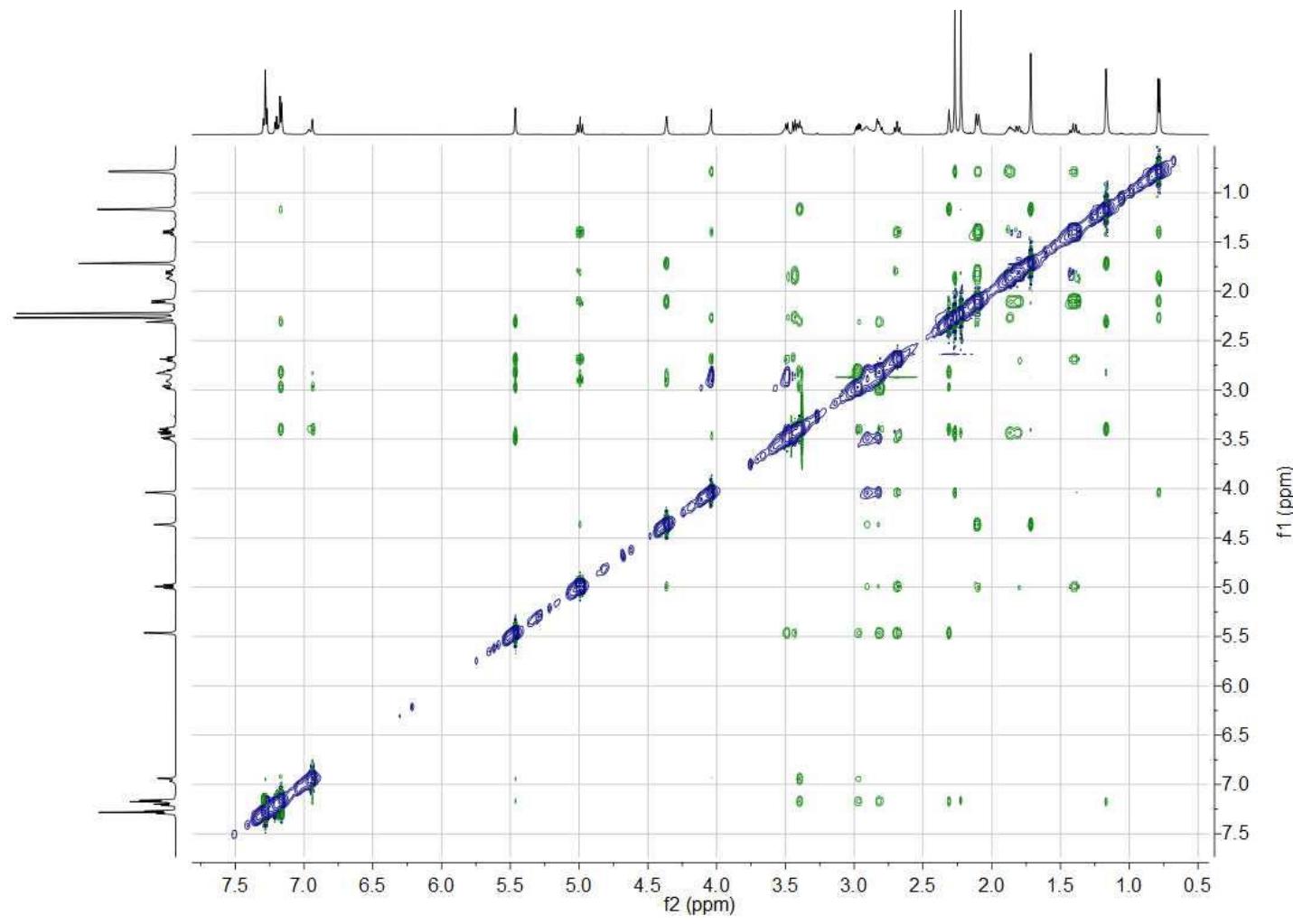
**Fig. S54.** HSQC of **6**.



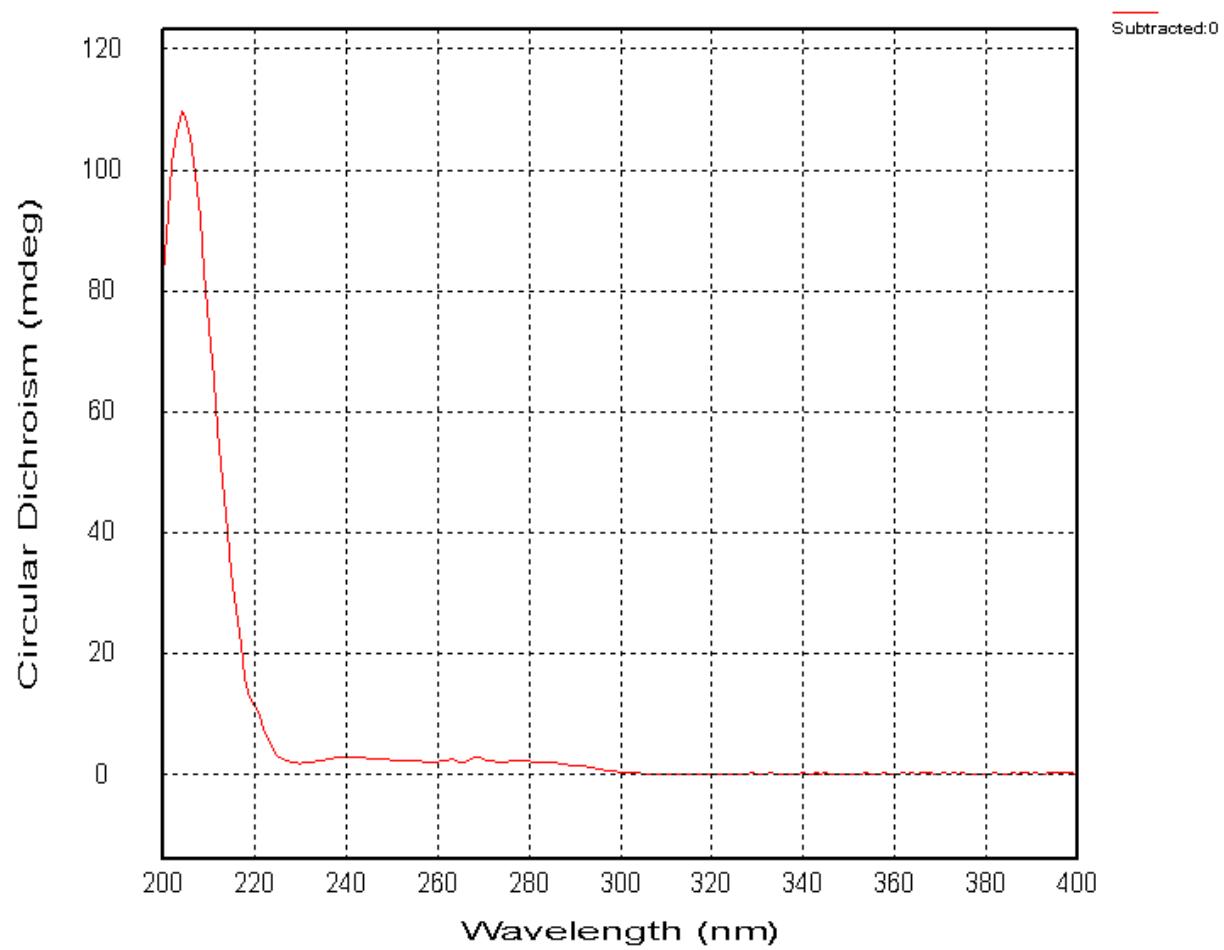
**Fig. S55.** HMBC of **6**.



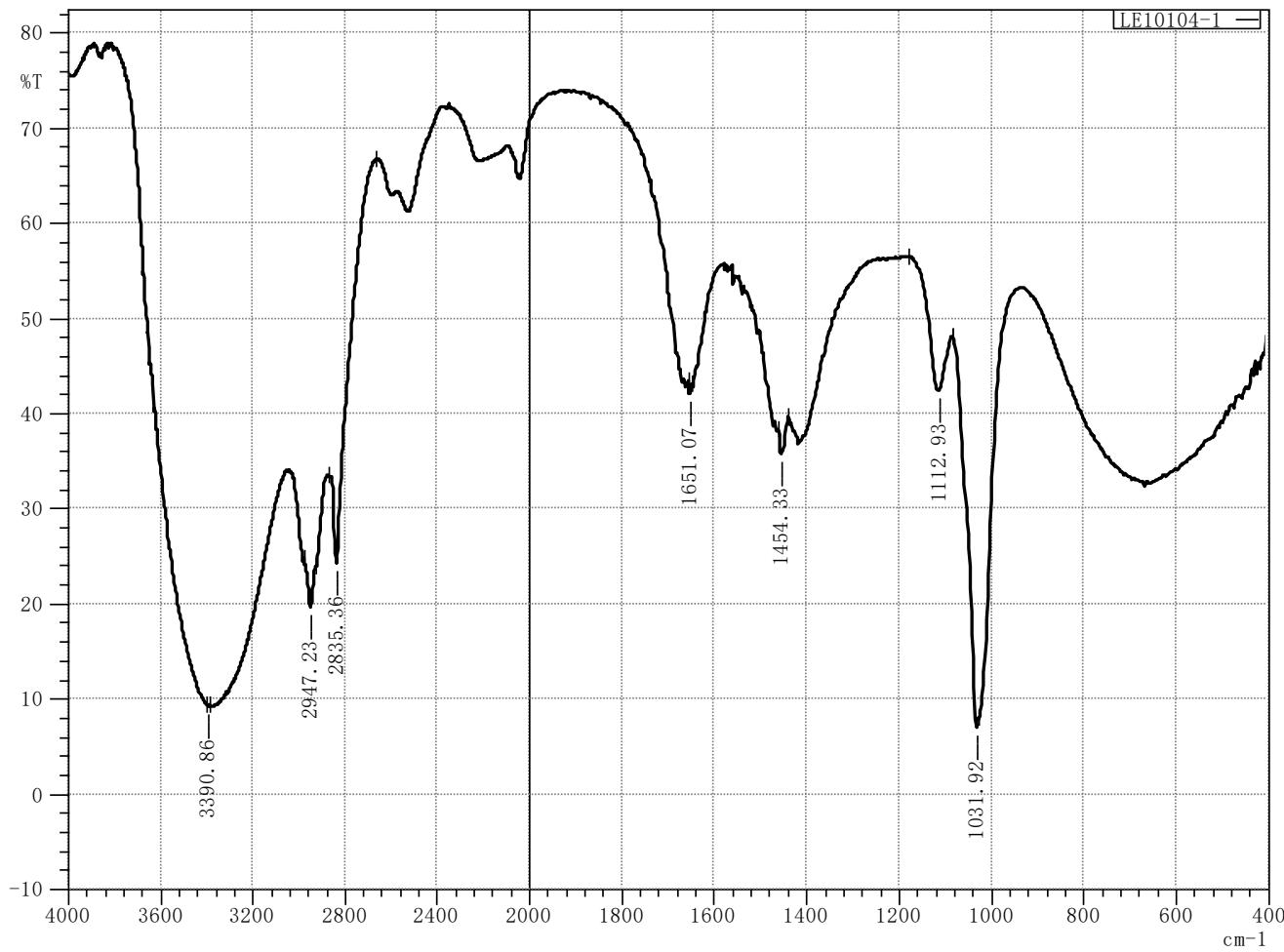
**Fig. S56.**  $^1\text{H}$ - $^1\text{H}$  COSY of 6.



**Fig. S57.** ROESY of **6**.



**Fig. S58.** Experimental ECD of **6**.



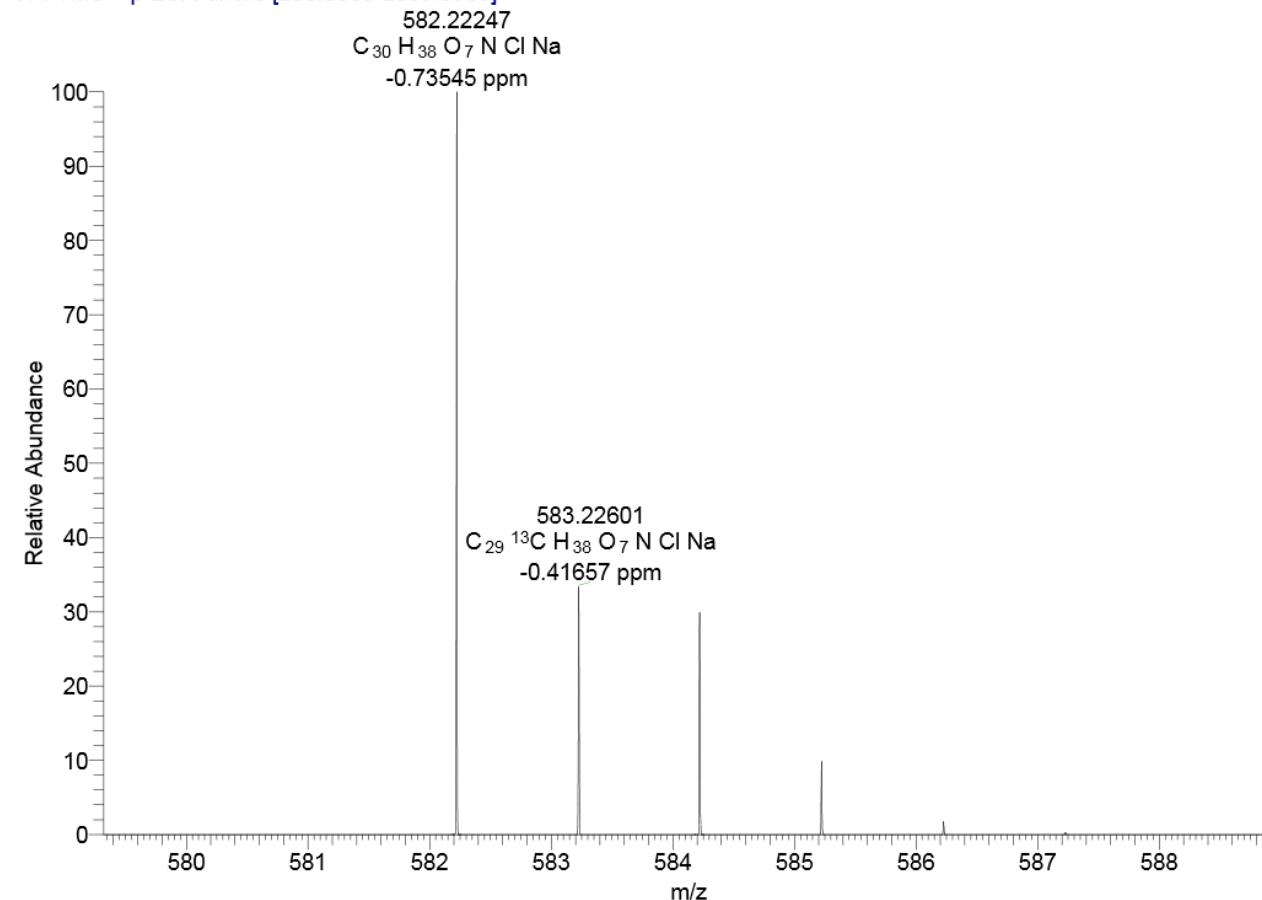
**Fig. S59.** IR of **6**.

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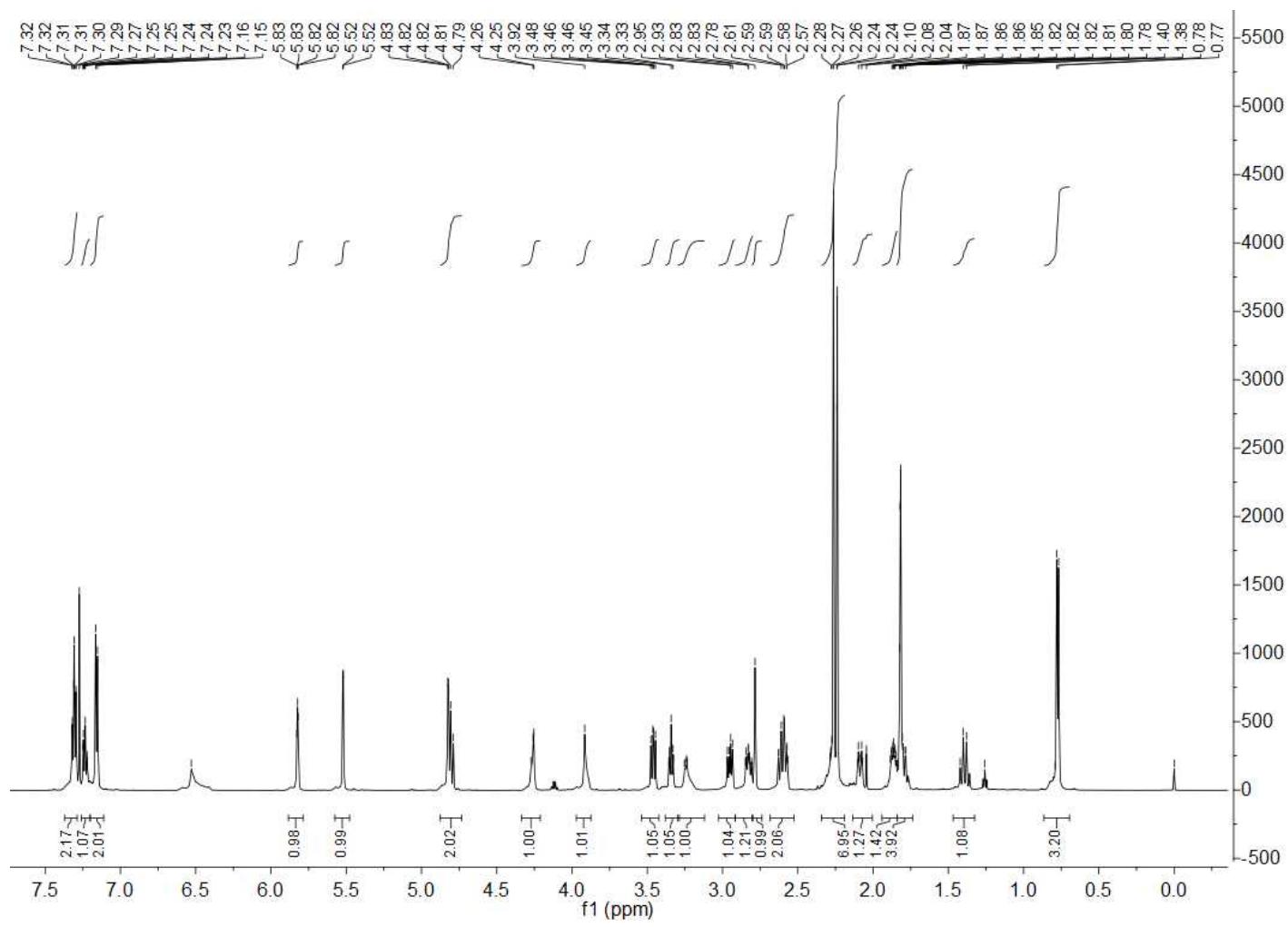
08/24/18 17:09:38

E106R324 #992 RT: 13.82 AV: 1 NL: 1.93E8

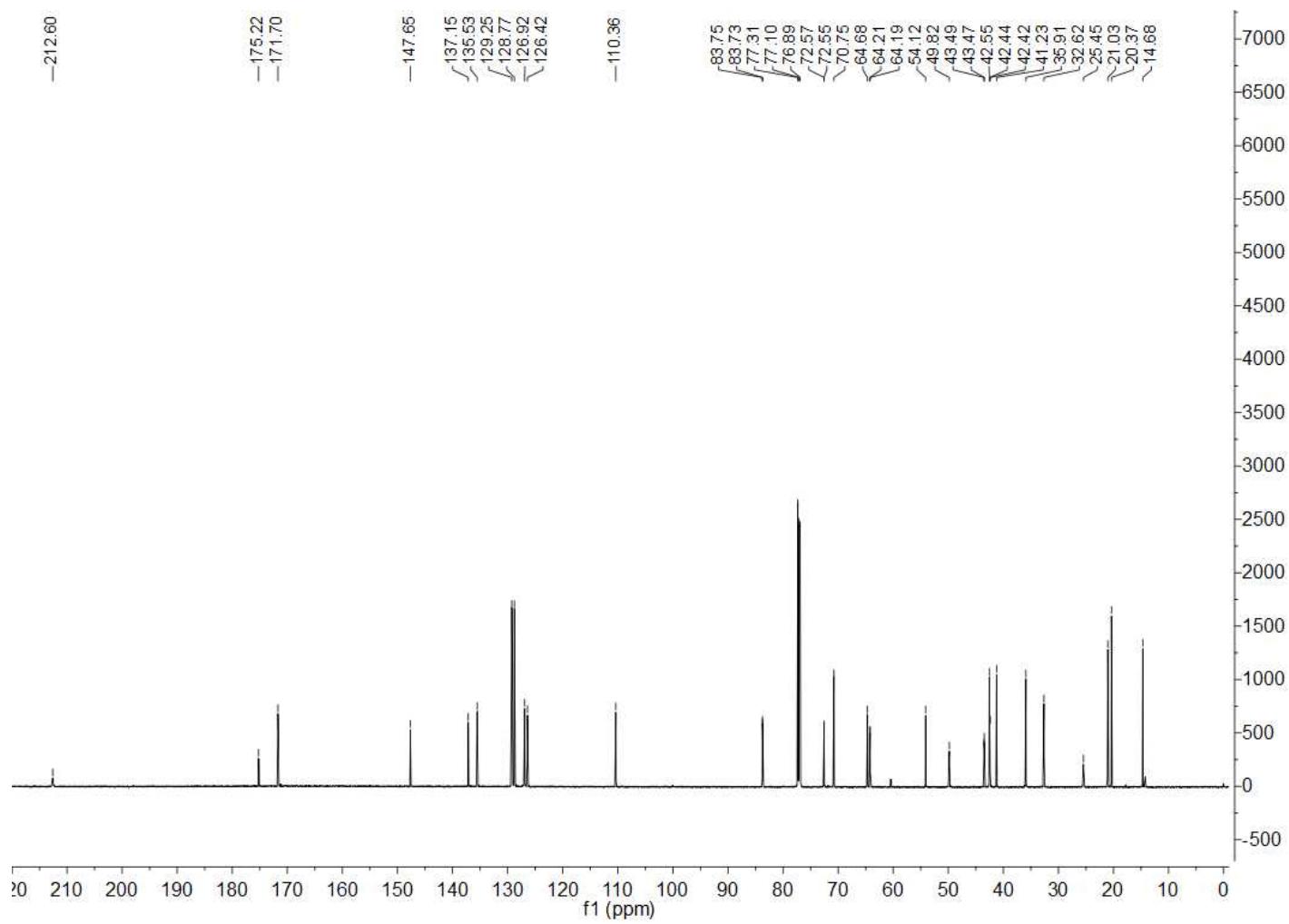
T: FTMS + p ESI Full ms [200.0000-2000.0000]



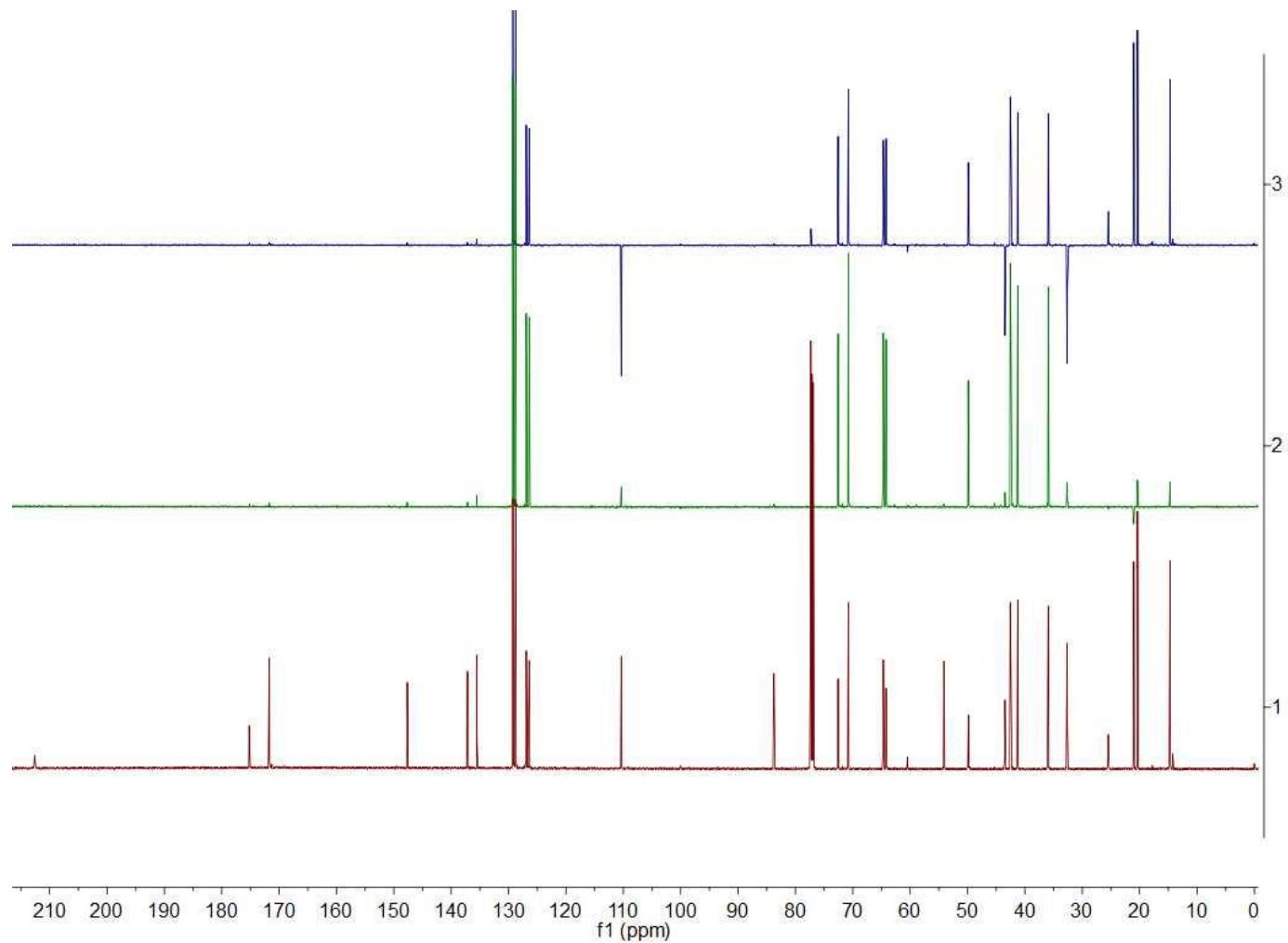
**Fig. S60.** HRESIMS of **6**.



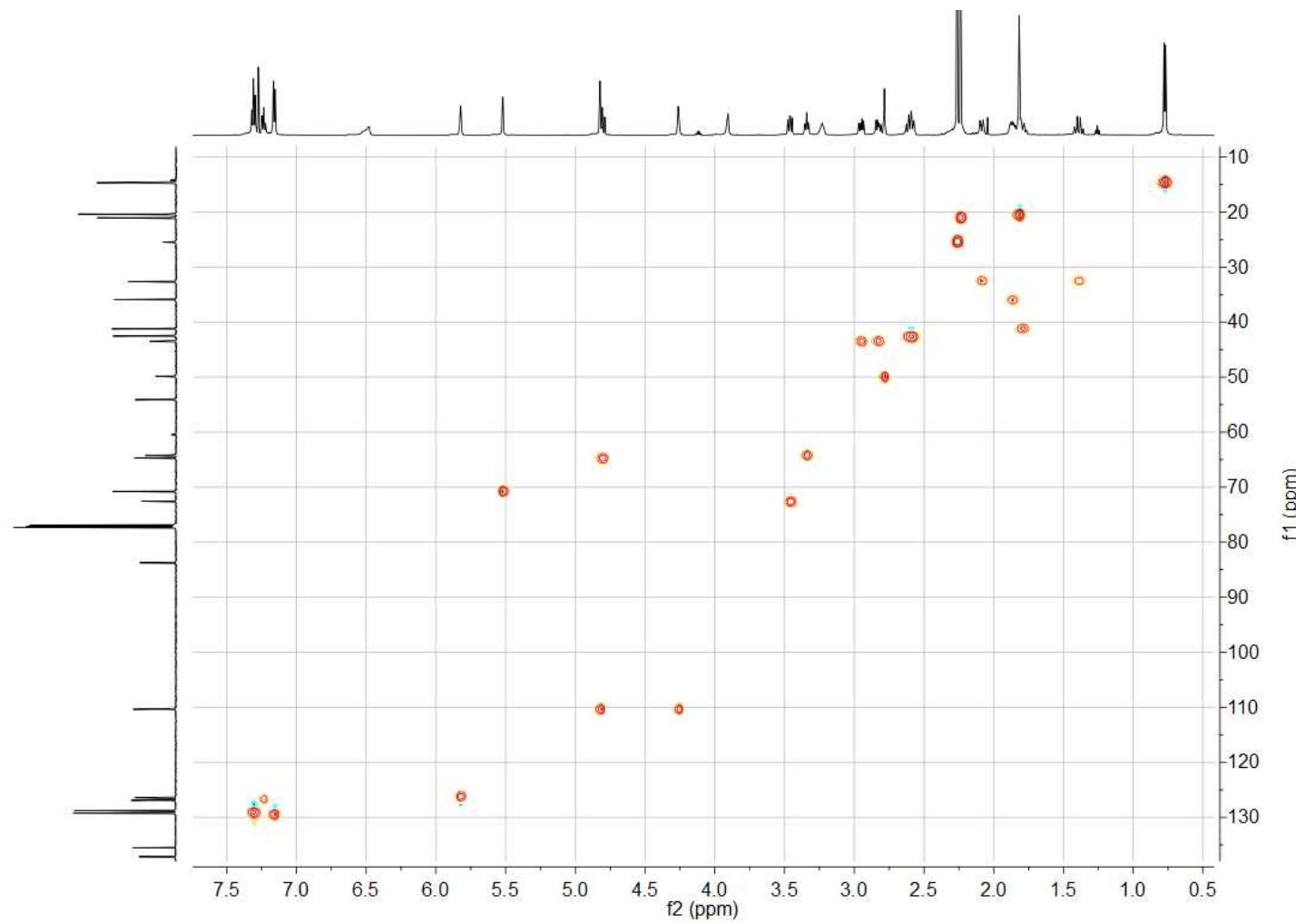
**Fig. S61.** <sup>1</sup>H NMR of 7.



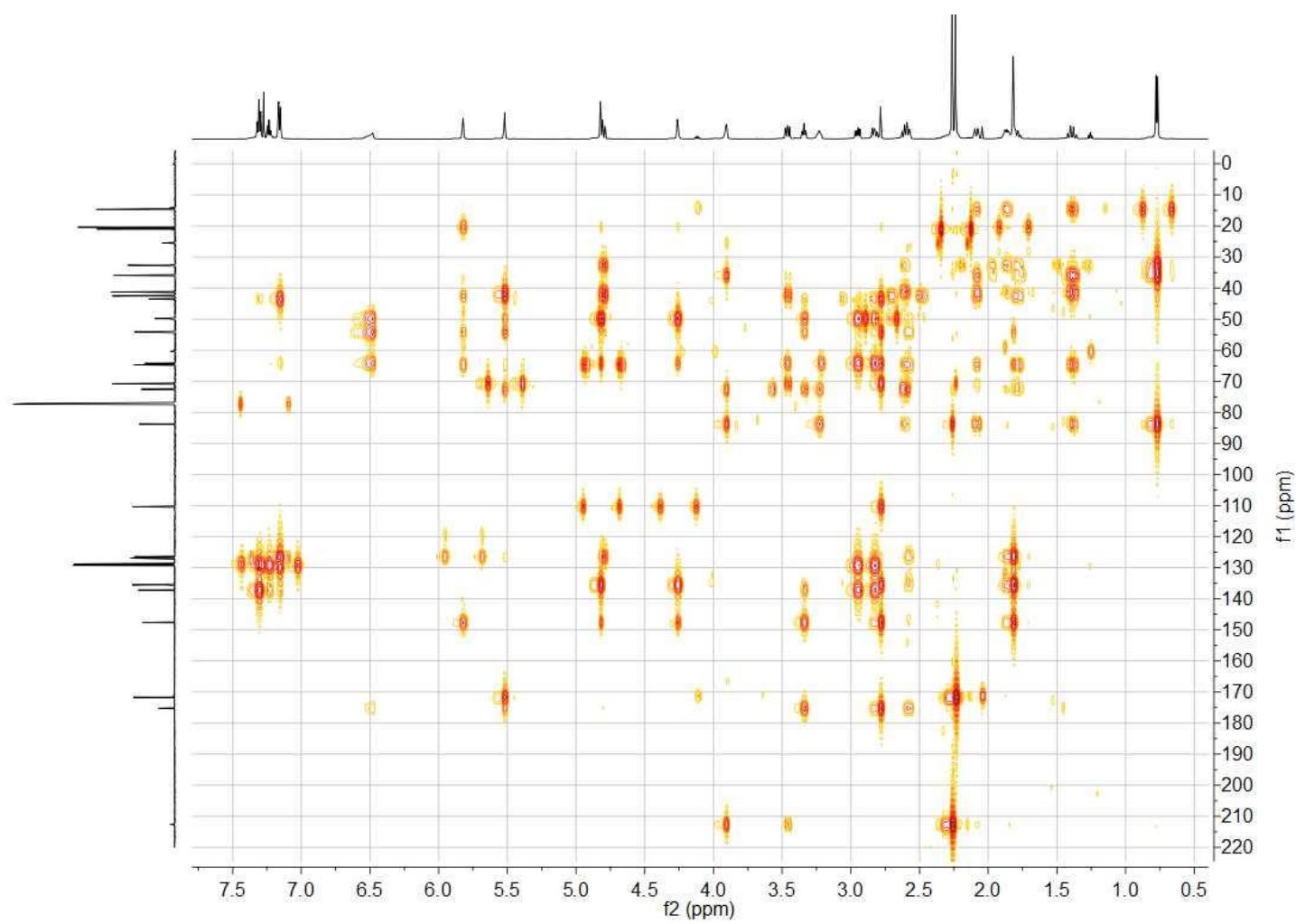
**Fig. S62.**  $^{13}\text{C}$  NMR of 7.



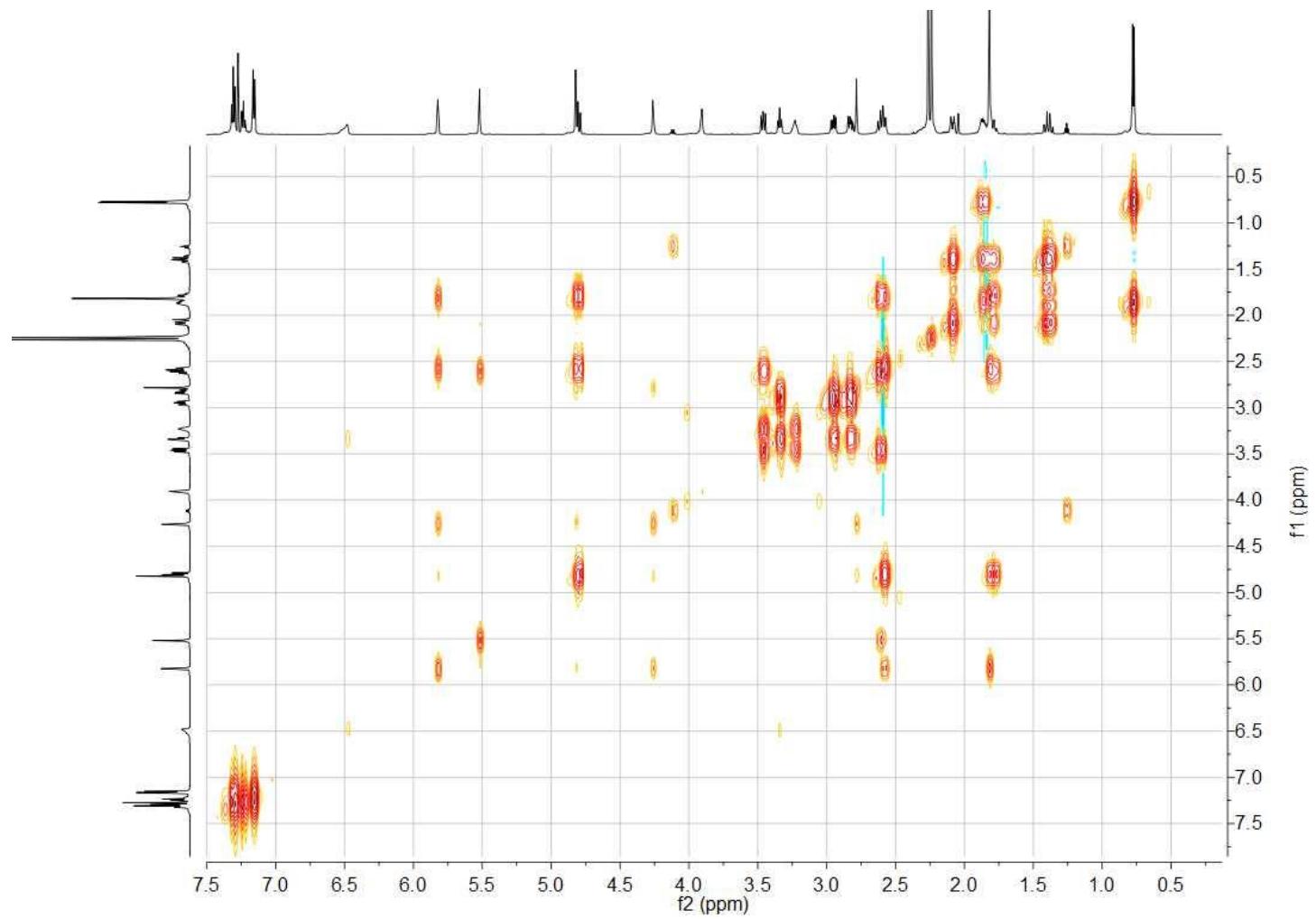
**Fig. S63.** DEPT of 7.



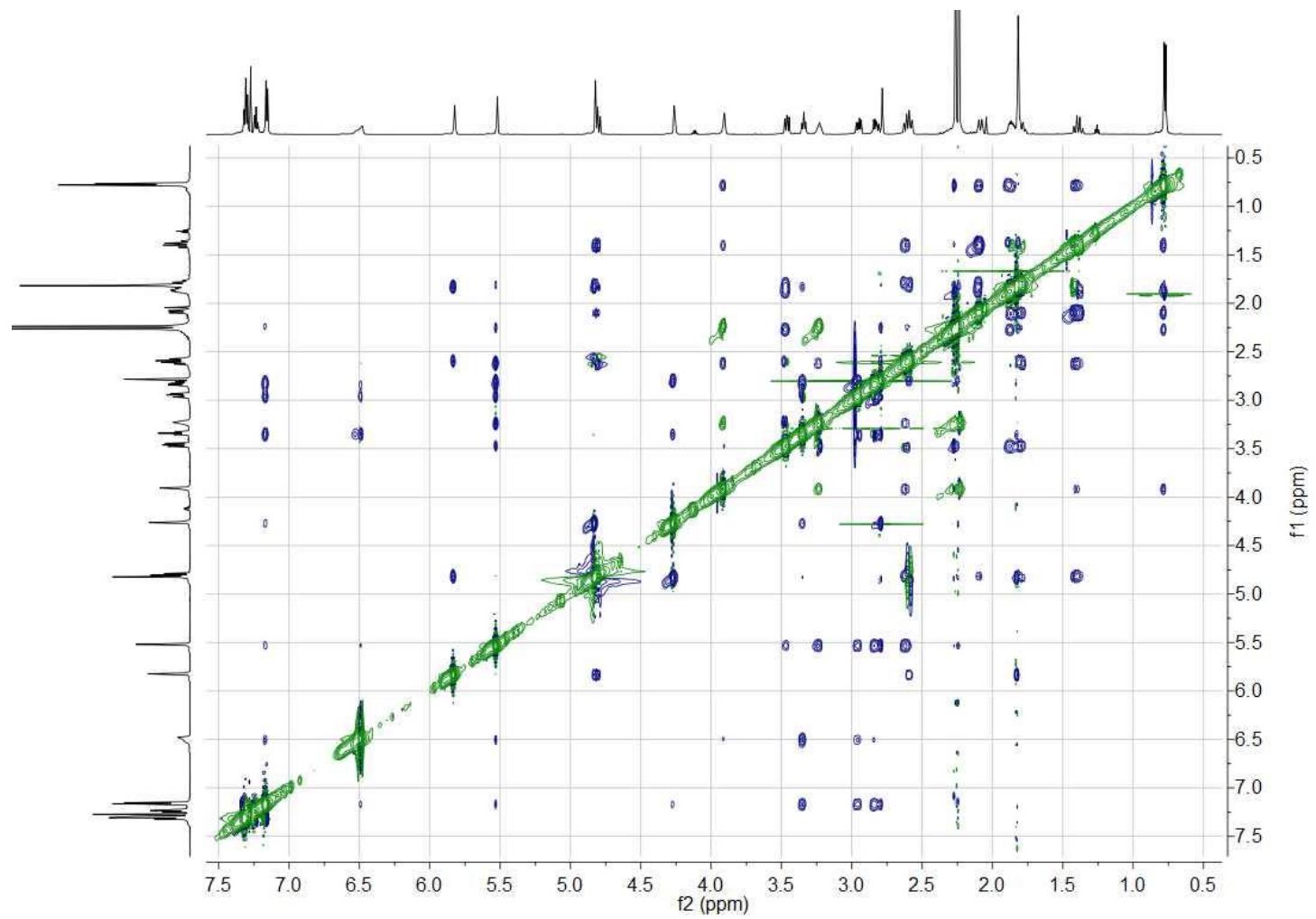
**Fig. S64.** HSQC of 7.



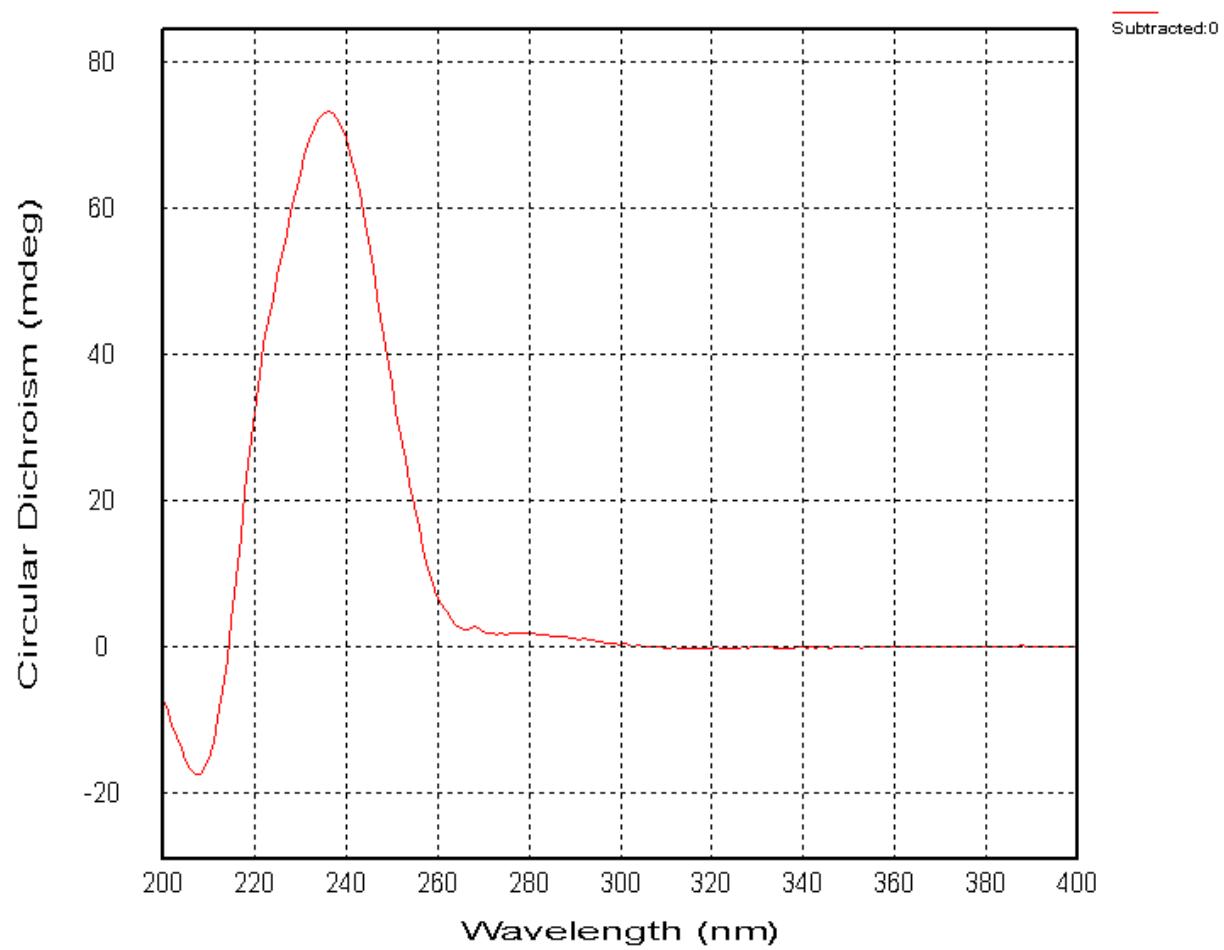
**Fig. S65.** HMBC of **7**.



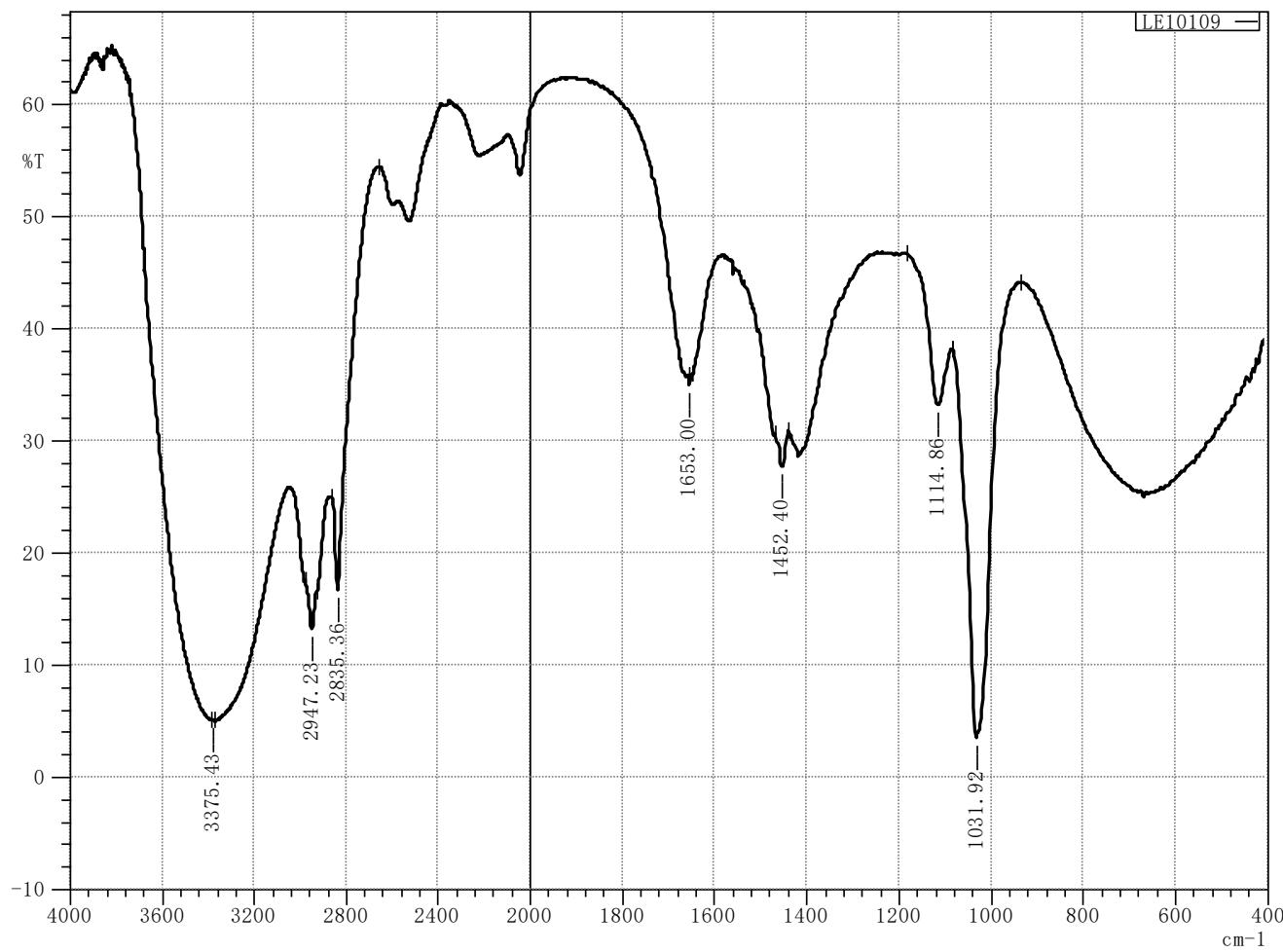
**Fig. S66.**  $^1\text{H}$ - $^1\text{H}$  COSY of **7**.



**Fig. S67.** ROESY of **7**.



**Fig. S68.** Experimental ECD of **7**.



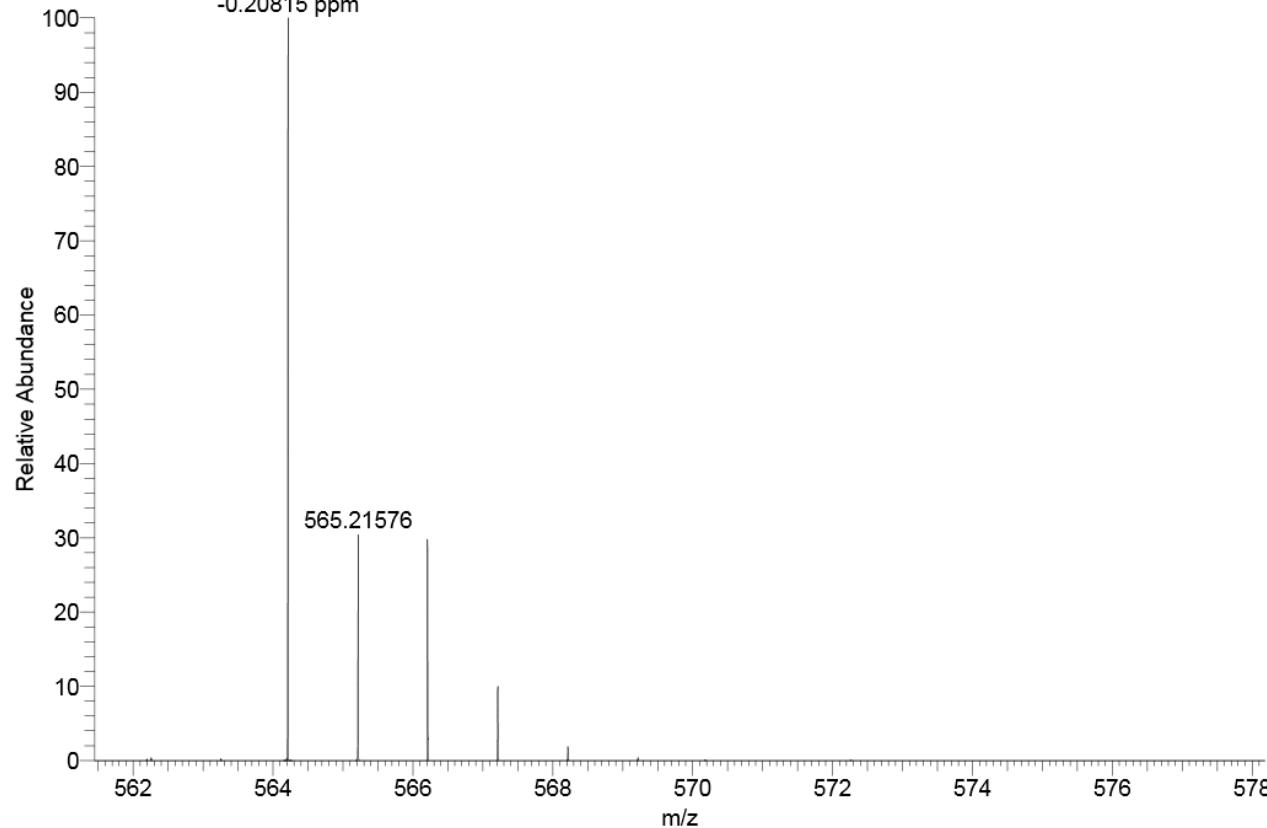
**Fig. S69.** IR of **7**.

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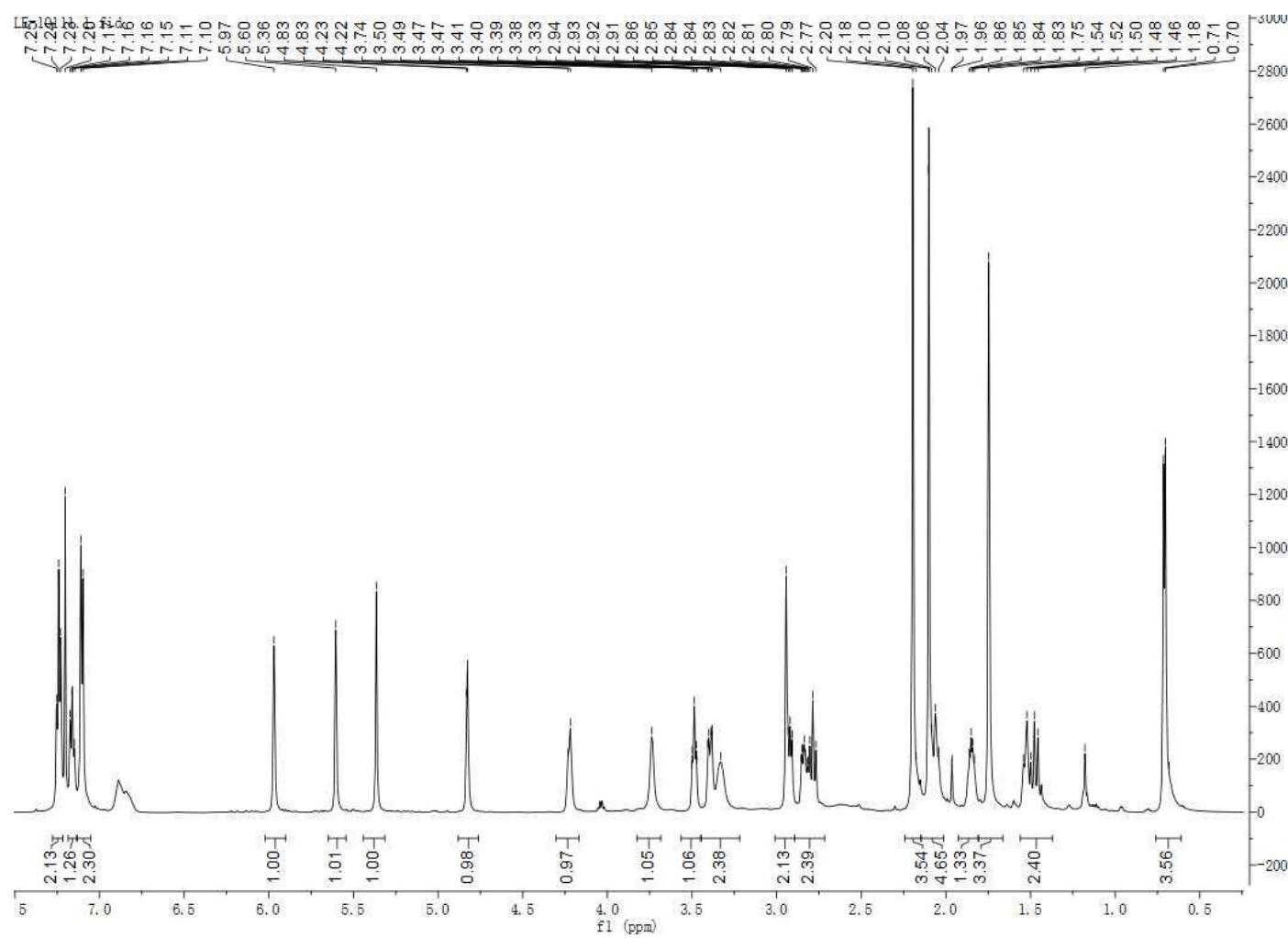
09/10/18 15:31:16

E106R25 #1302 RT: 18.09 AV: 1 NL: 1.55E8  
T: FTMS + p ESI Full lock ms [200.0000-2000.0000]

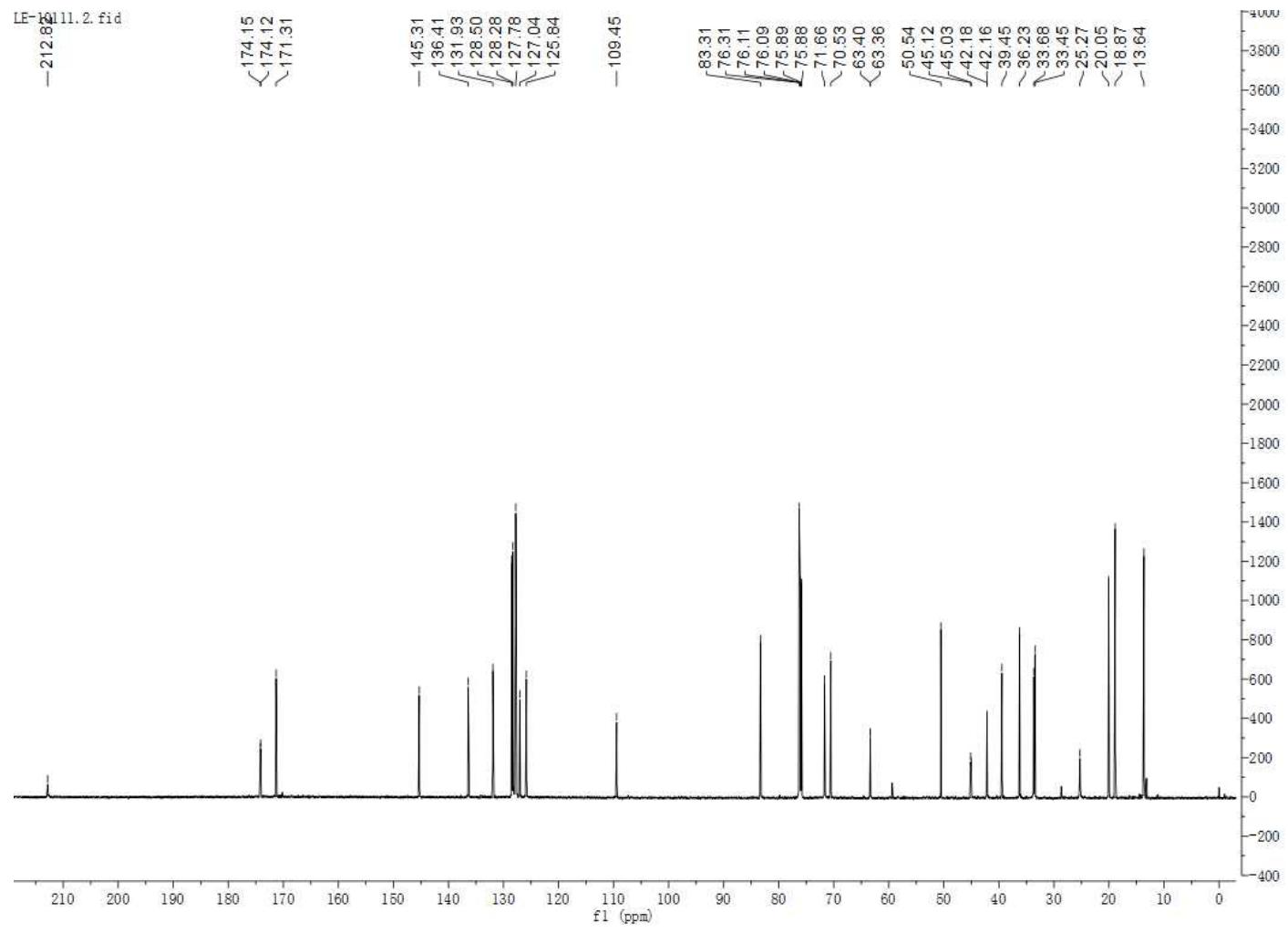
564.21222  
 $C_{30}H_{36}O_6NClNa$   
-0.20815 ppm



**Fig. S70.** HRESIMS of **7**.

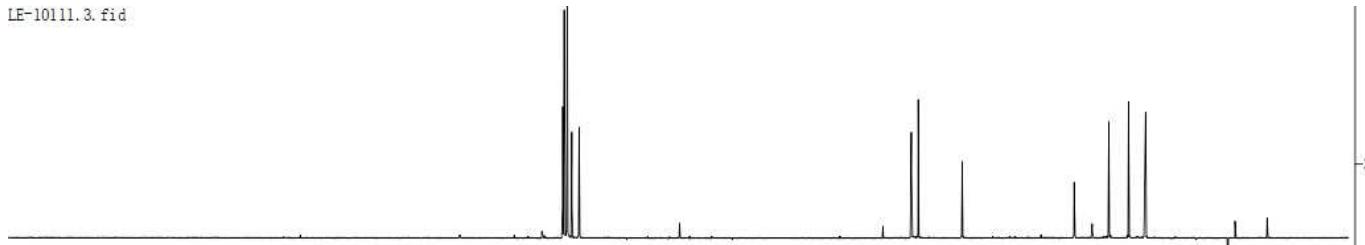


**Fig. S71.**  $^1\text{H}$  NMR of **8** ( $\text{CDCl}_3$ , 600 MHz).

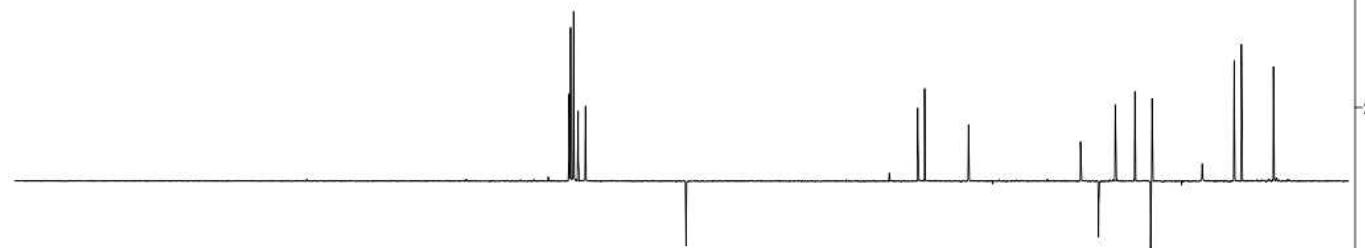


**Fig. S72.**  $^{13}\text{C}$  NMR of **8** ( $\text{CDCl}_3$ , 150 MHz).

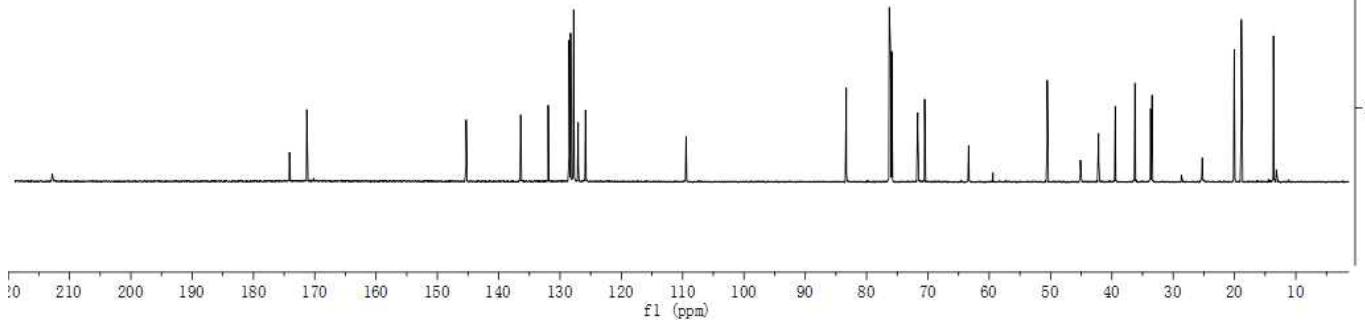
LE-10111.3.fid



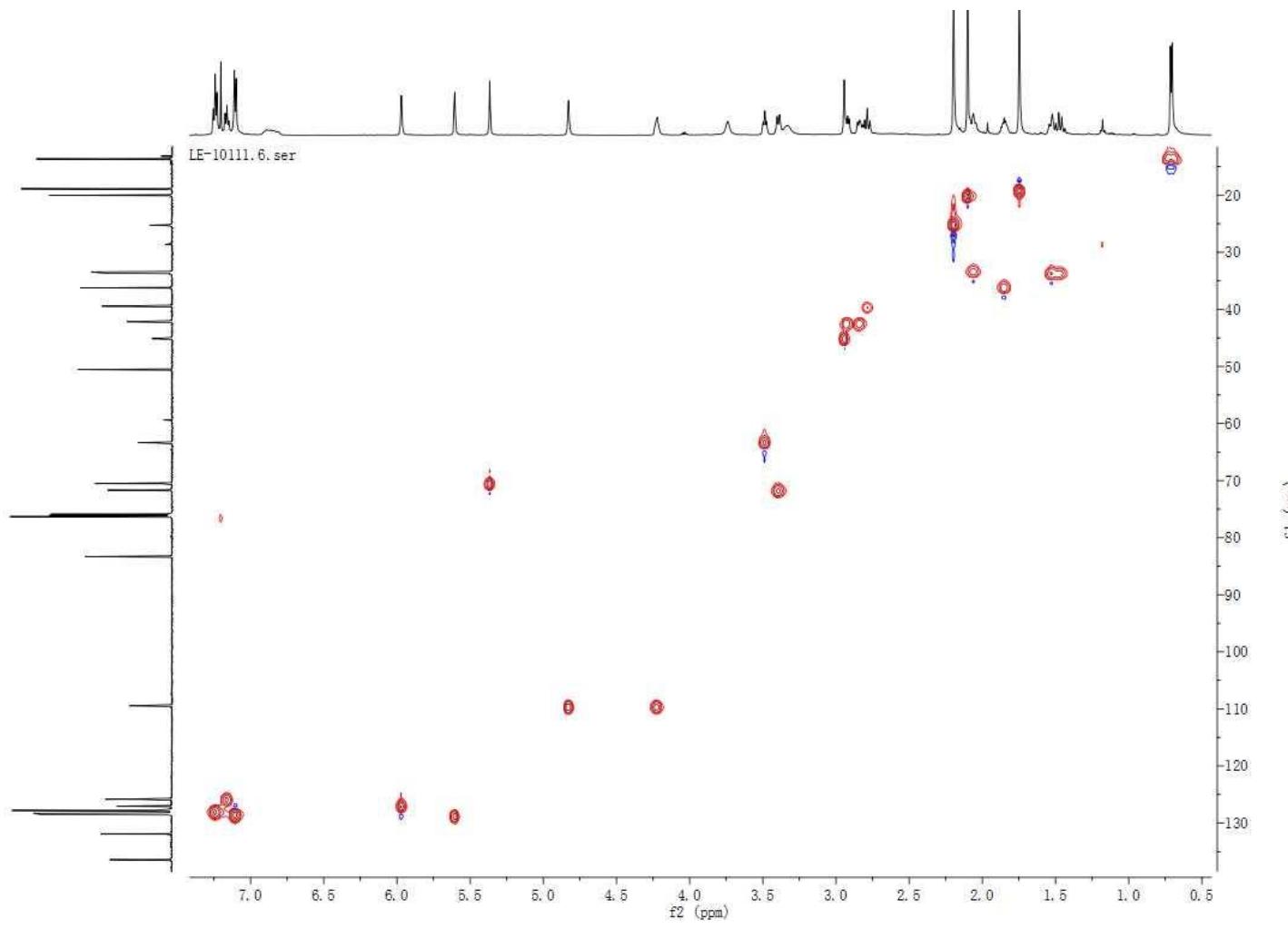
LE-10111.4.fid



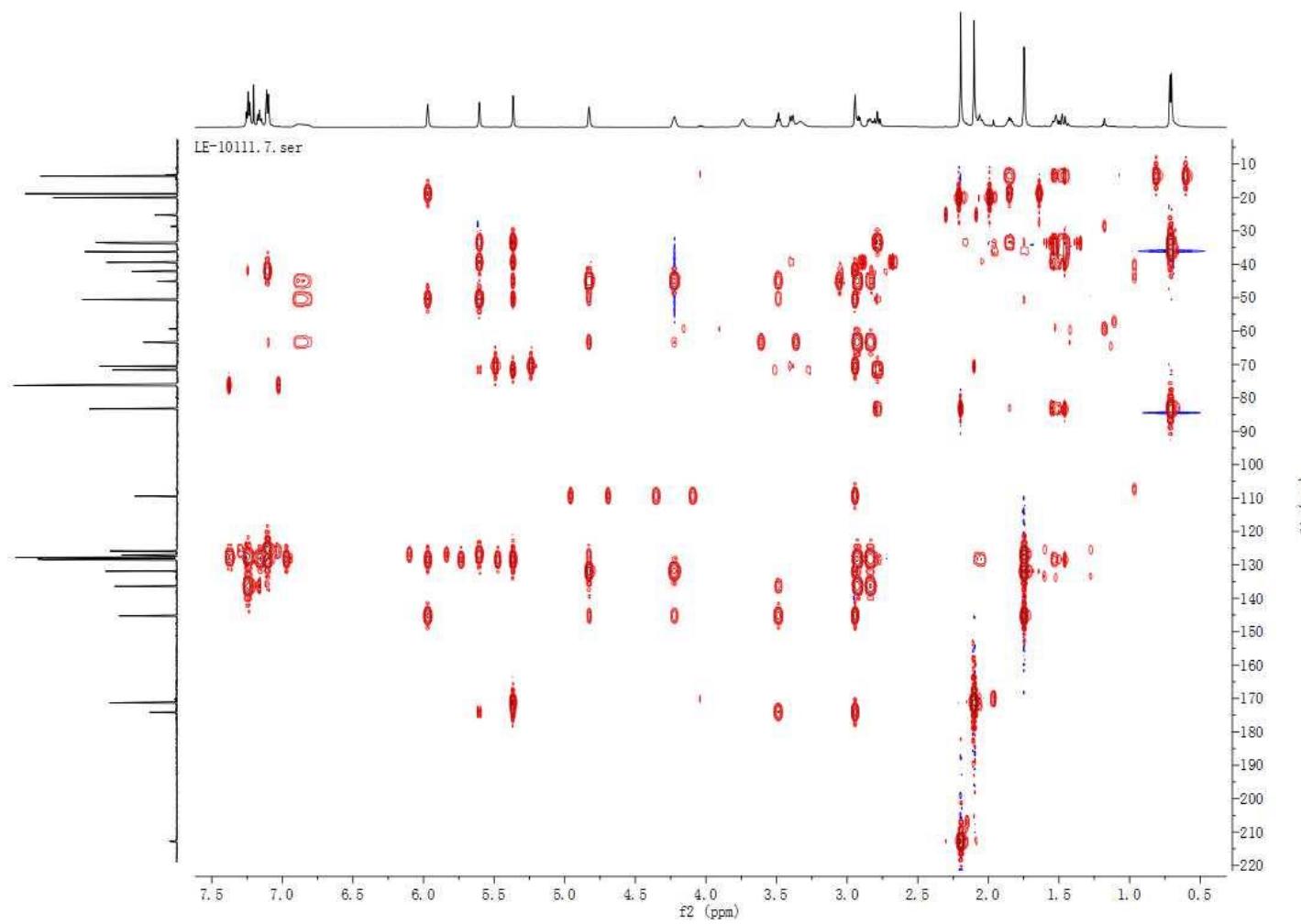
LE-10111.2.fid



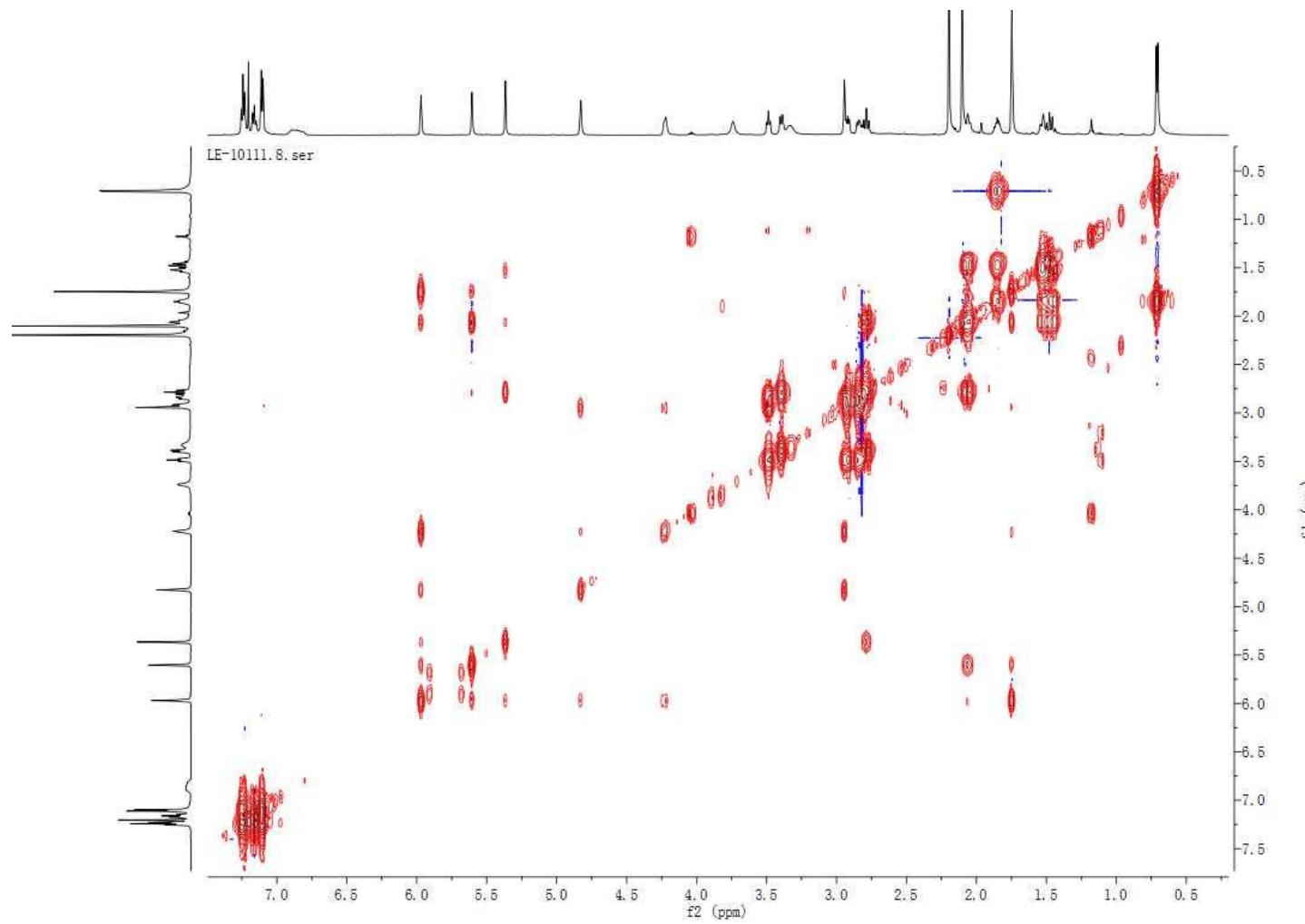
**Fig. S73.** DEPT of **8**.



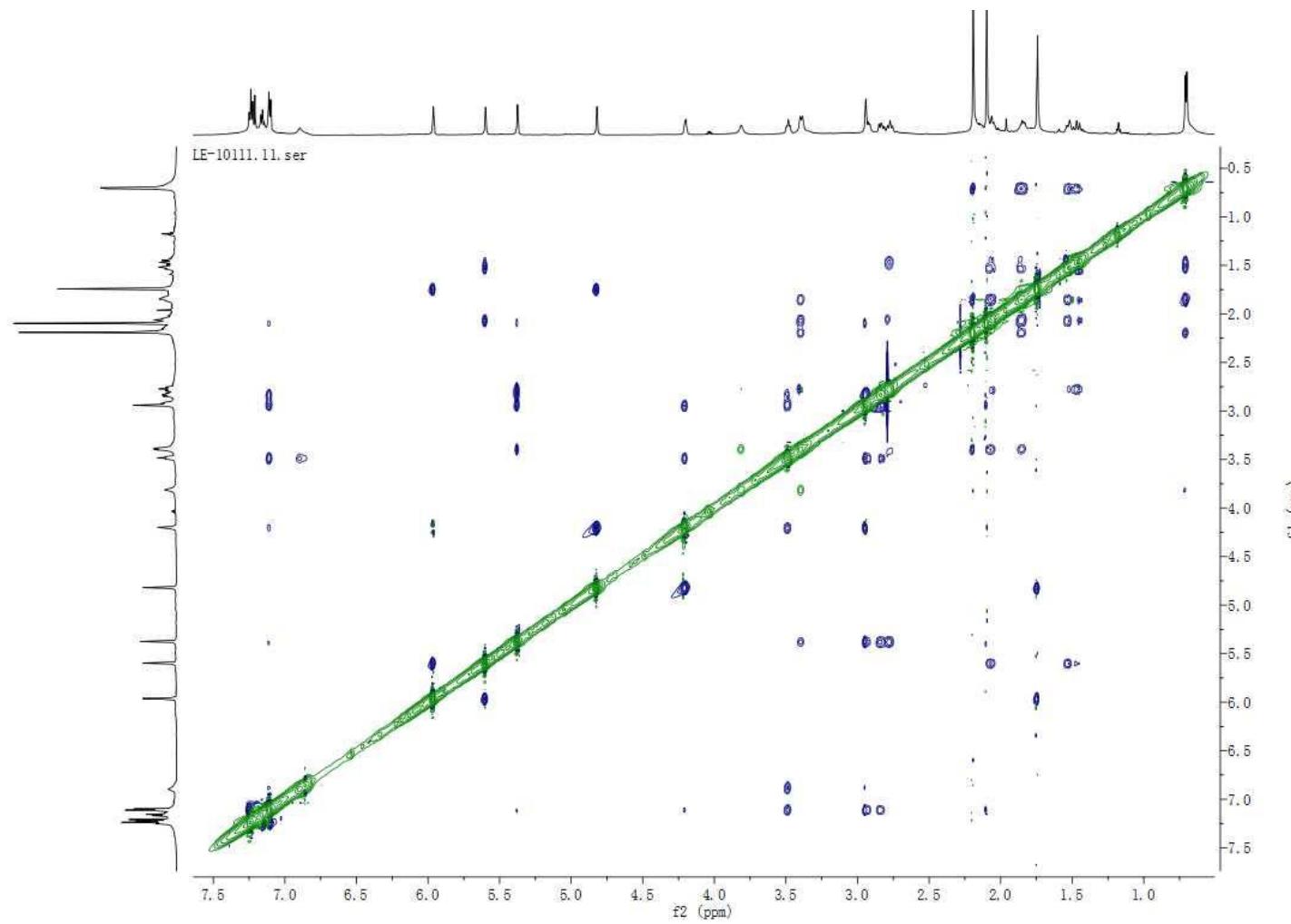
**Fig. S74.** HSQC of **8**.



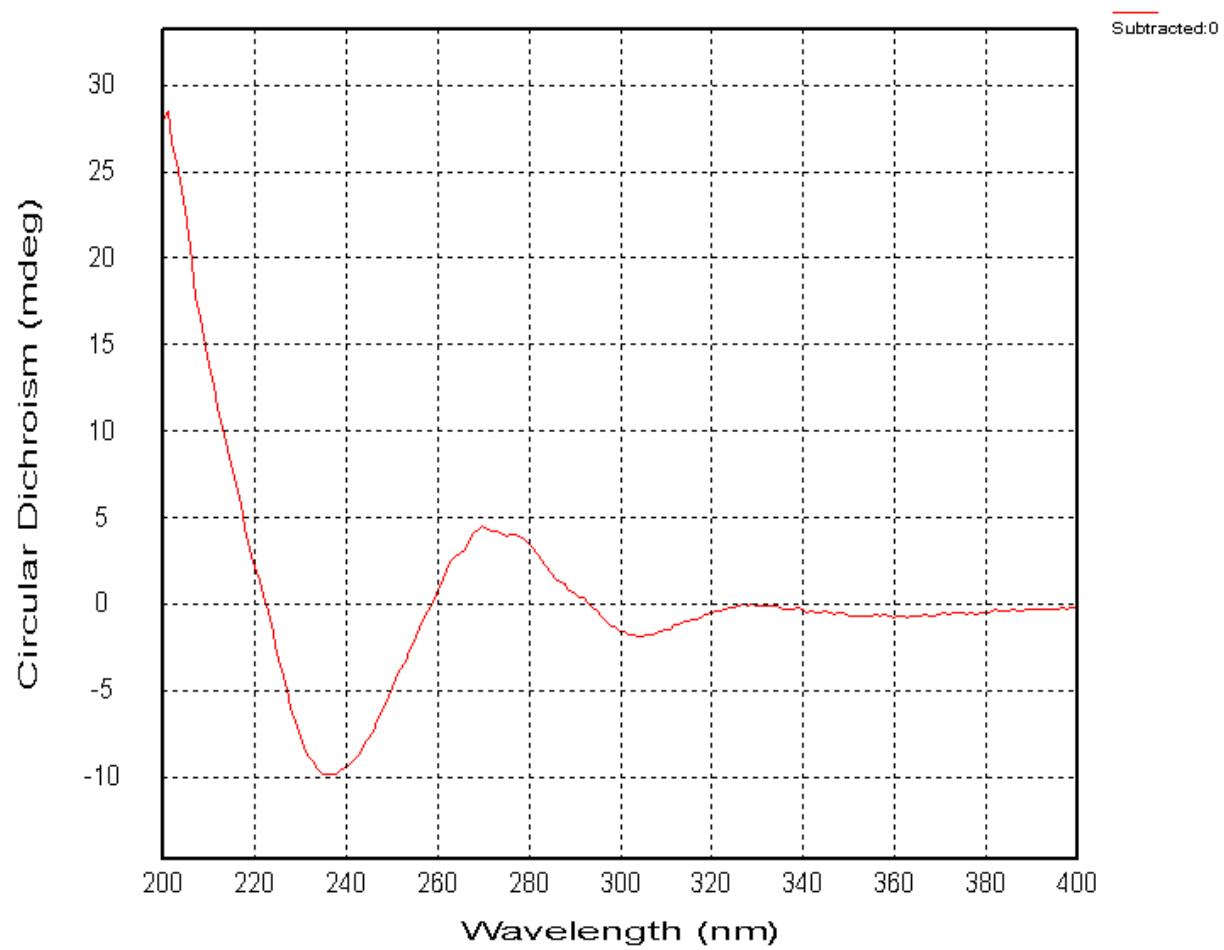
**Fig. S75.** HMBC of **8**.



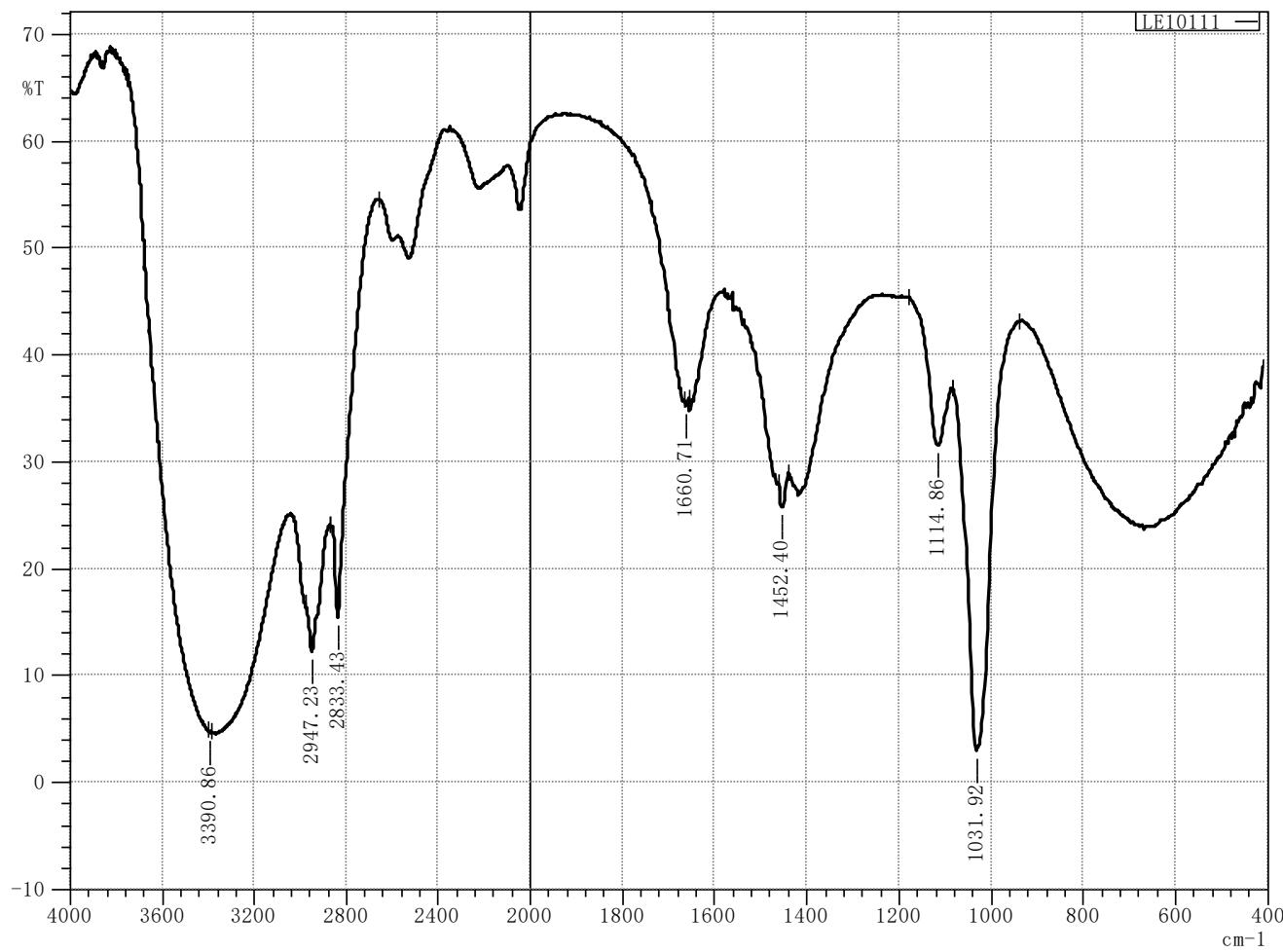
**Fig. S76.**  $^1\text{H}$ - $^1\text{H}$  COSY of **8**.



**Fig. S77.** ROESY of **8**.



**Fig. S78.** Experimental ECD of **8**.

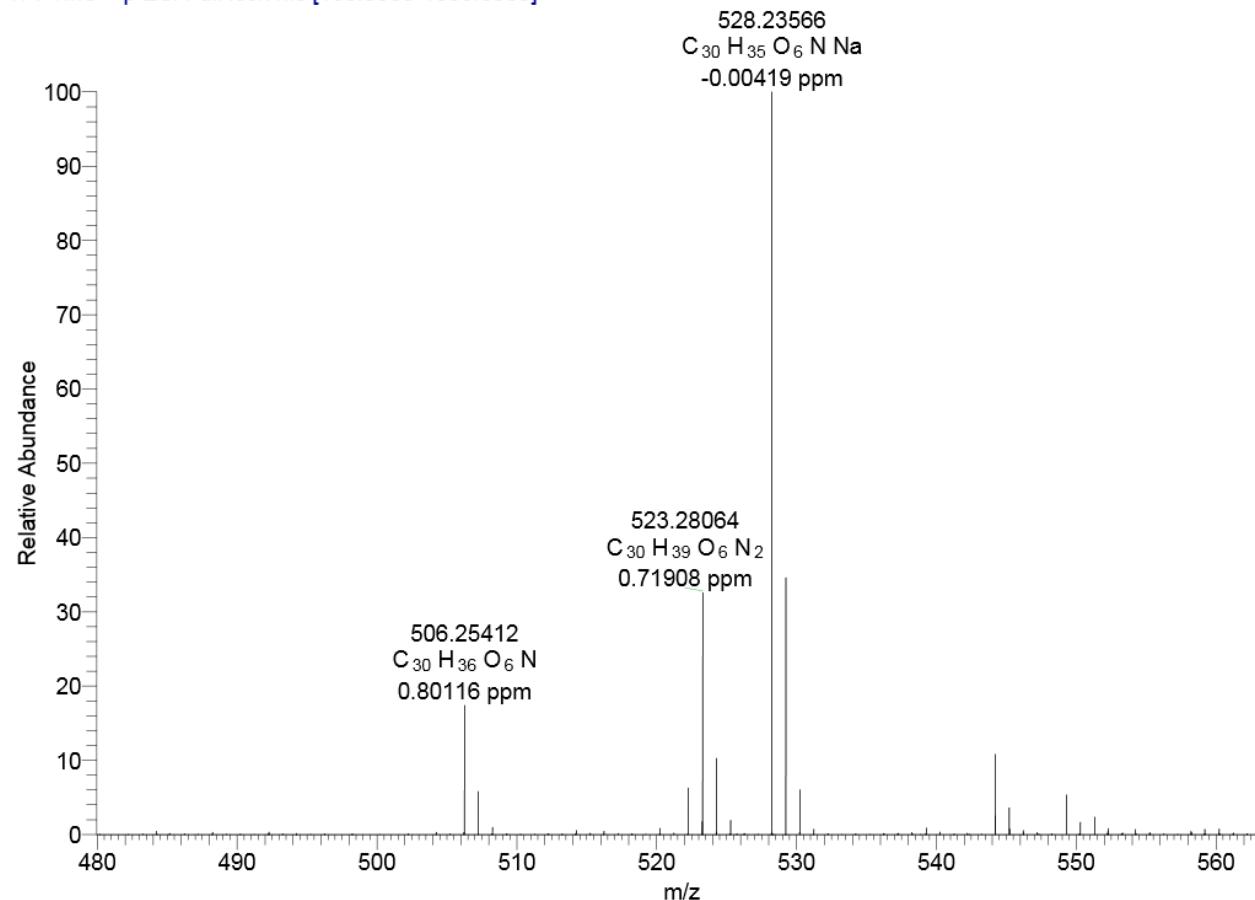


**Fig. S79.** IR of **8**.

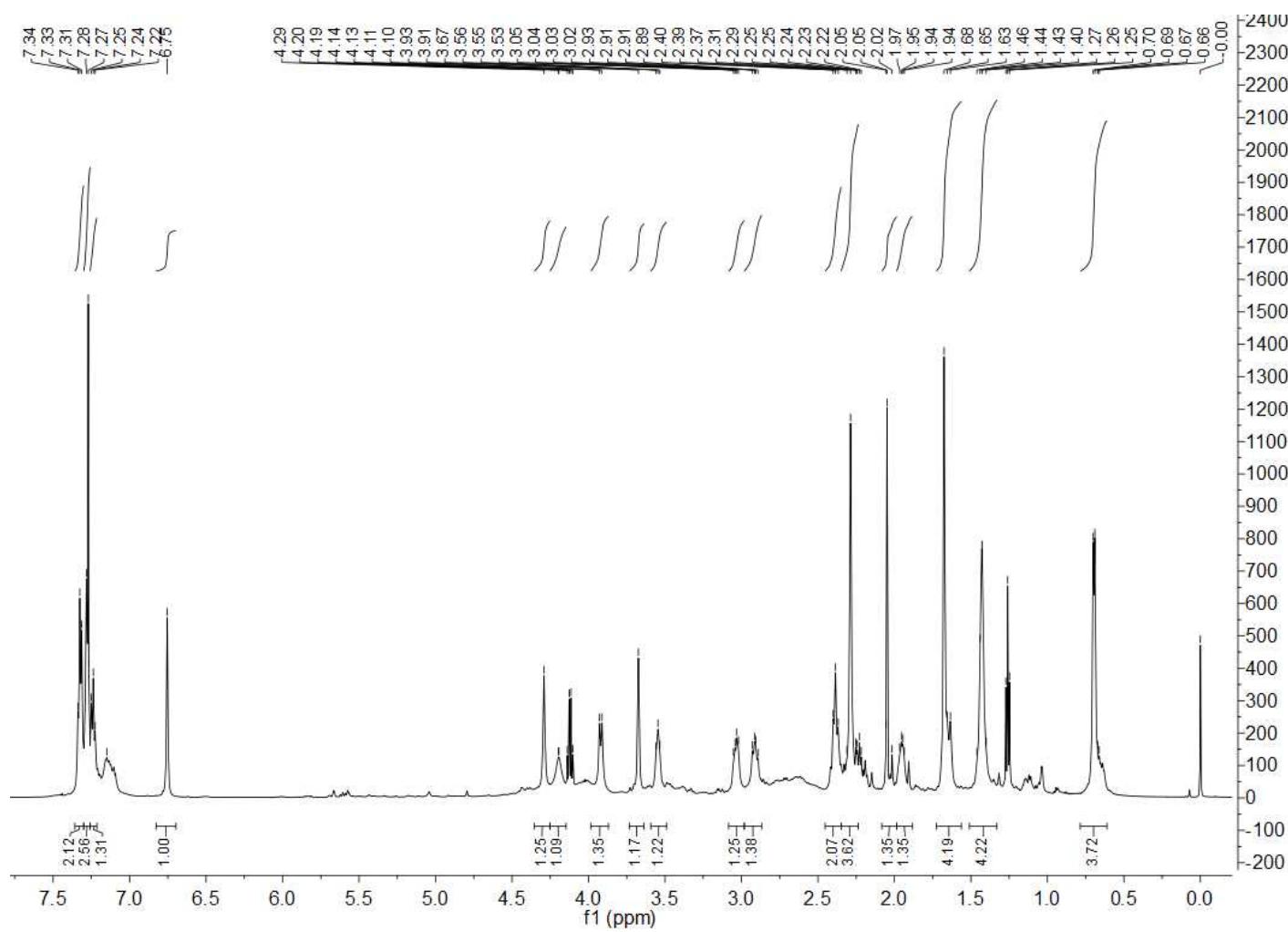
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07/02/19 19:02:57

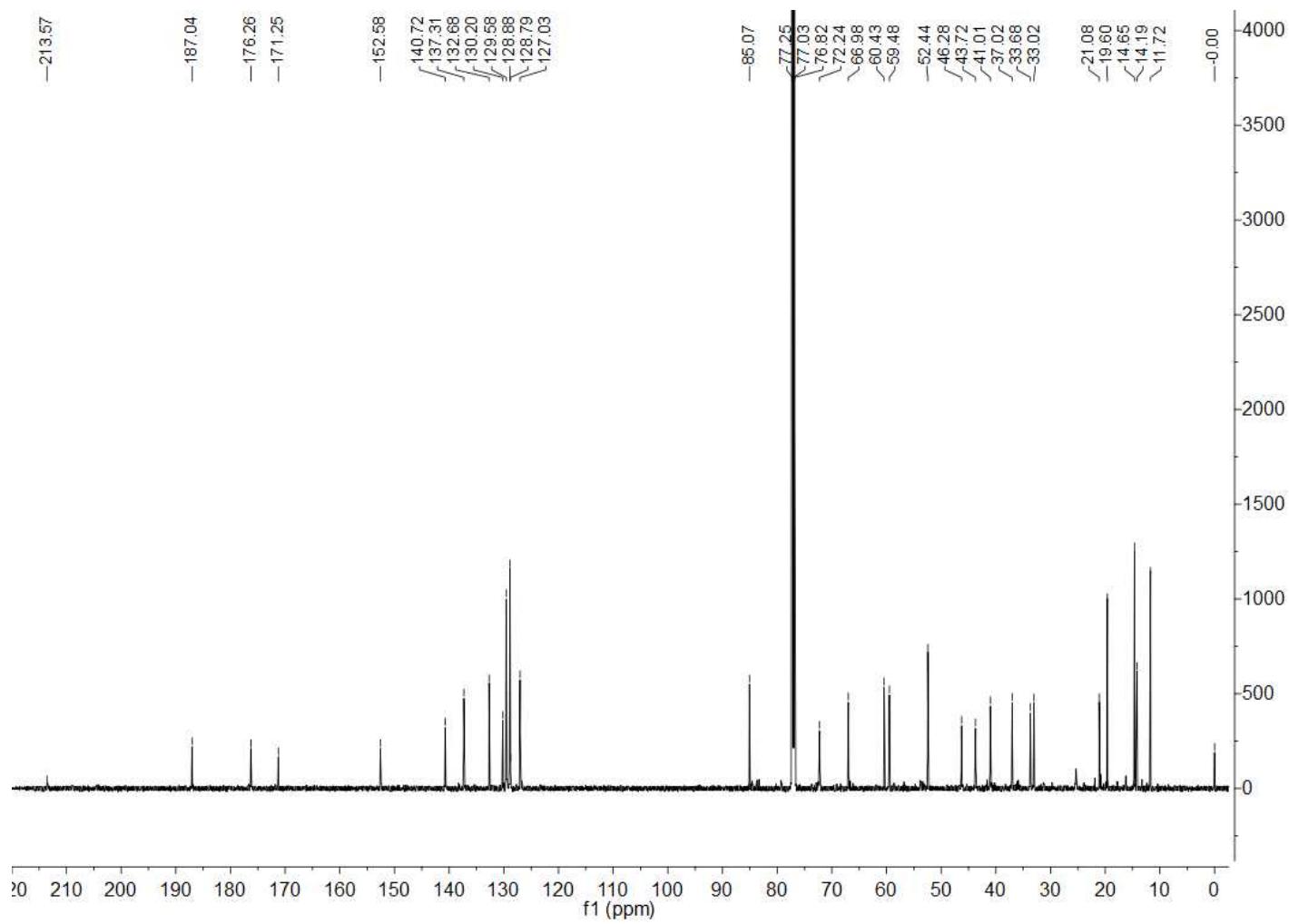
LE10111 #1704 RT: 14.86 AV: 1 NL: 1.92E8  
T: FTMS + p ESI Full lock ms [100.0000-1000.0000]



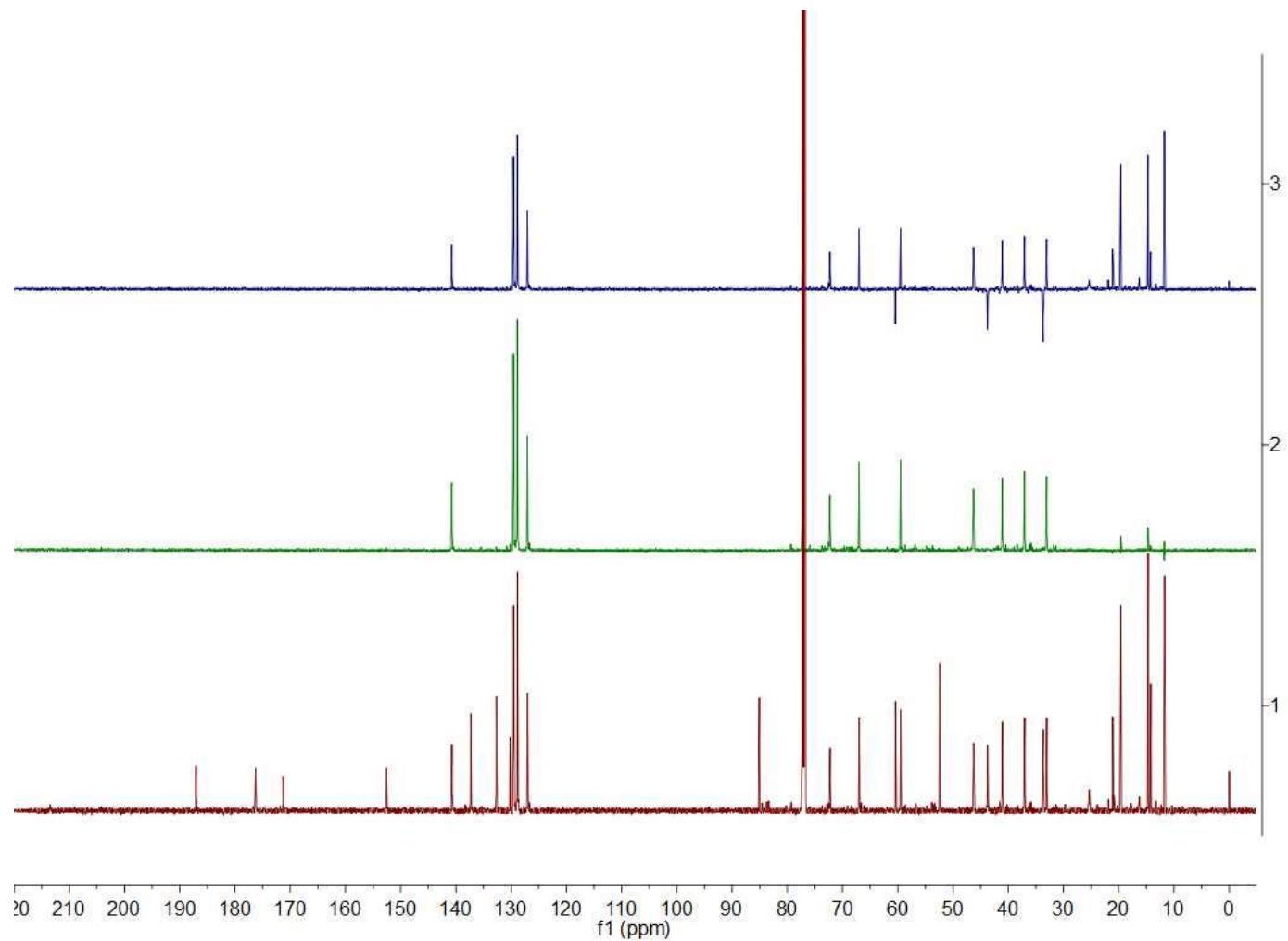
**Fig. S80.** HRESIMS of **8**.



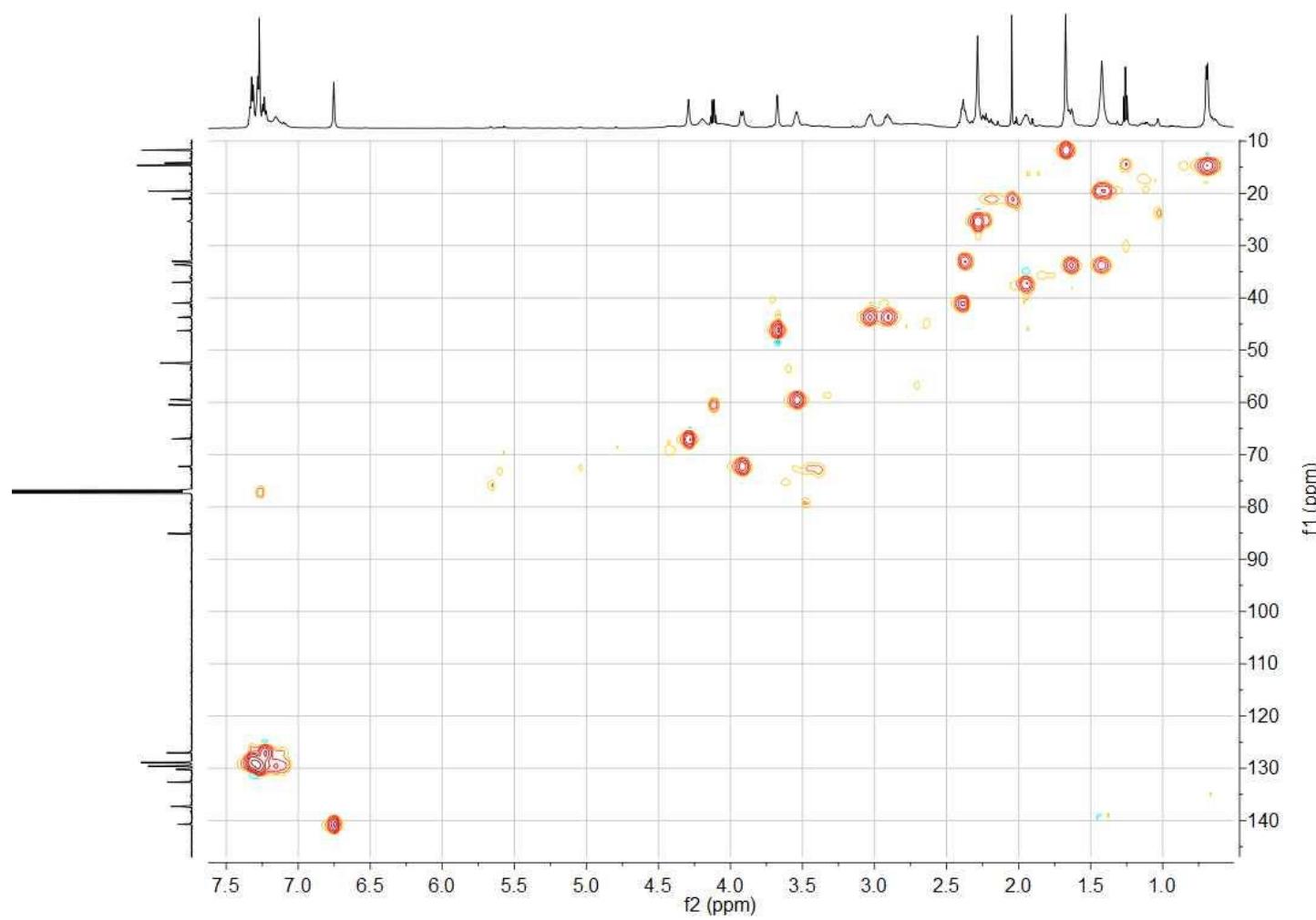
**Fig. S81.**  $^1\text{H}$  NMR of **9** ( $\text{CDCl}_3$ , 600 MHz).



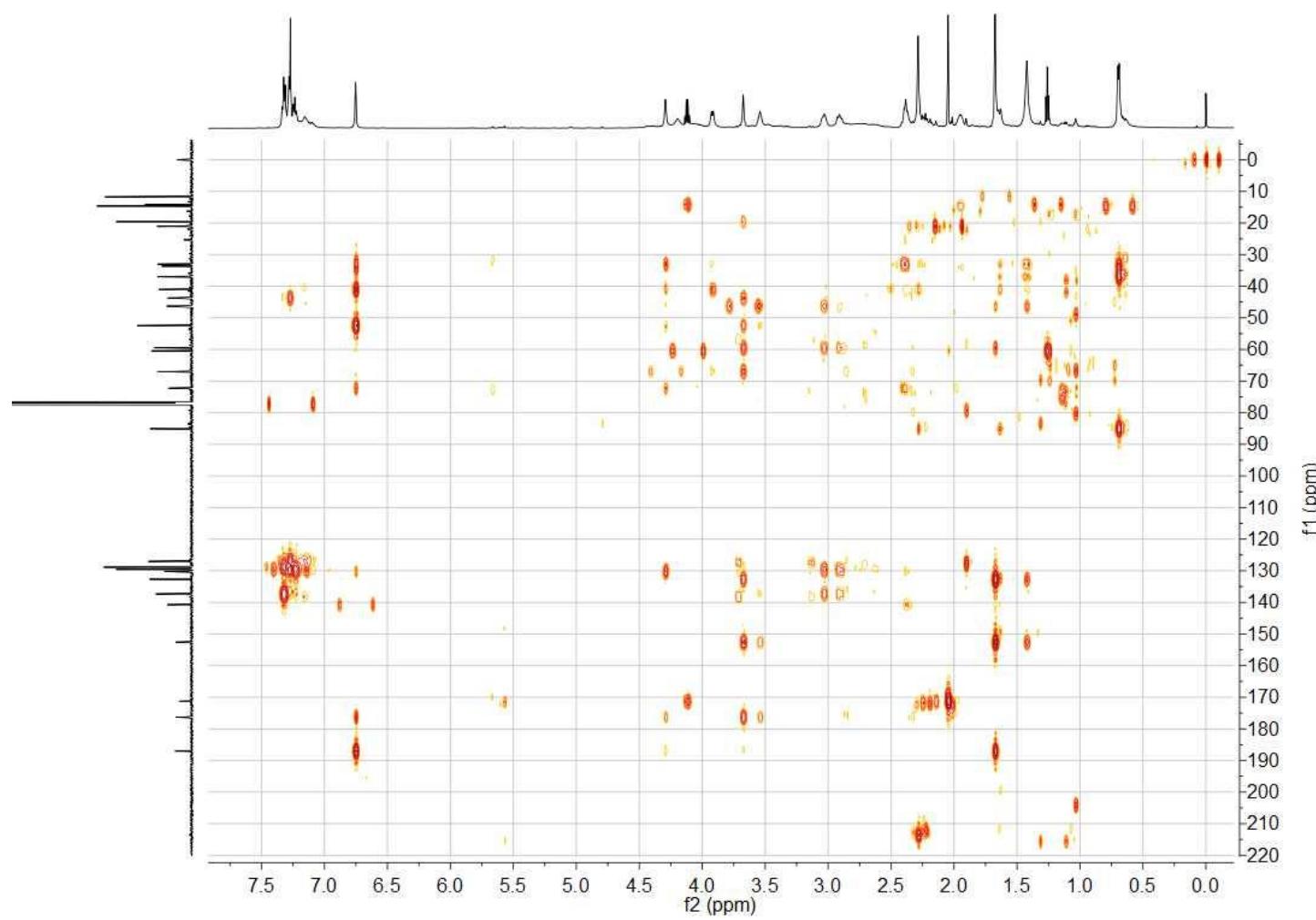
**Fig. S82.**  $^{13}\text{C}$  NMR of **9** ( $\text{CDCl}_3$ , 150 MHz).



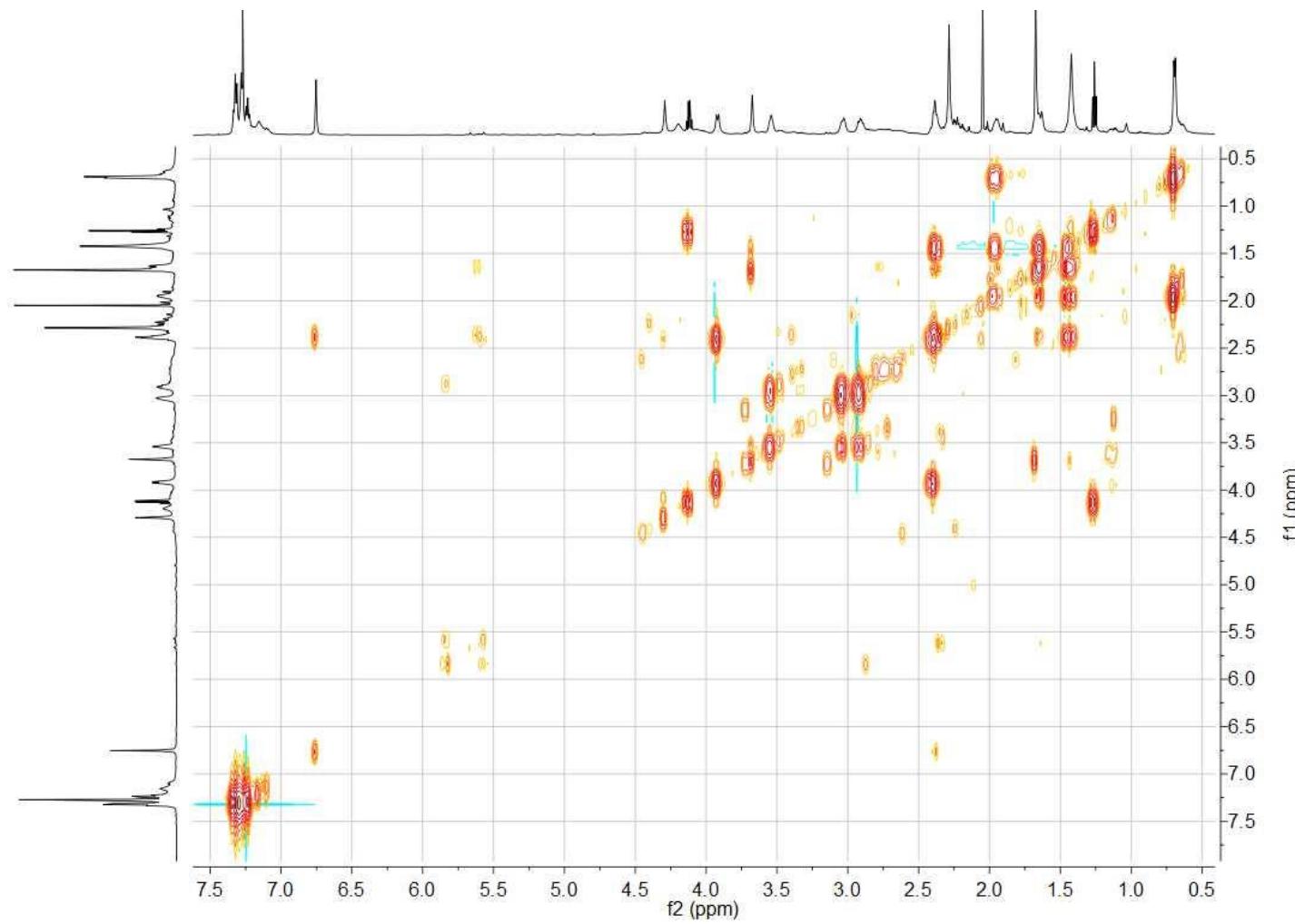
**Fig. S83.** DEPT of 9.



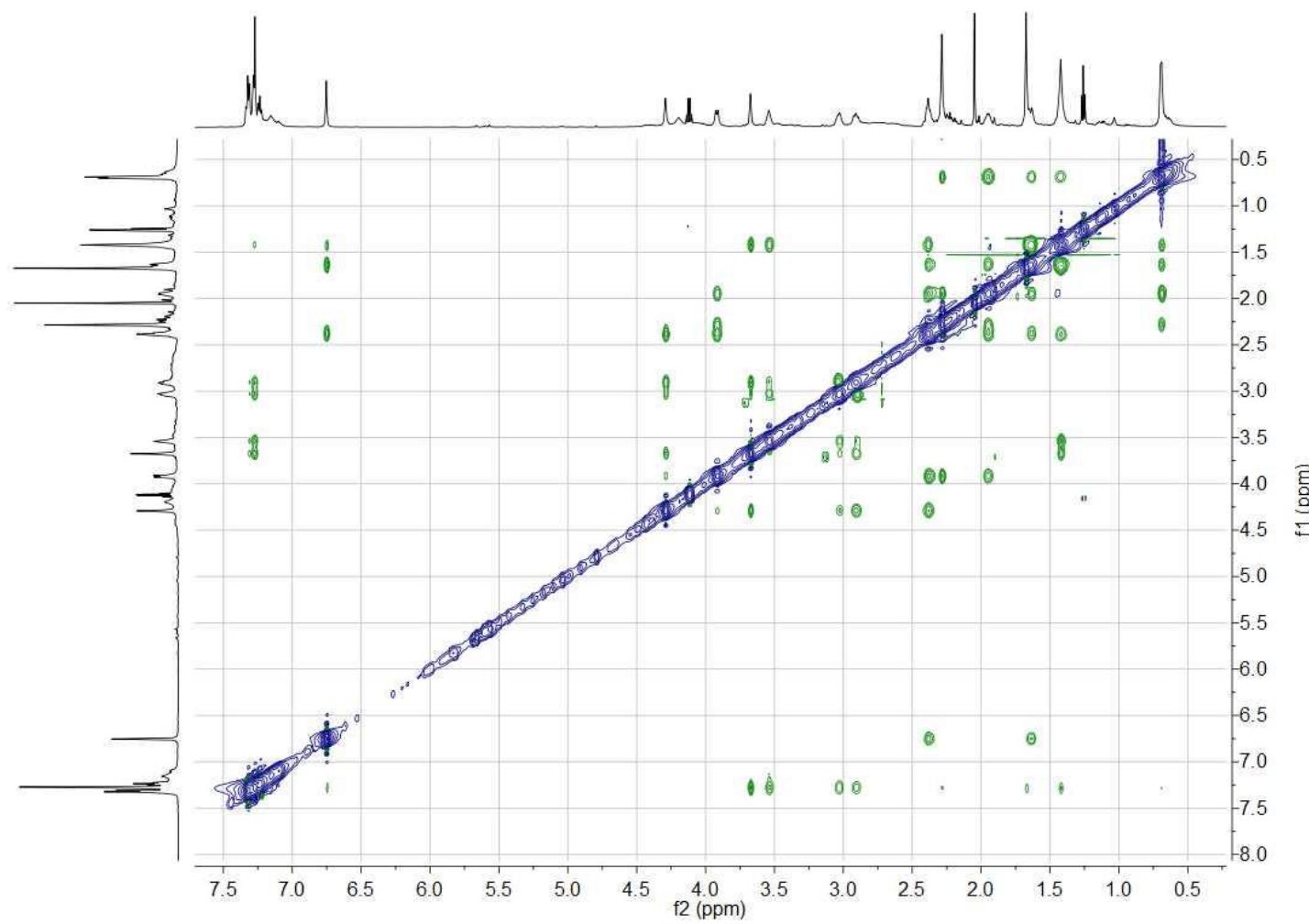
**Fig. S84.** HSQC of **9**.



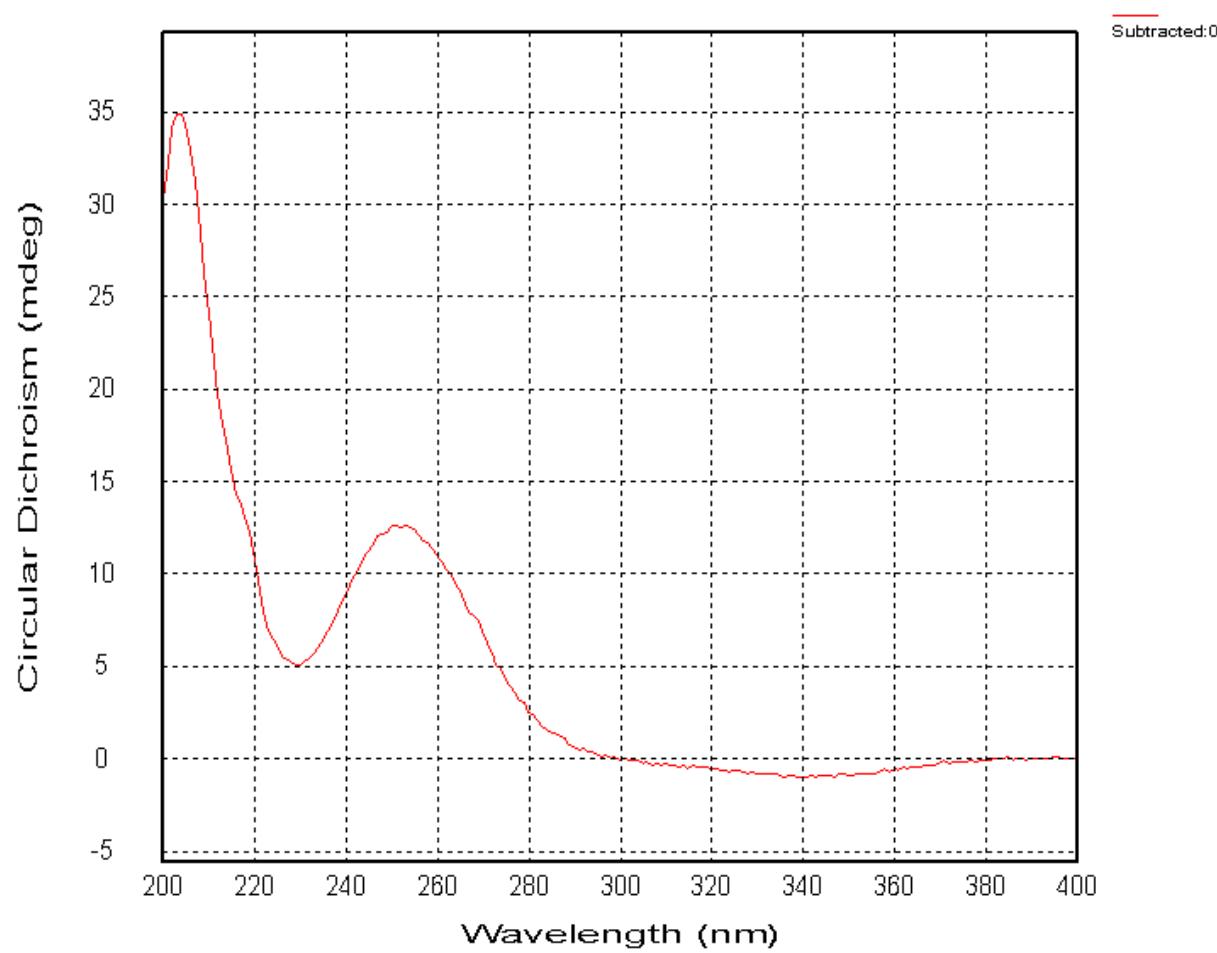
**Fig. S85.** HMBC of **9**.



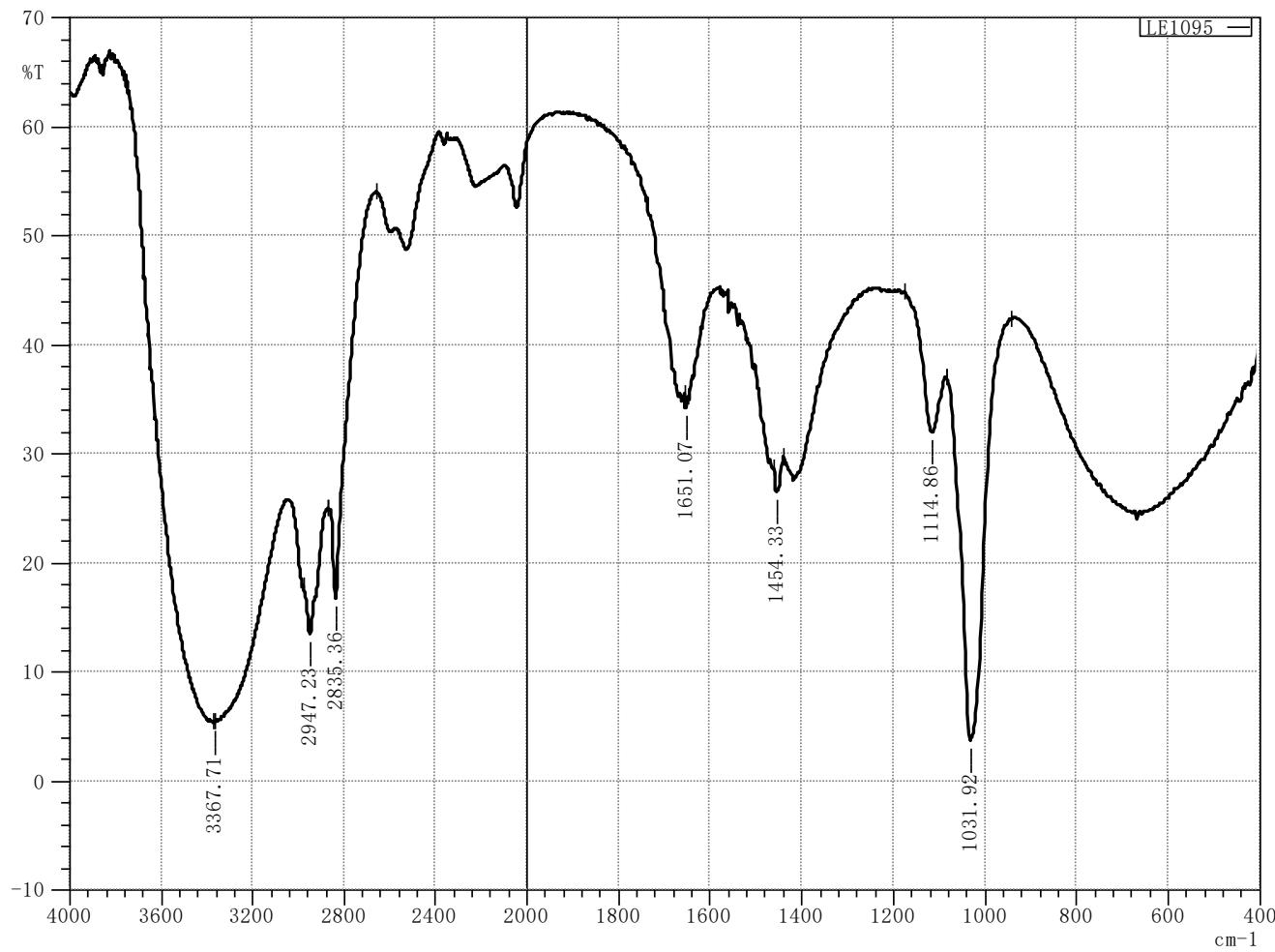
**Fig. S86.**  $^1\text{H}$ - $^1\text{H}$  COSY of 9.



**Fig. S87.** ROESY of **9**.



**Fig. S88.** Experimental ECD of **9**.

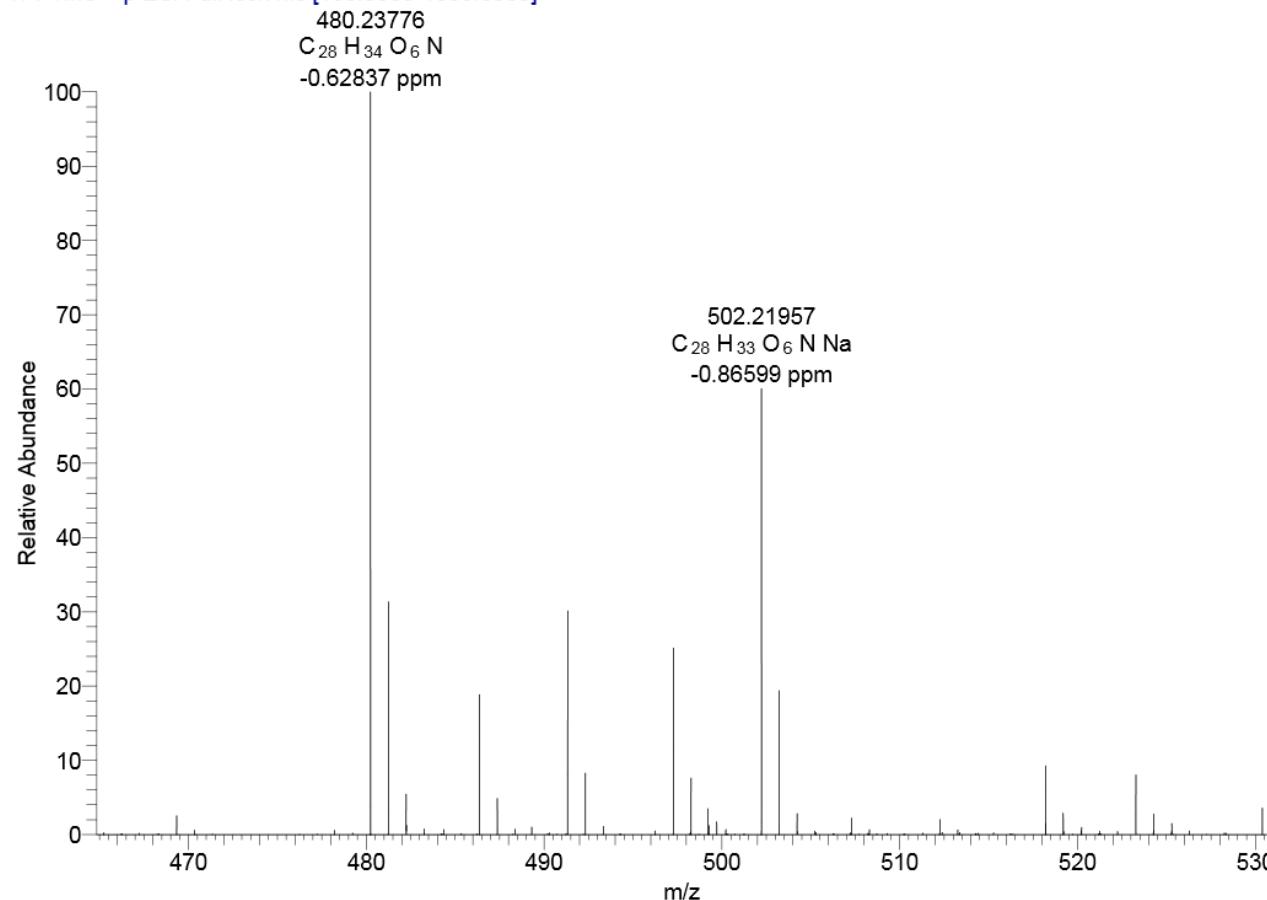


**Fig. S89.** IR of **9**.

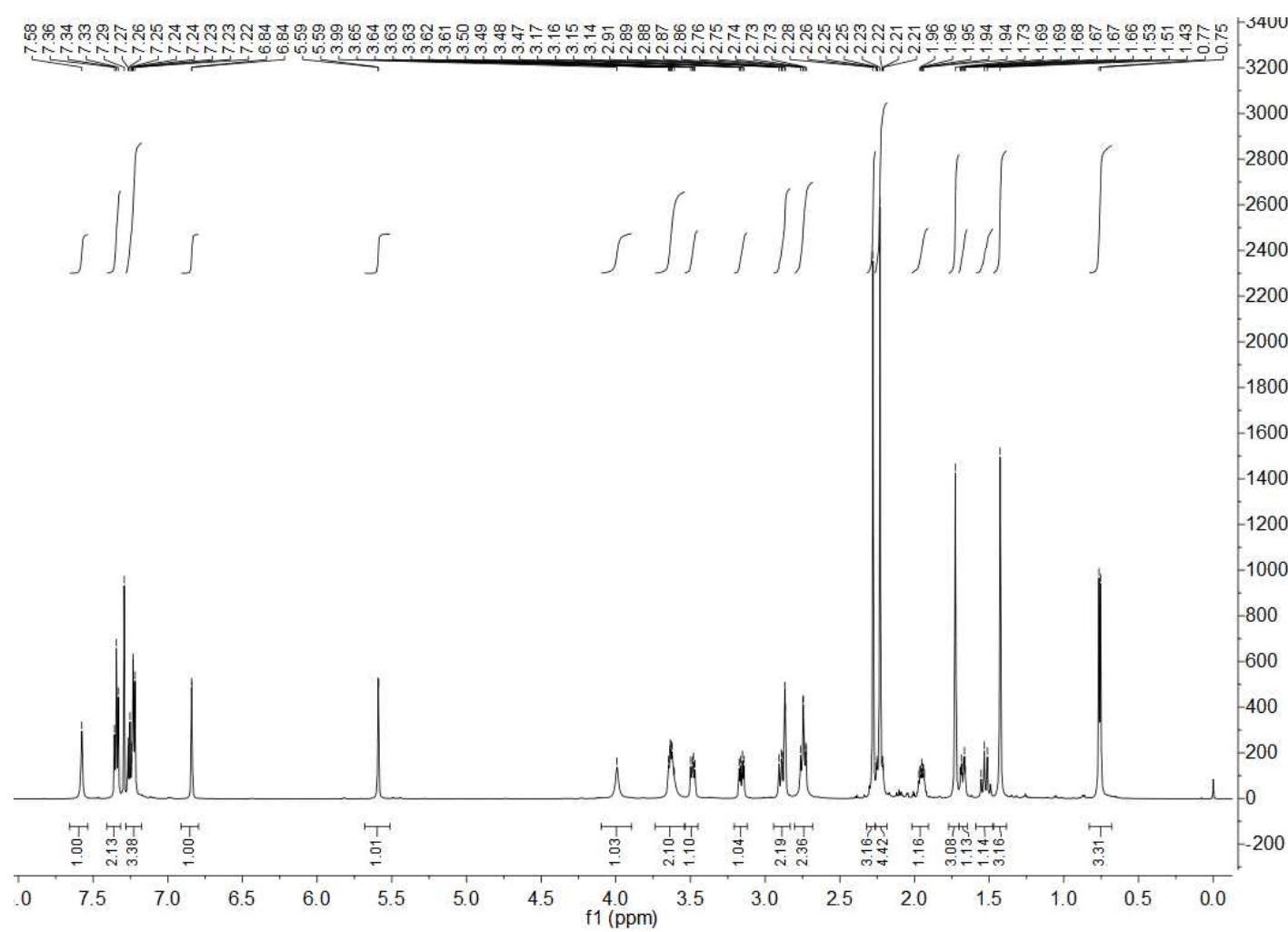
D:\Wen-Xuan\2019\20190604\LE1095

07/02/19 18:30:09

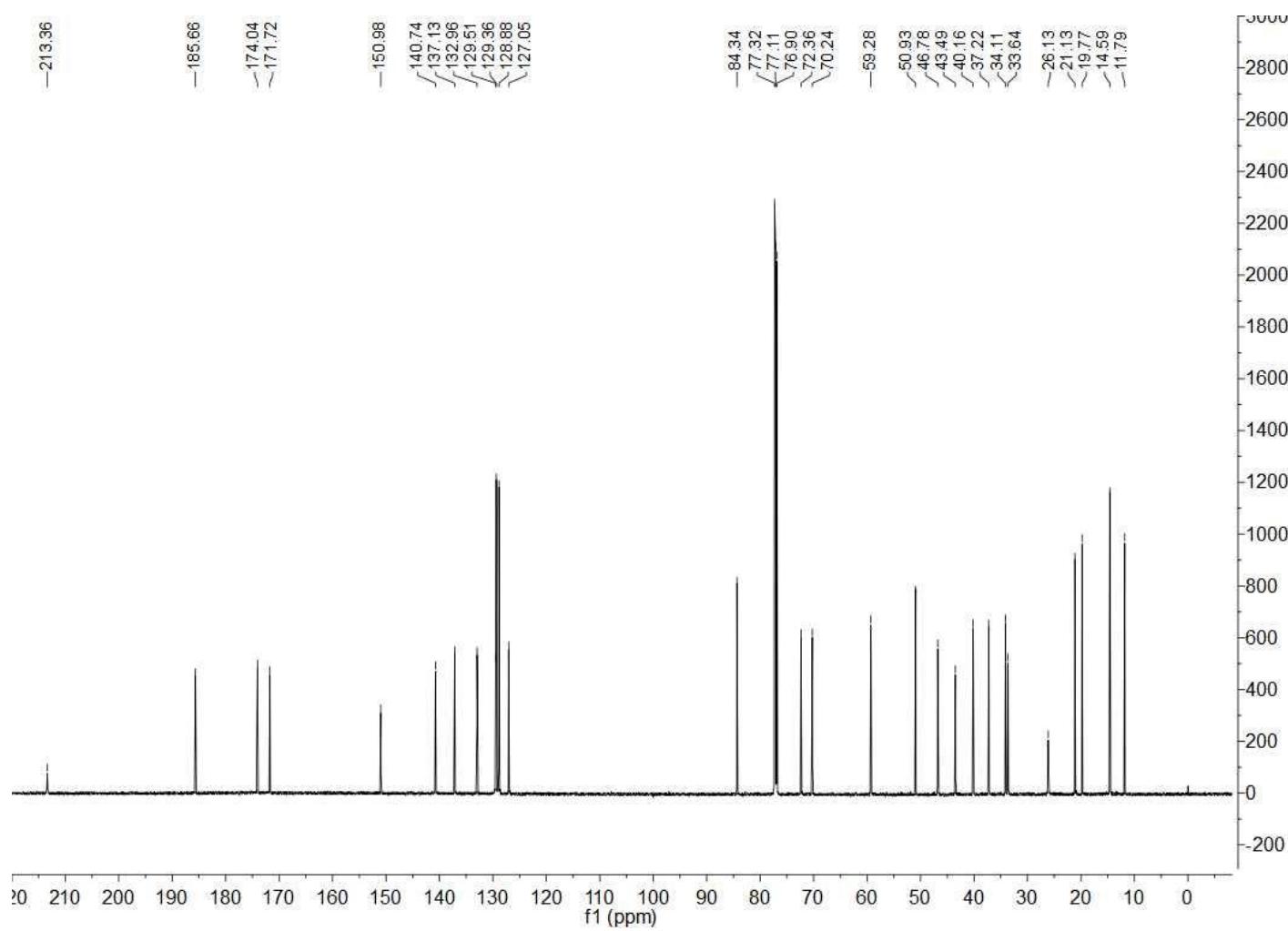
LE1095 #1610 RT: 14.04 AV: 1 NL: 3.14E8  
T: FTMS + p ESI Full lock ms [100.0000-1000.0000]



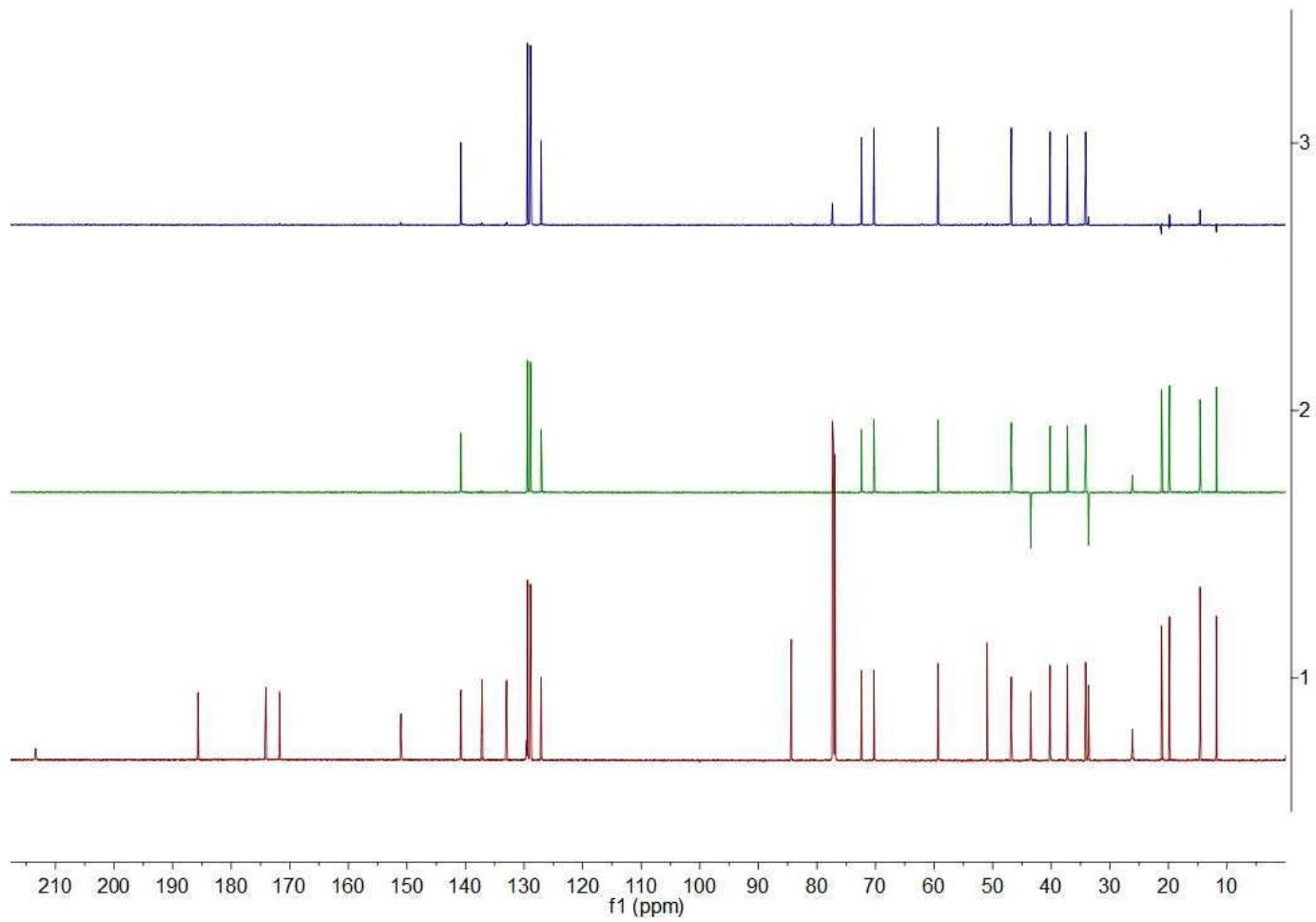
**Fig. S90.** HRESIMS of **9**.



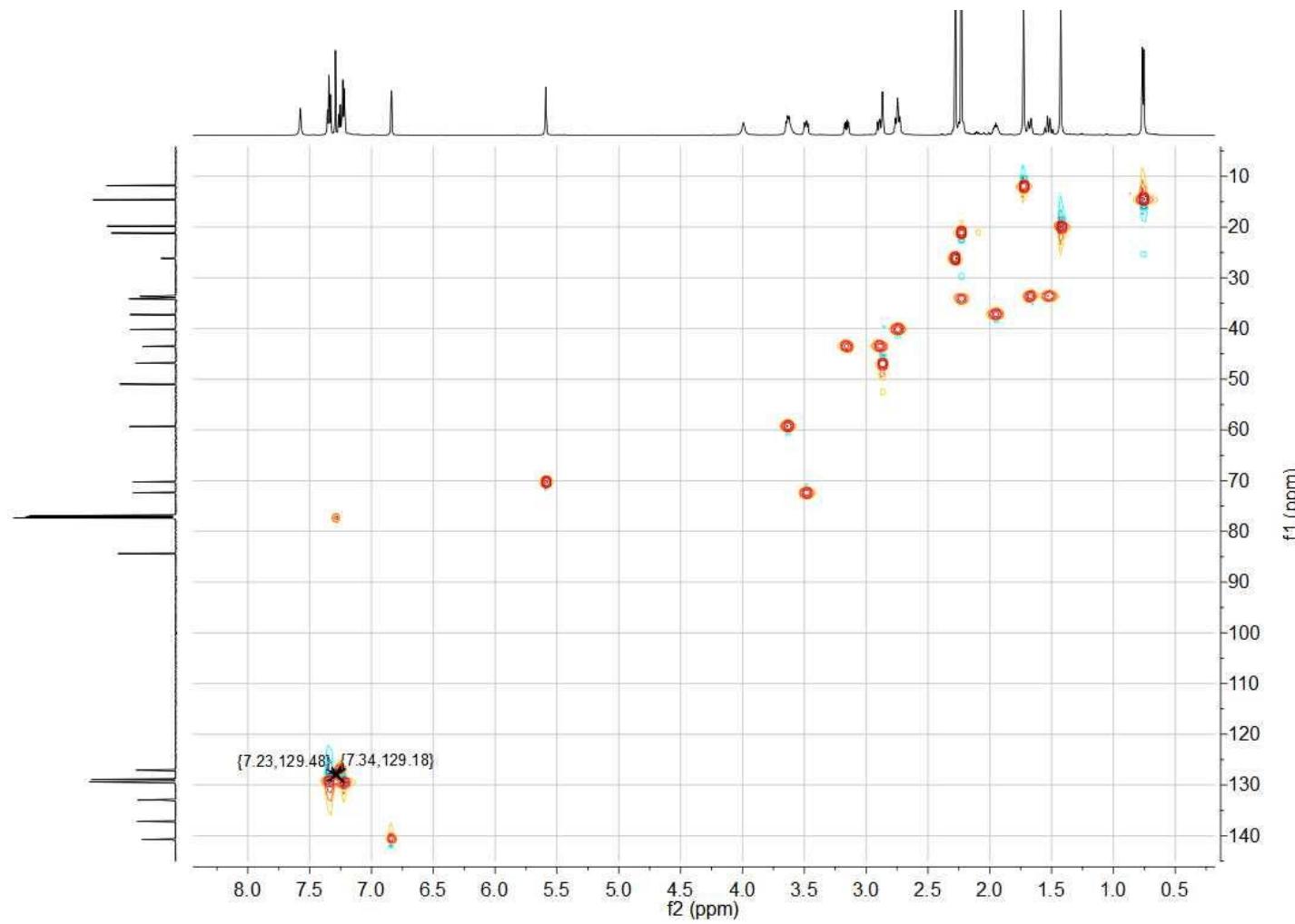
**Fig. S91.** <sup>1</sup>H NMR of **10** ( $\text{CDCl}_3$ , 600 MHz).



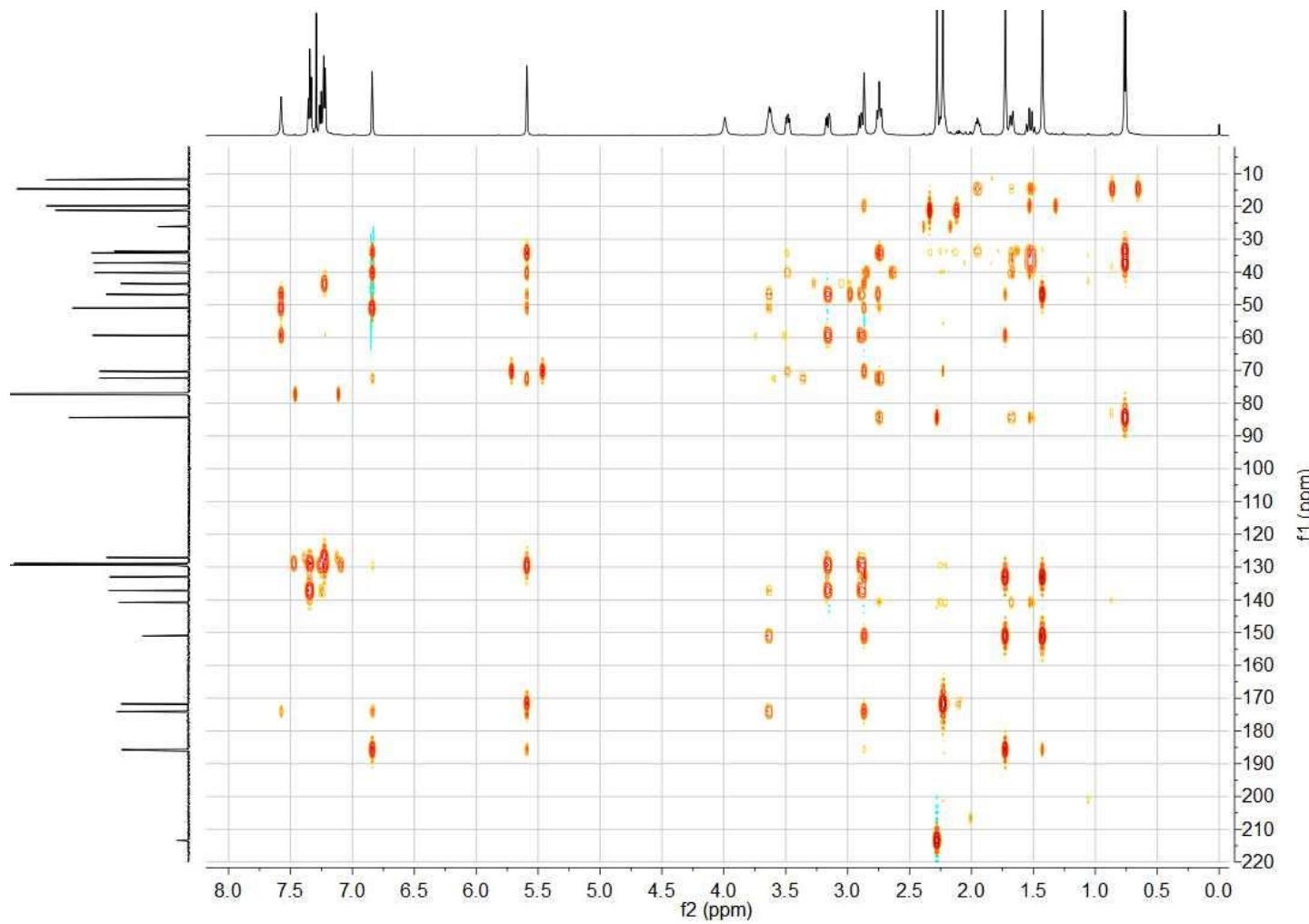
**Fig. S92.**  $^{13}\text{C}$  NMR of **10** ( $\text{CDCl}_3$ , 150 MHz).



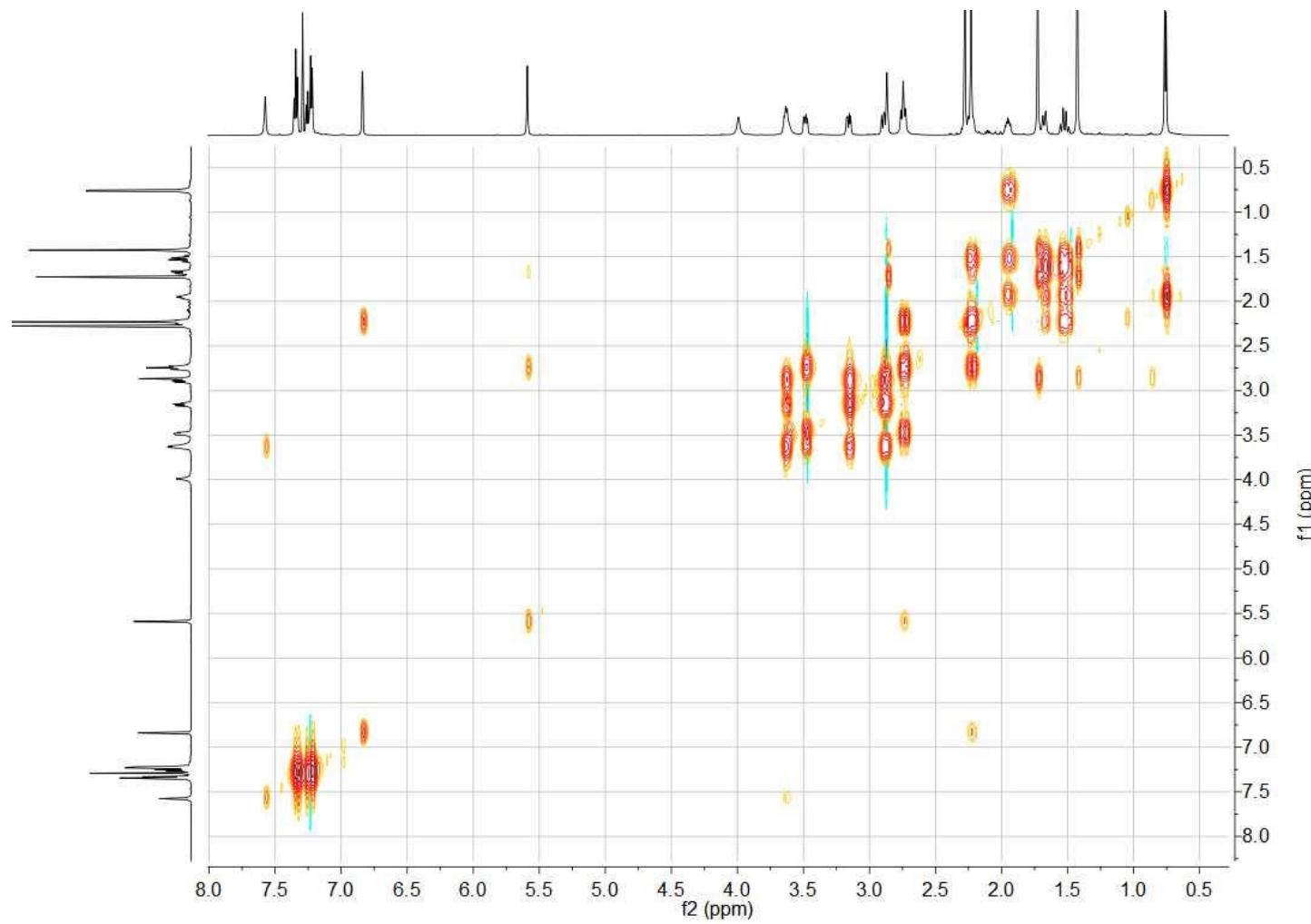
**Fig. S93.** DEPT of **10**.



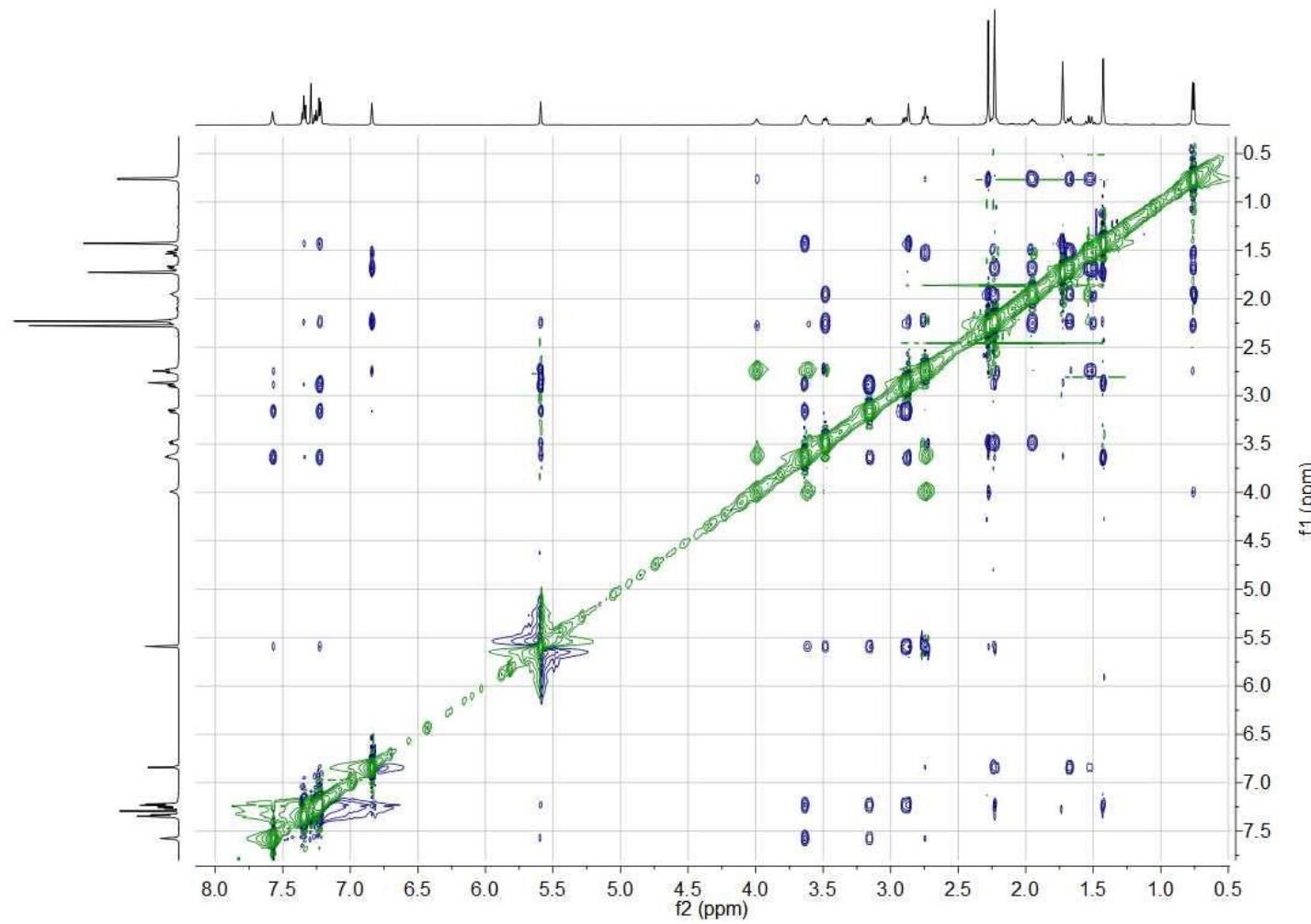
**Fig. S94.** HSQC of **10**.



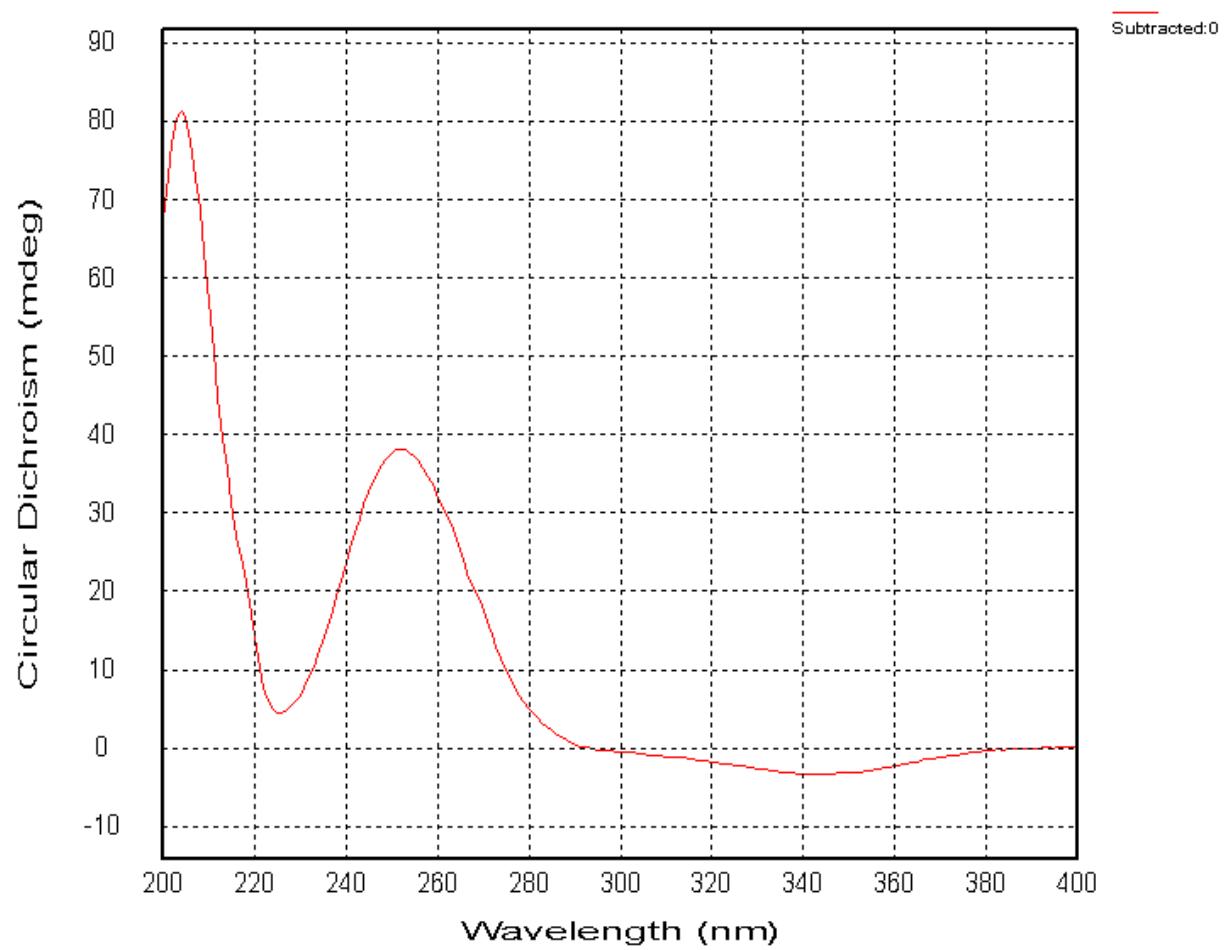
**Fig. S95.** HMBC of **10**.



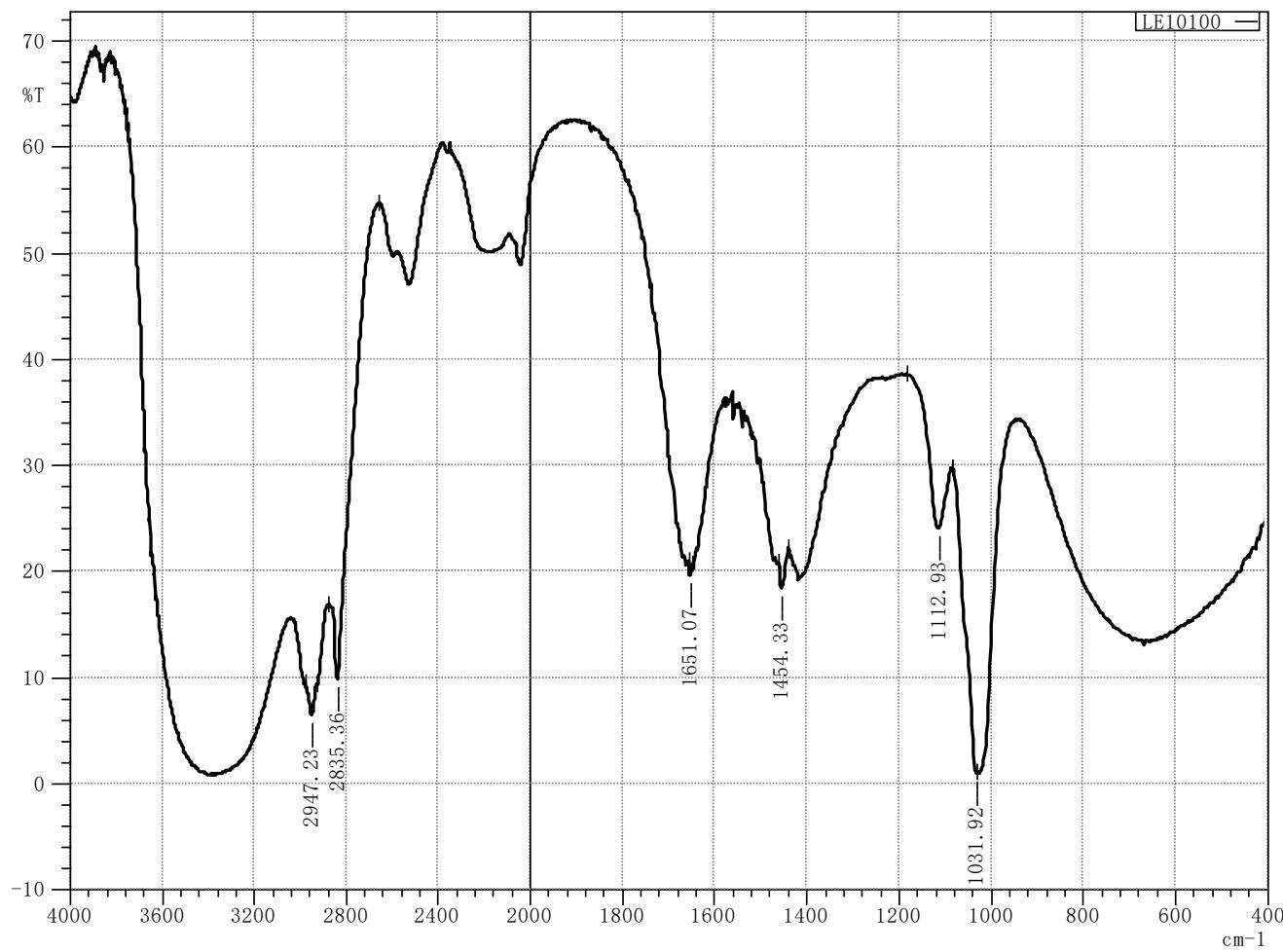
**Fig. S96.**  $^1\text{H}$ - $^1\text{H}$  COSY of **10**.



**Fig. S97.** ROESY of **10**.



**Fig. S98.** Experimental ECD of **10**.

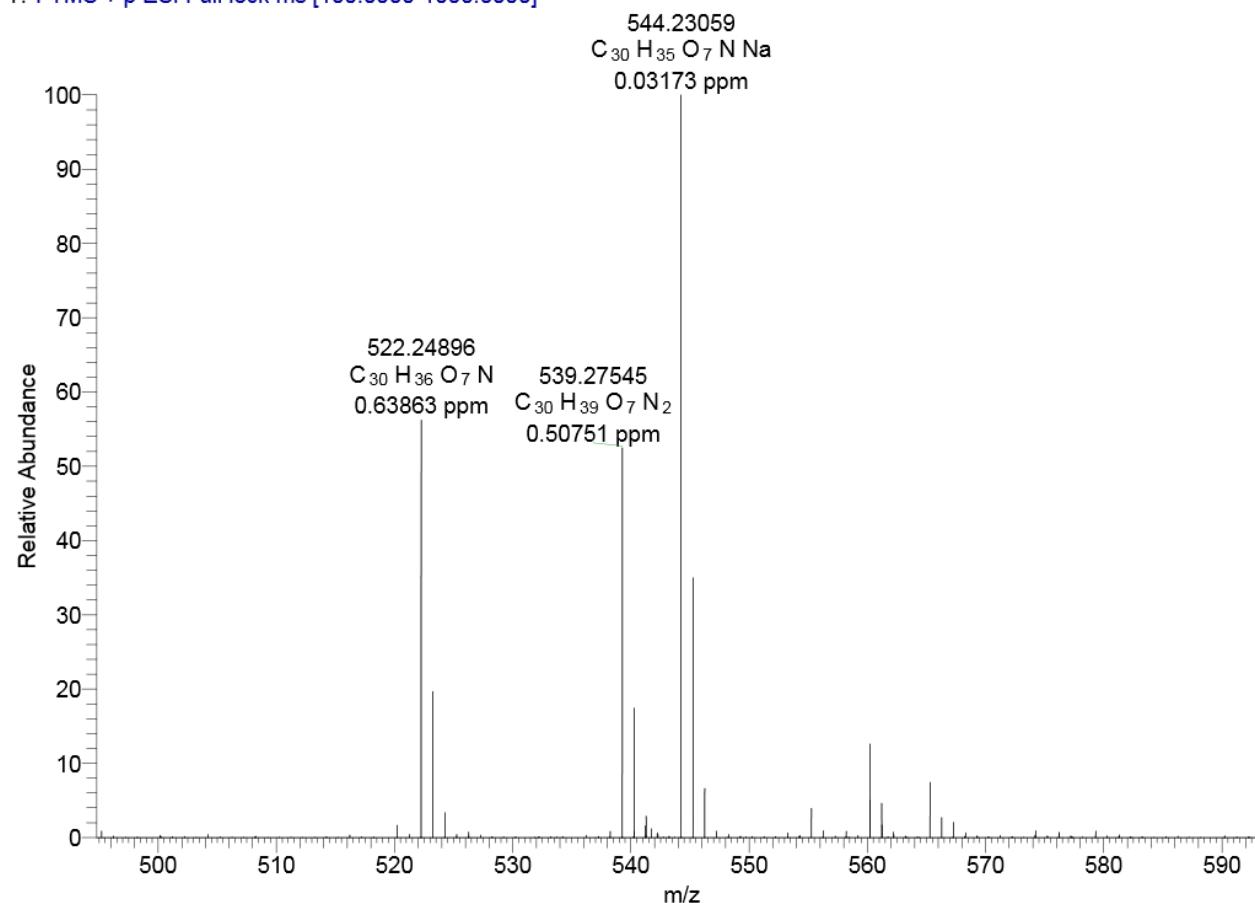


**Fig. S99.** IR of **10**.

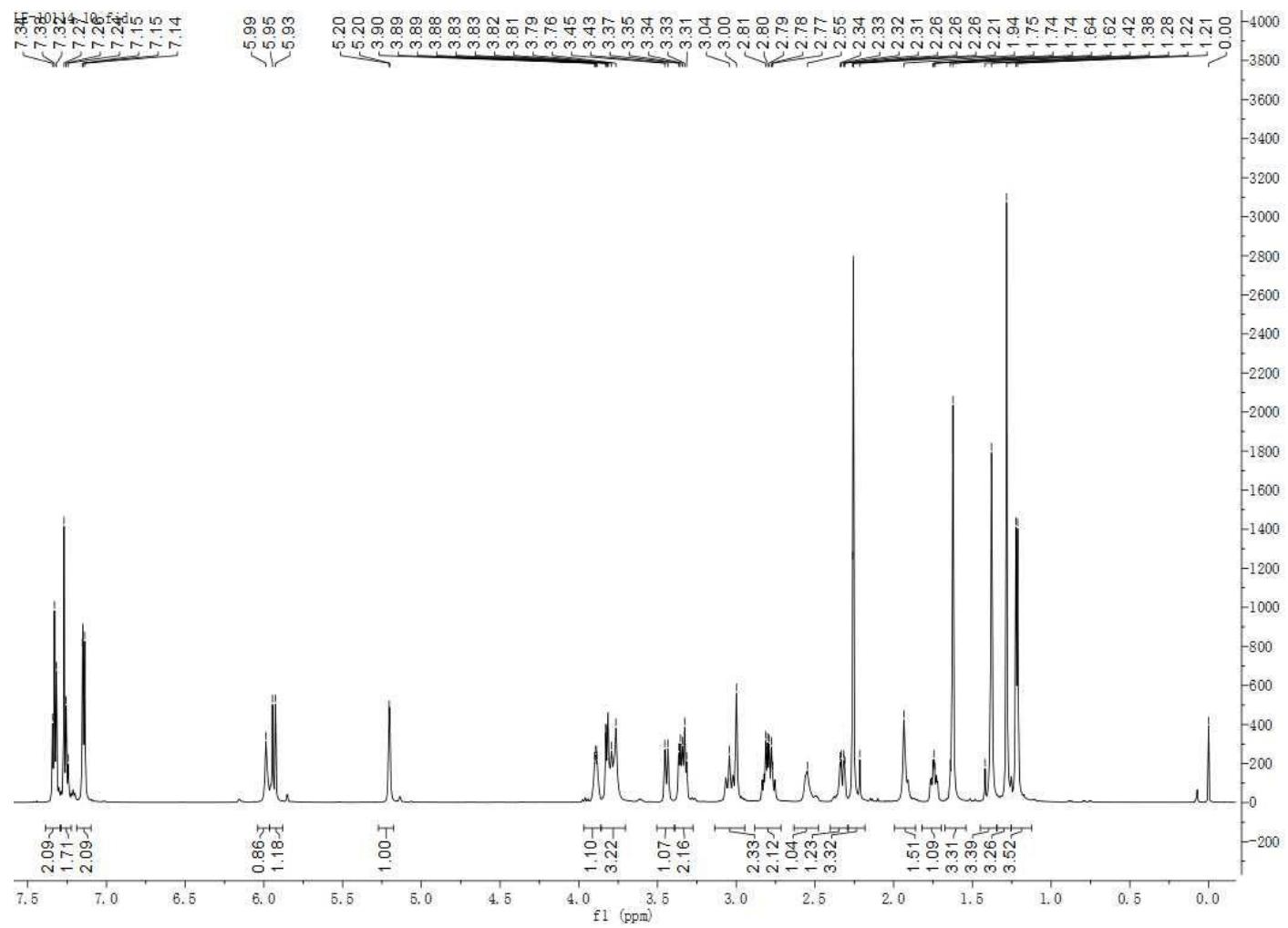
D:\Wen-Xuan\2019\20190604\LE10100

07/02/19 18:46:33

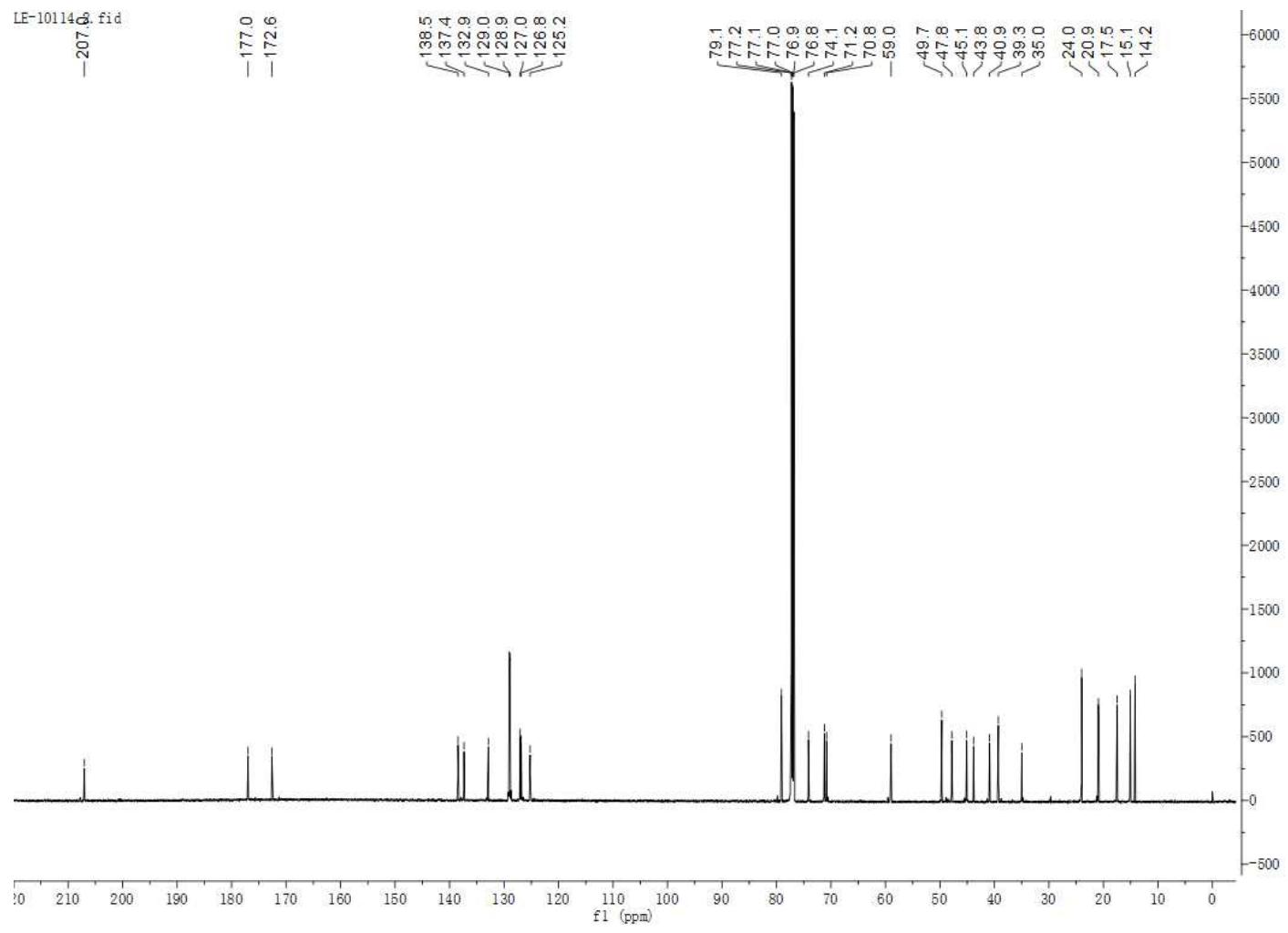
LE10100 #1654 RT: 14.42 AV: 1 NL: 1.79E8  
T: FTMS + p ESI Full lock ms [100.0000-1000.0000]



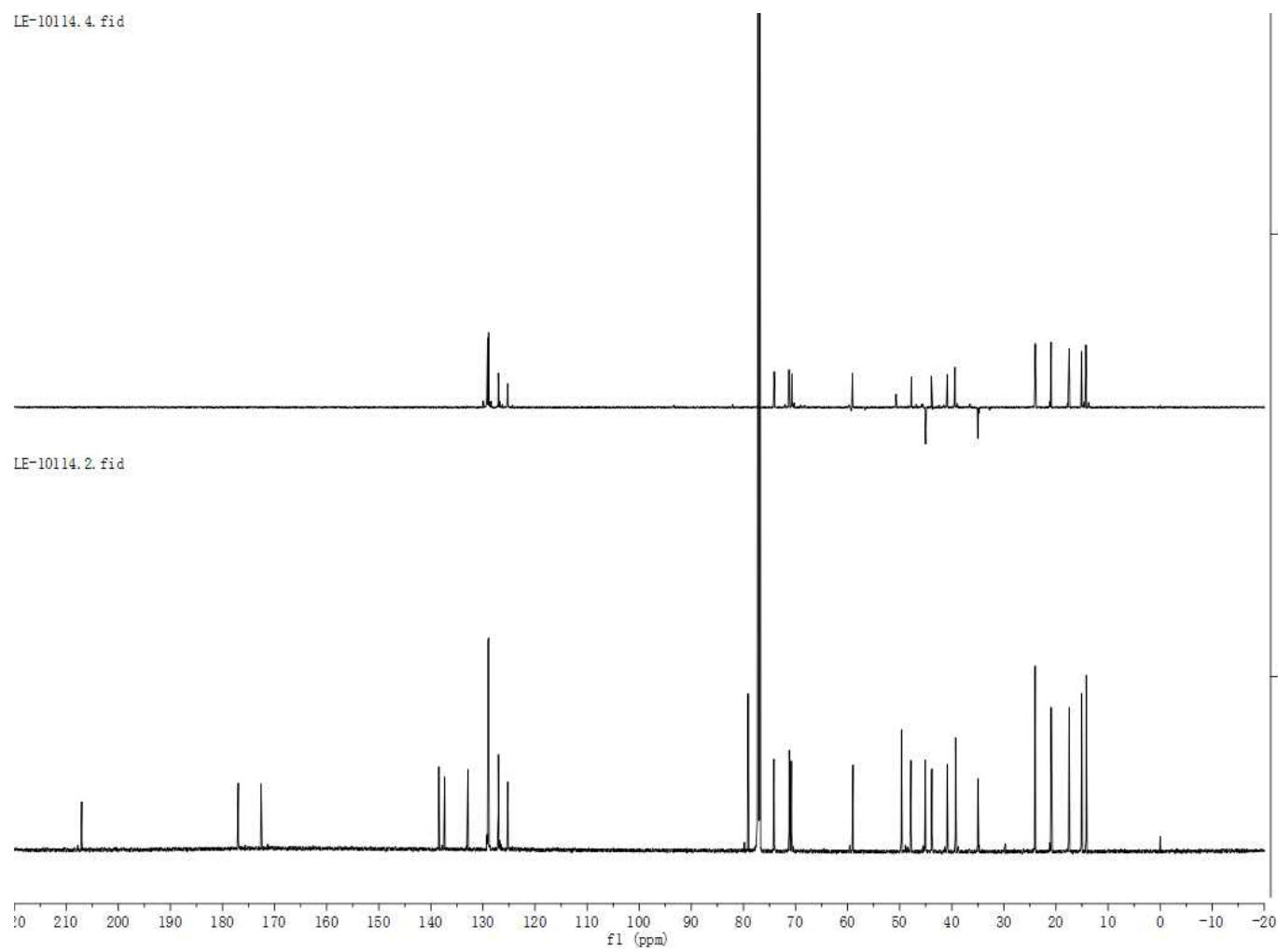
**Fig. S100.** HRESIMS of **10**.



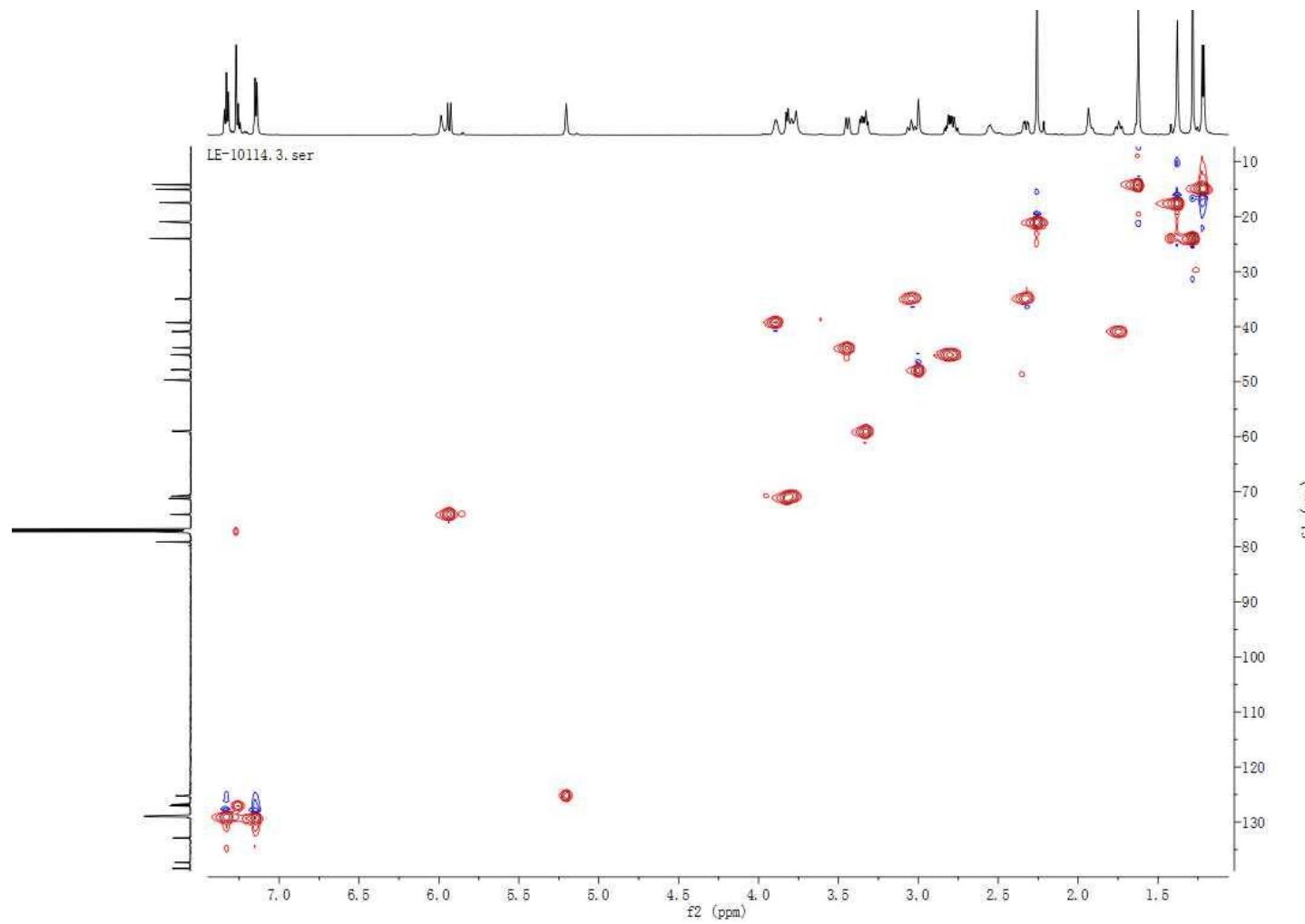
**Fig. S101.**  $^1\text{H}$  NMR of **11** ( $\text{CDCl}_3$ , 600 MHz).



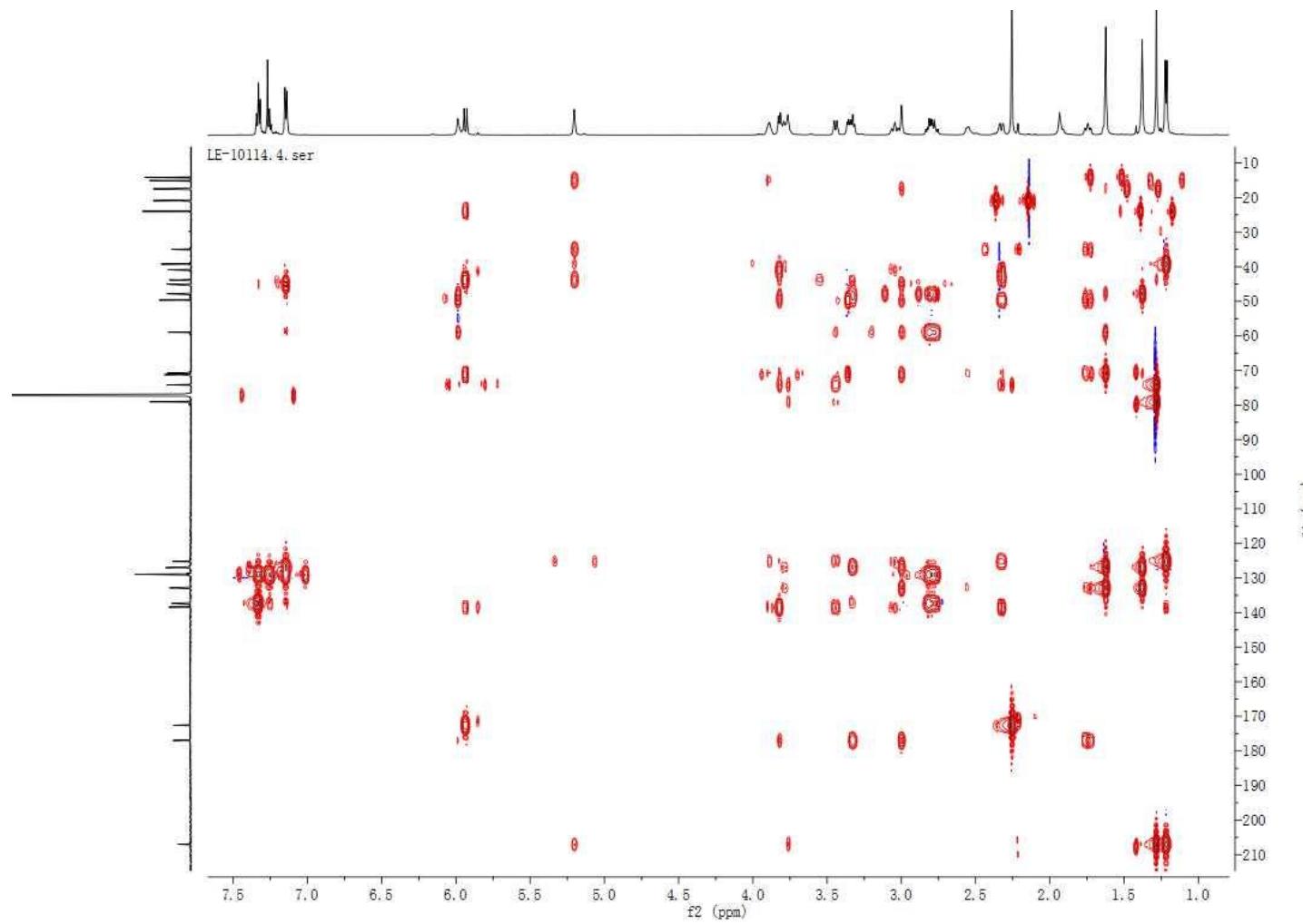
**Fig. S102.**  $^{13}\text{C}$  NMR of **11** ( $\text{CDCl}_3$ , 150 MHz).



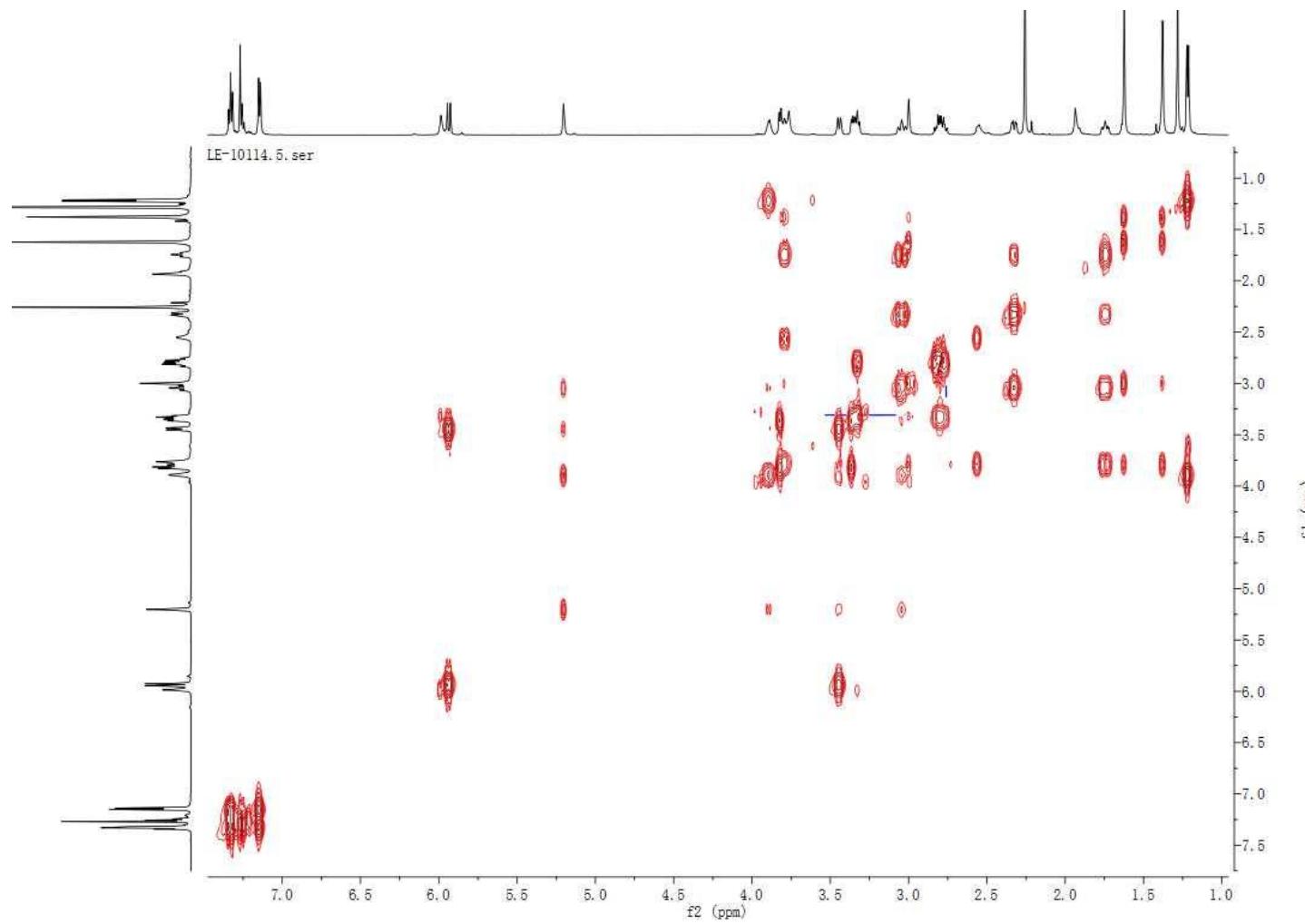
**Fig. S103.** DEPT of 11.



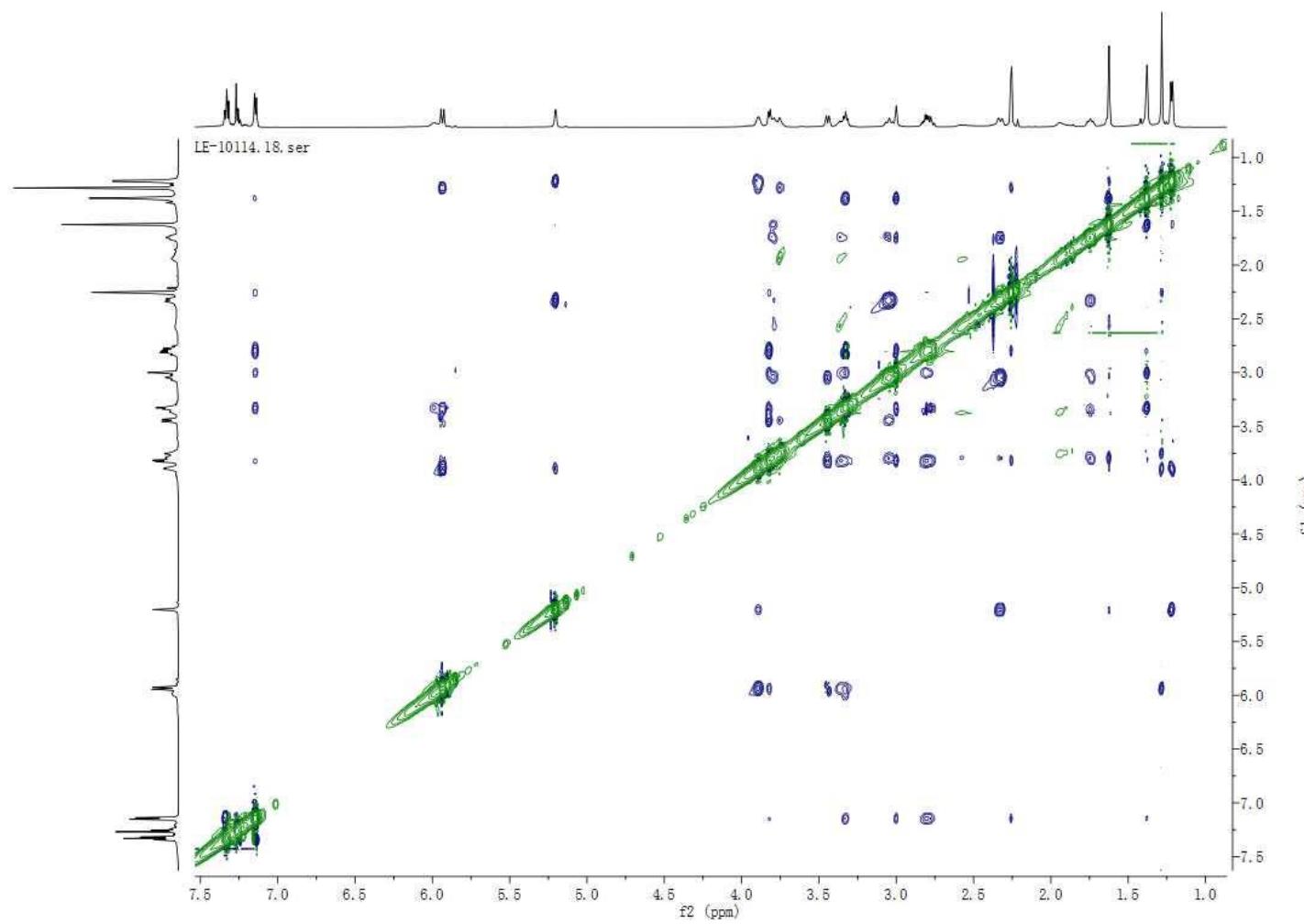
**Fig. S104.** HSQC of **11**.



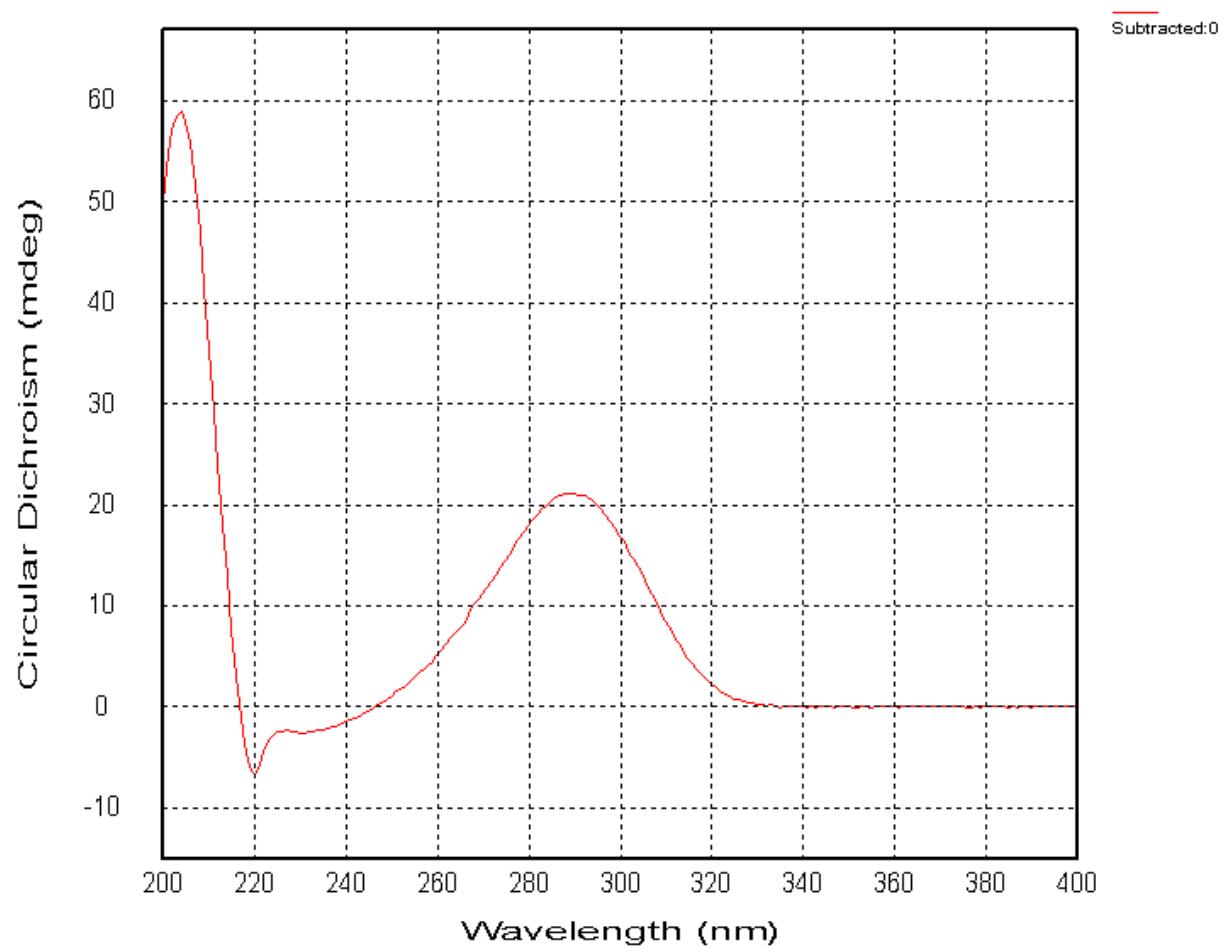
**Fig. S105.** HMBC of **11**.



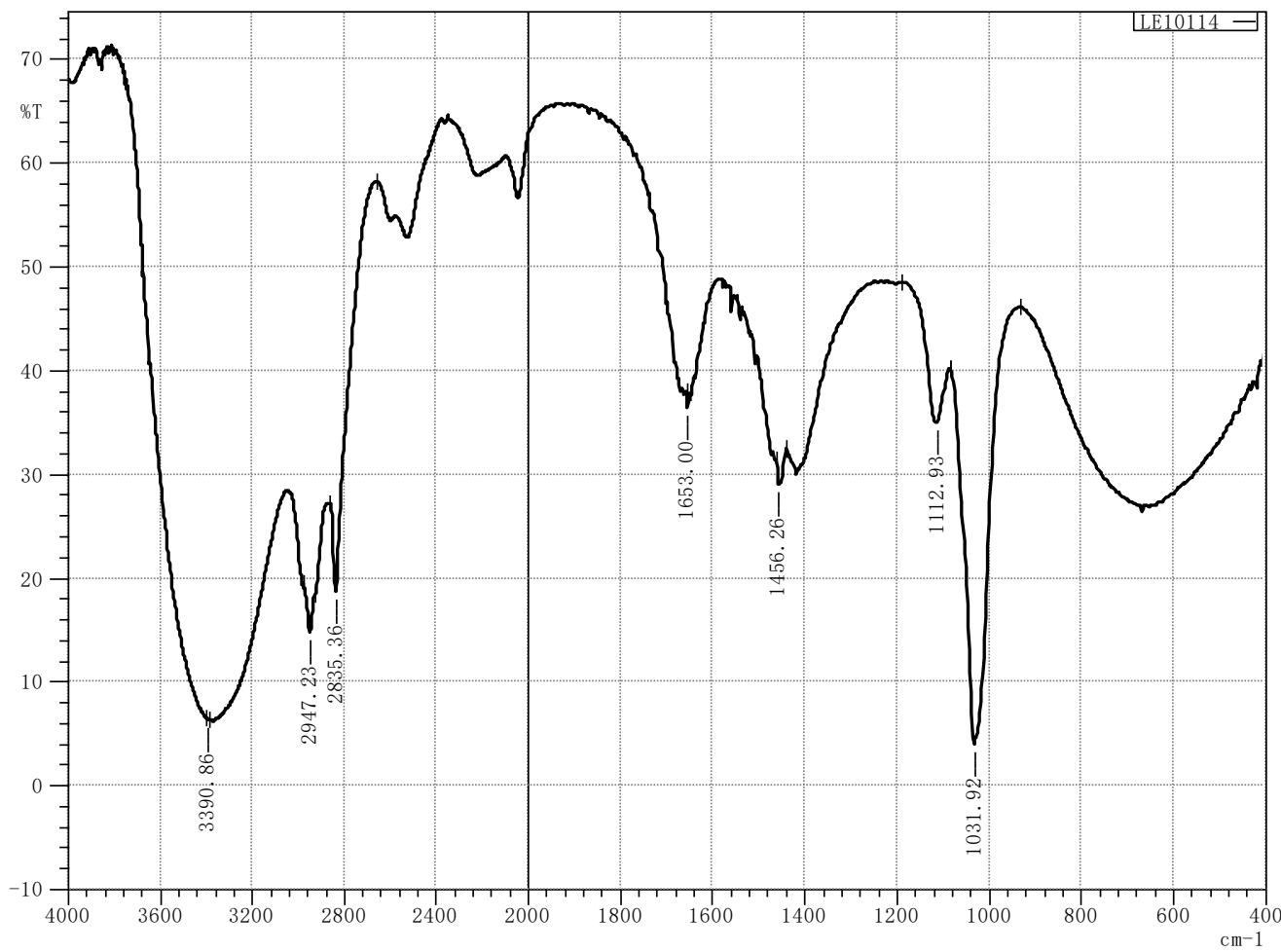
**Fig. S106.**  $^1\text{H}$ - $^1\text{H}$  COSY of **11**.



**Fig. S107.** ROESY of **11**.



**Fig. S108.** Experimental ECD of **11**.

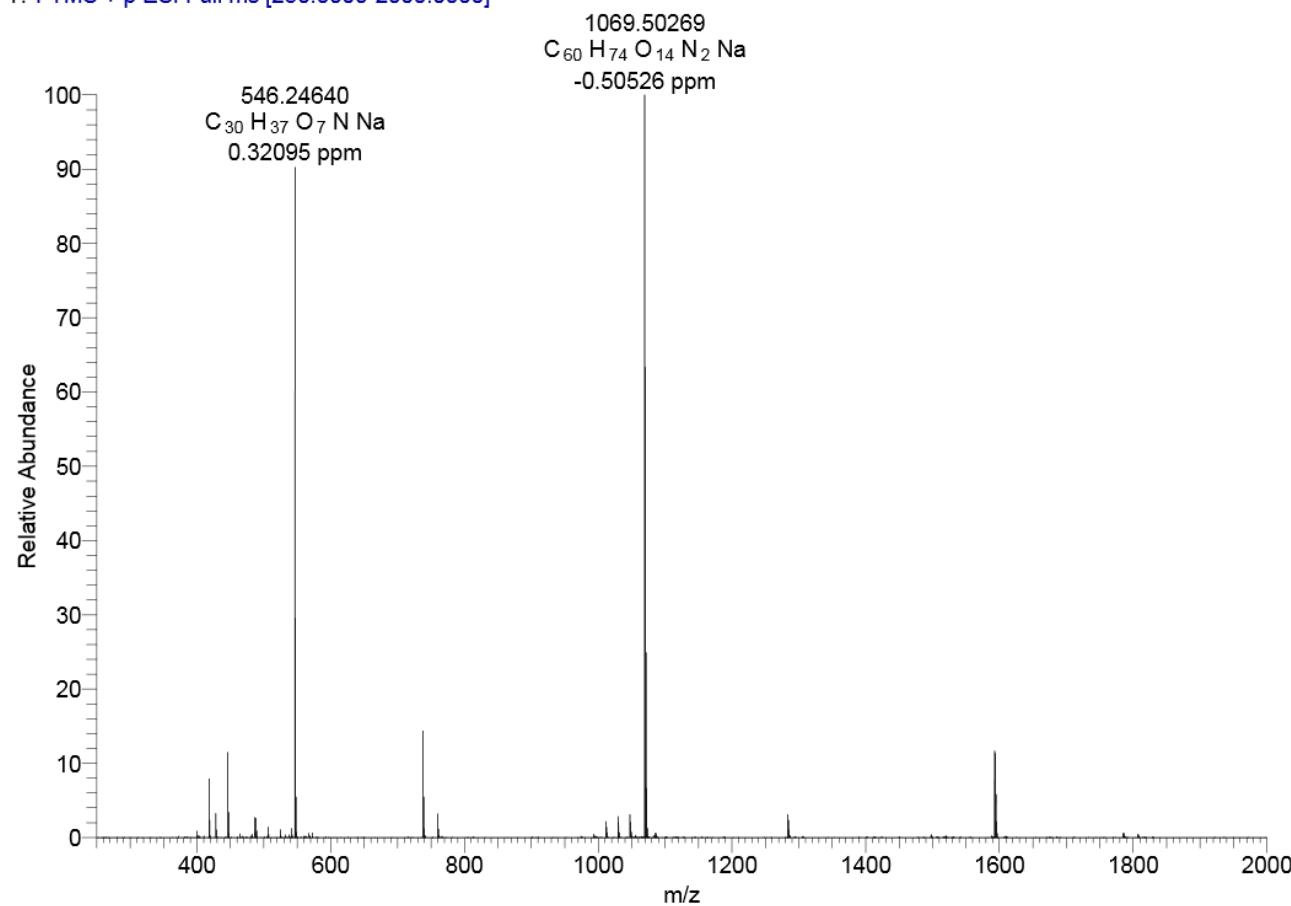


**Fig. S109.** IR of **11**.

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03/22/19 11:14:03

LE10114 #1645 RT: 14.35 AV: 1 NL: 2.08E9  
T: FTMS + p ESI Full ms [250.0000-2000.0000]



**Fig. S110.** HRESIMS of **11**.