

## Highly efficient macrolactonization of $\omega$ -hydroxy acids using benzotriazole esters: Synthesis of Sansalvamide A

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## Supporting Information

### 1. NMR Spectra

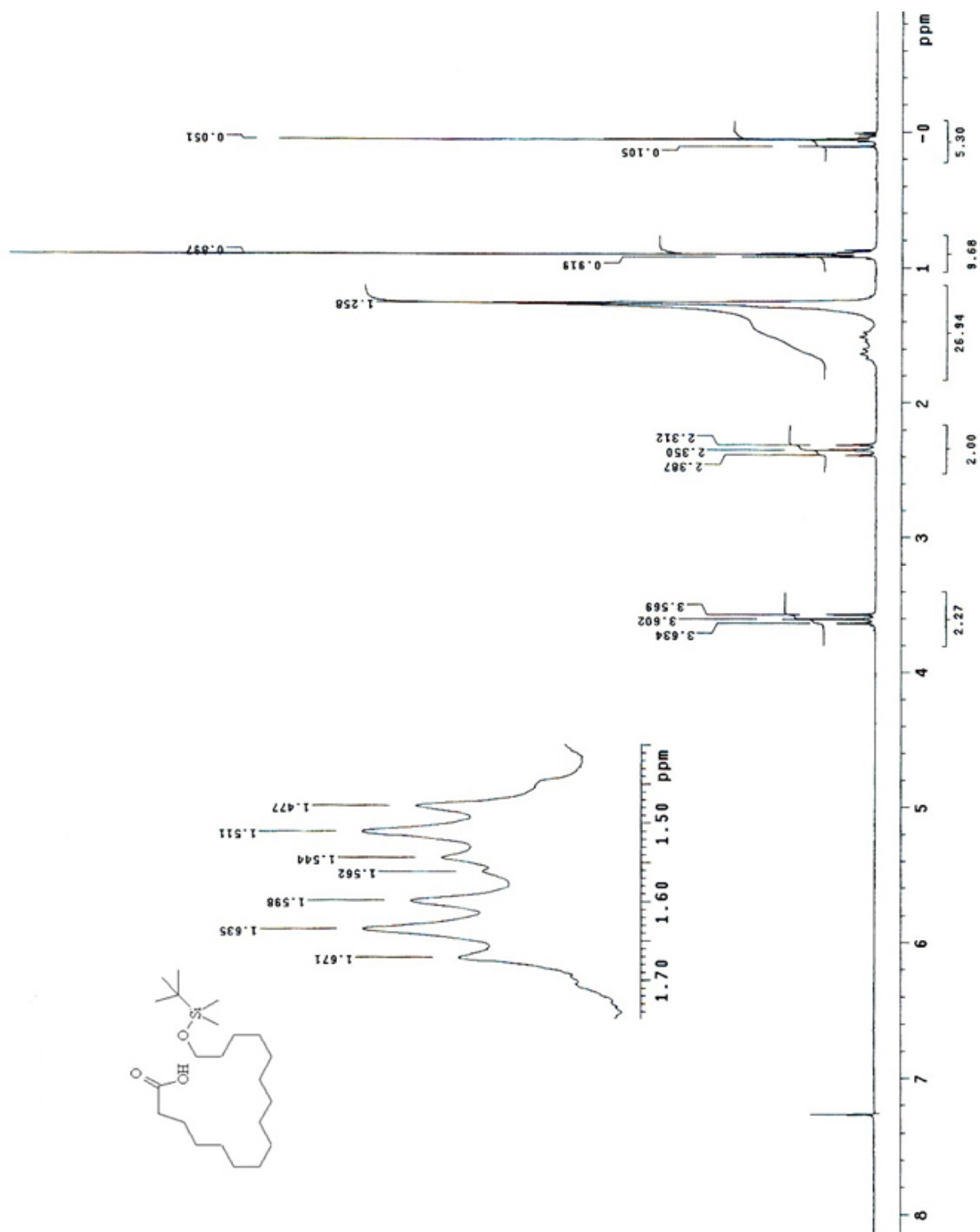


Figure S1.  $^1\text{H}$  NMR in  $\text{CDCl}_3$

Supporting Information

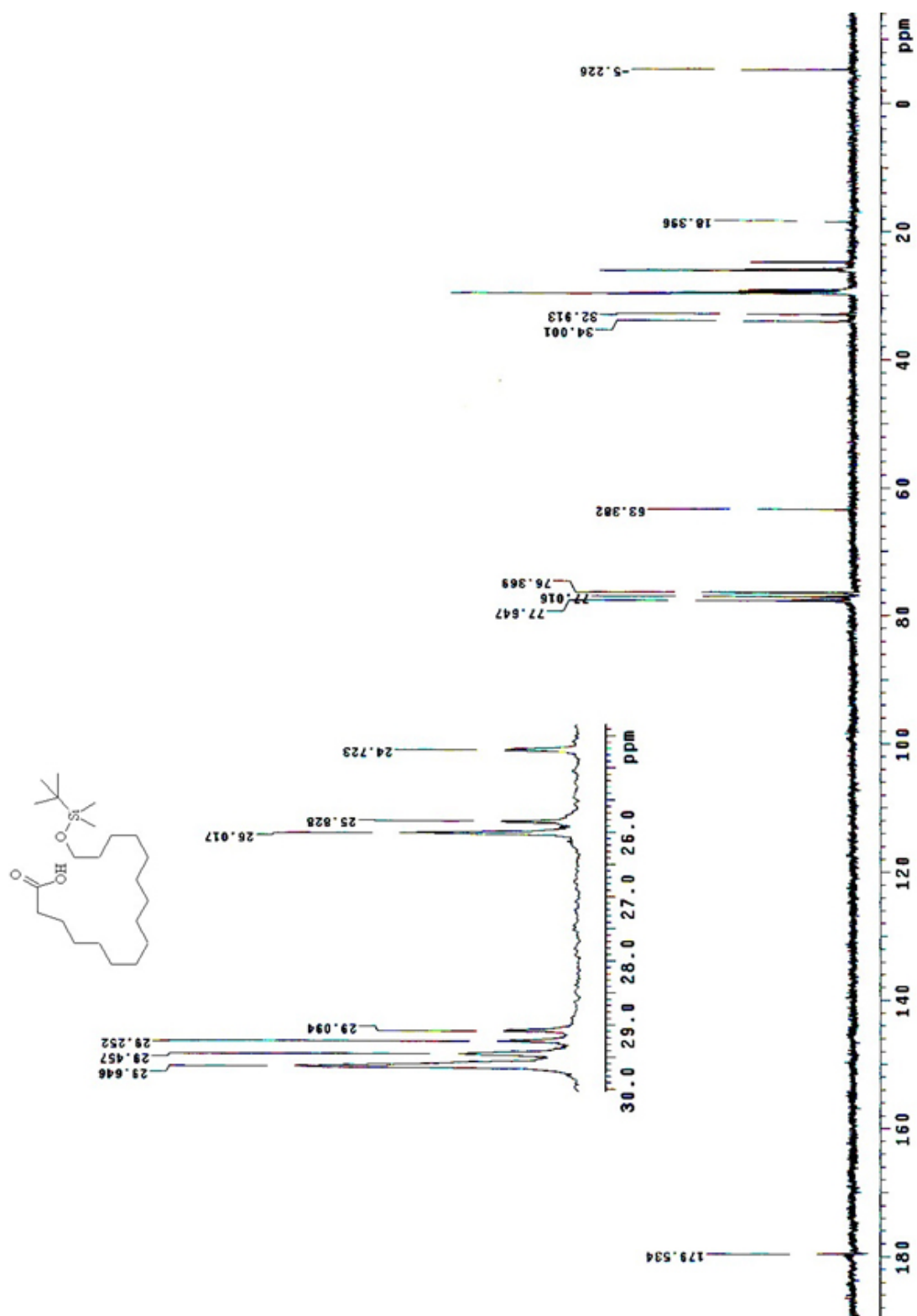


Figure S2.  $^{13}\text{C}$  NMR in  $\text{CDCl}_3$

Supporting Information

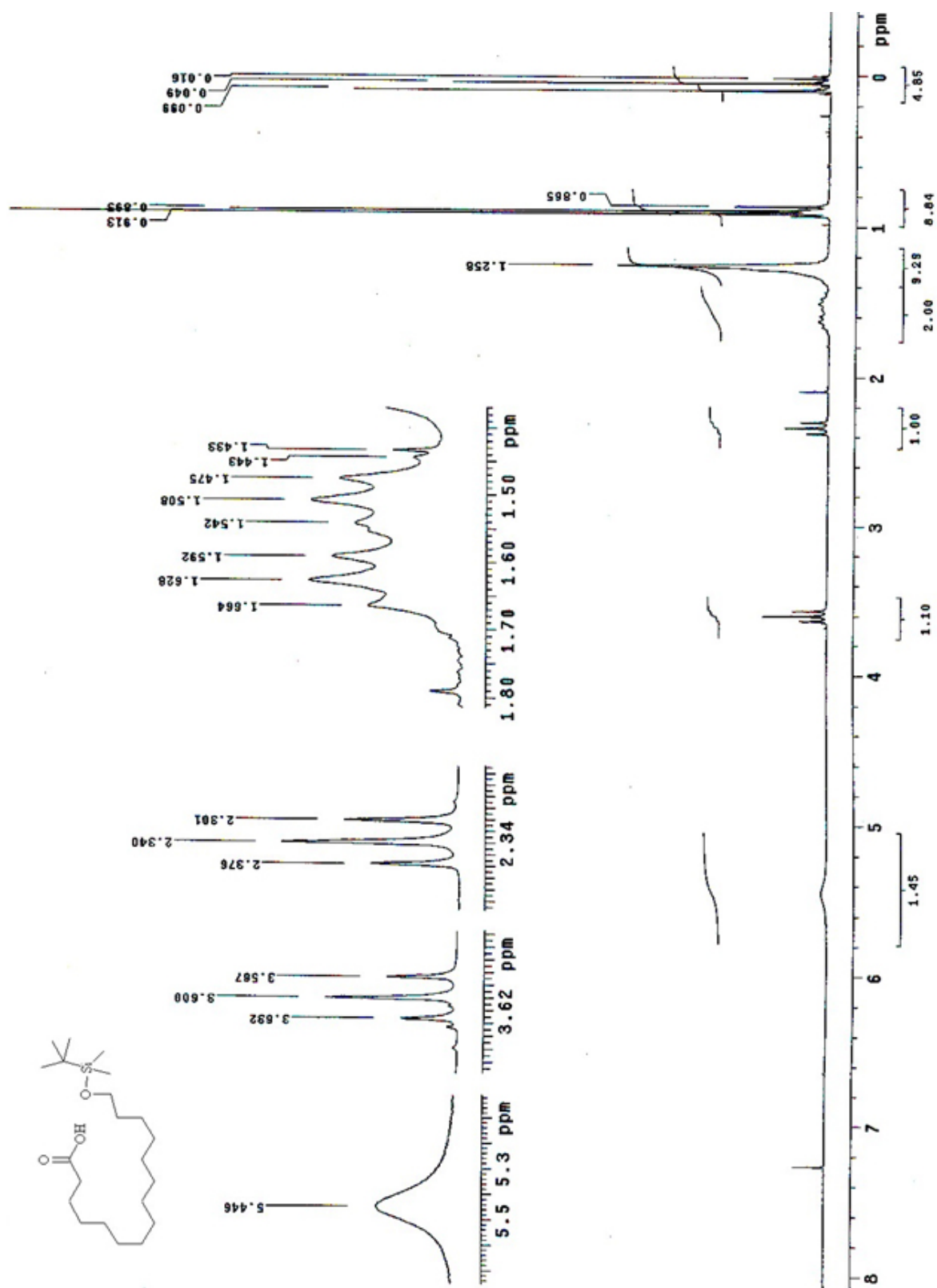


Figure S3.  $^1\text{H}$  NMR in  $\text{CDCl}_3$

Supporting Information

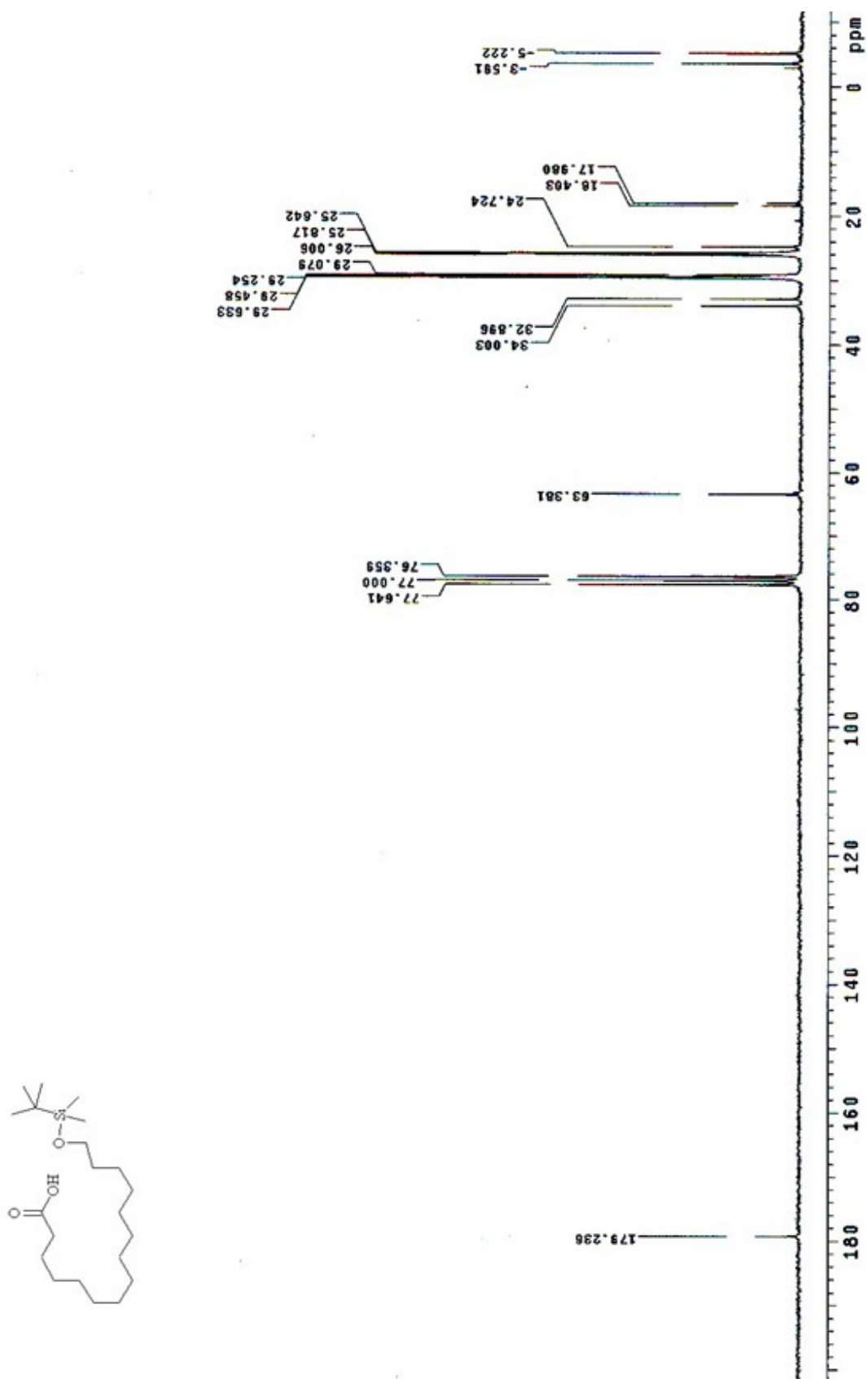


Figure S4. <sup>13</sup>C NMR CDCl<sub>3</sub>

Supporting Information

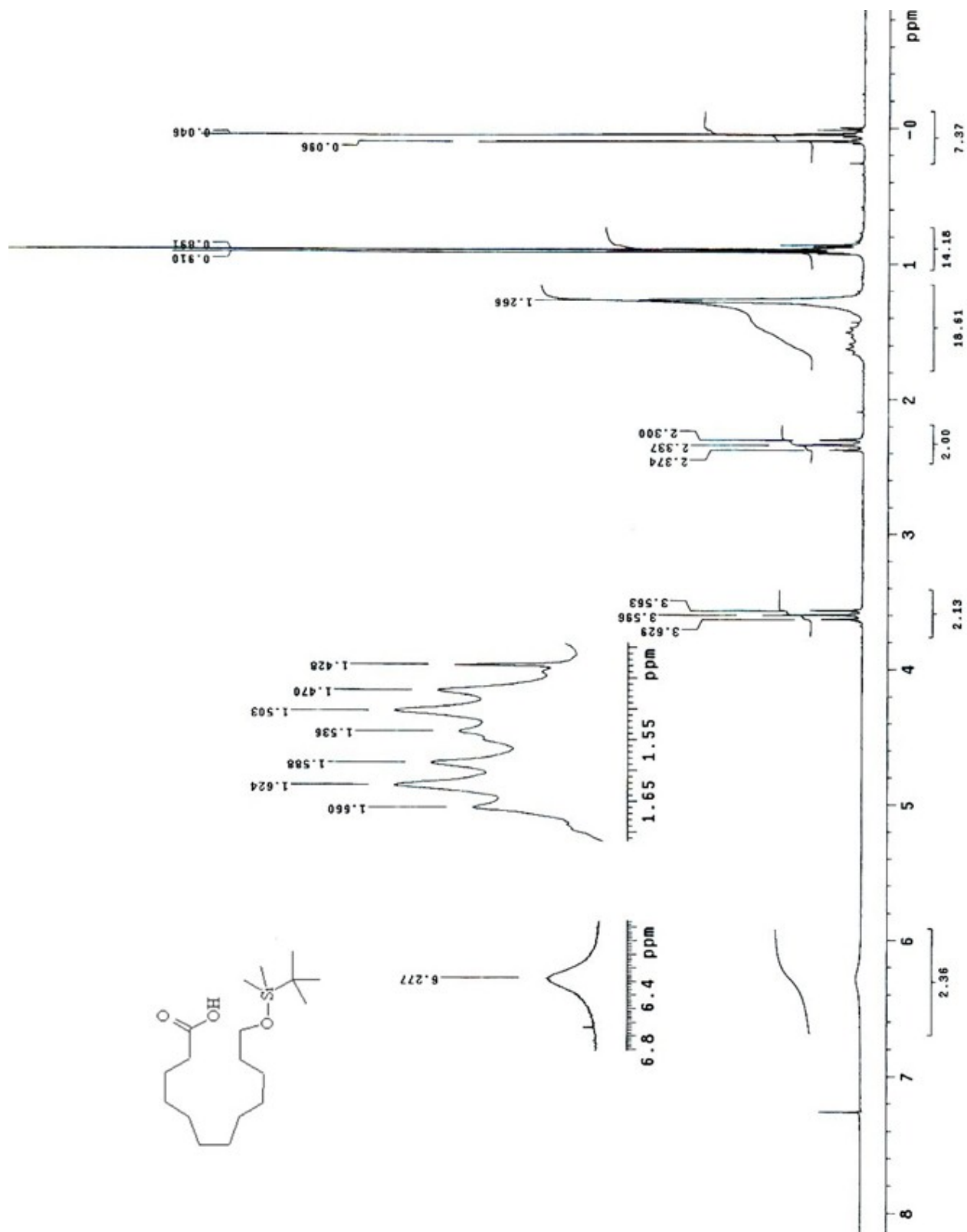


Figure S5.  $^1\text{H}$  NMR in  $\text{CDCl}_3$

Supporting Information

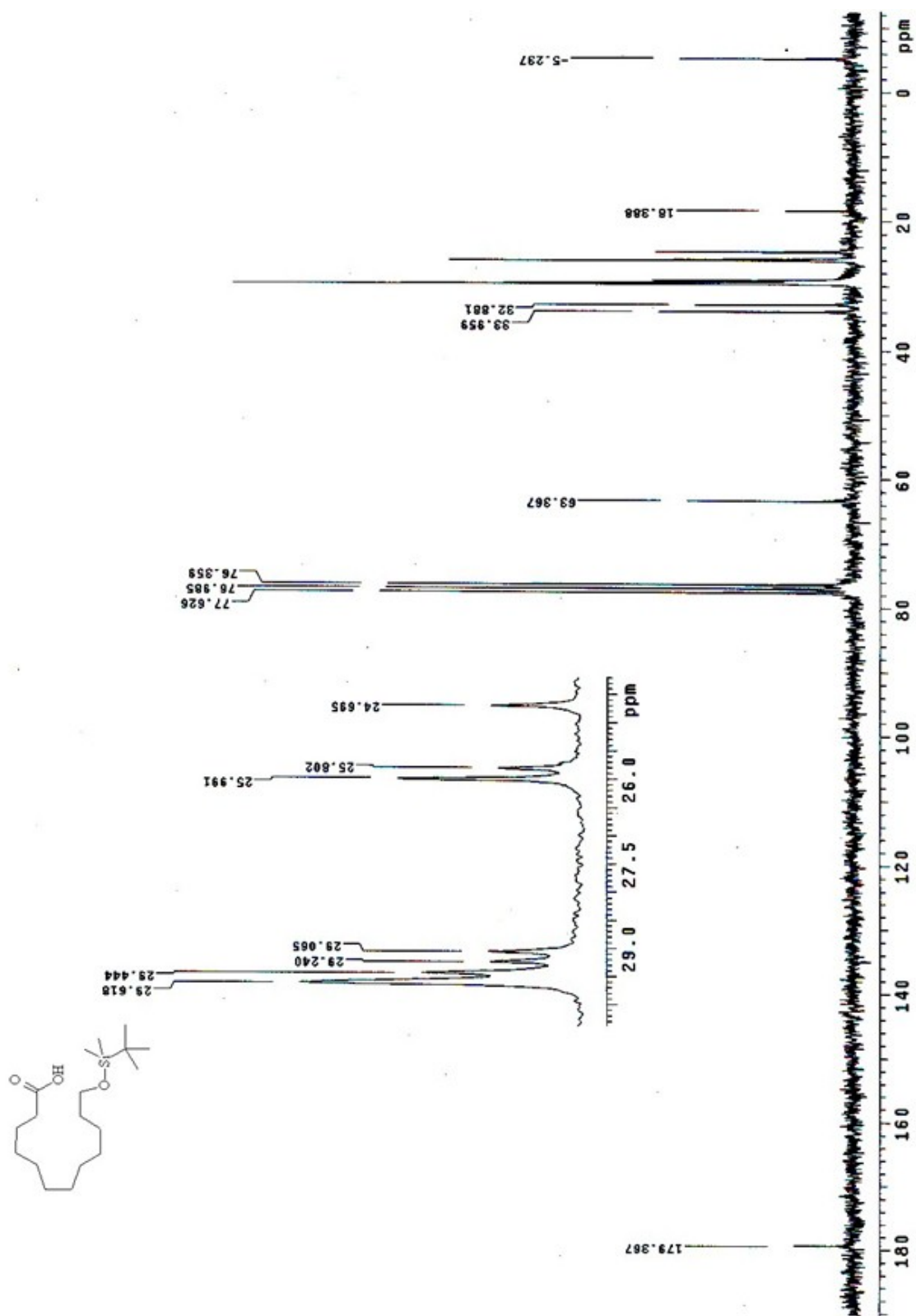


Figure S6.  $^{13}\text{C}$  NMR in  $\text{CDCl}_3$

Supporting Information

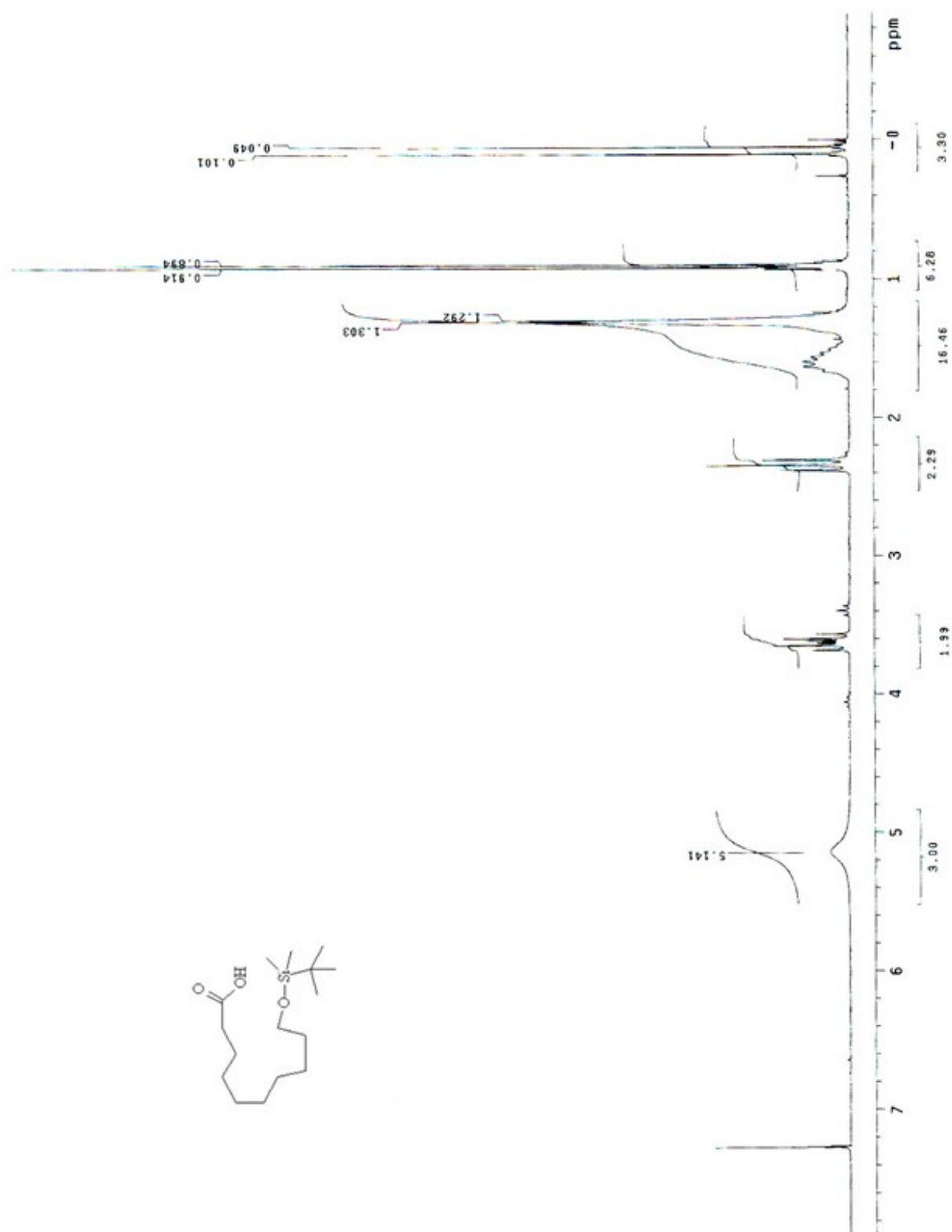


Figure S7. <sup>1</sup>H NMR in CDCl<sub>3</sub>



Supporting Information

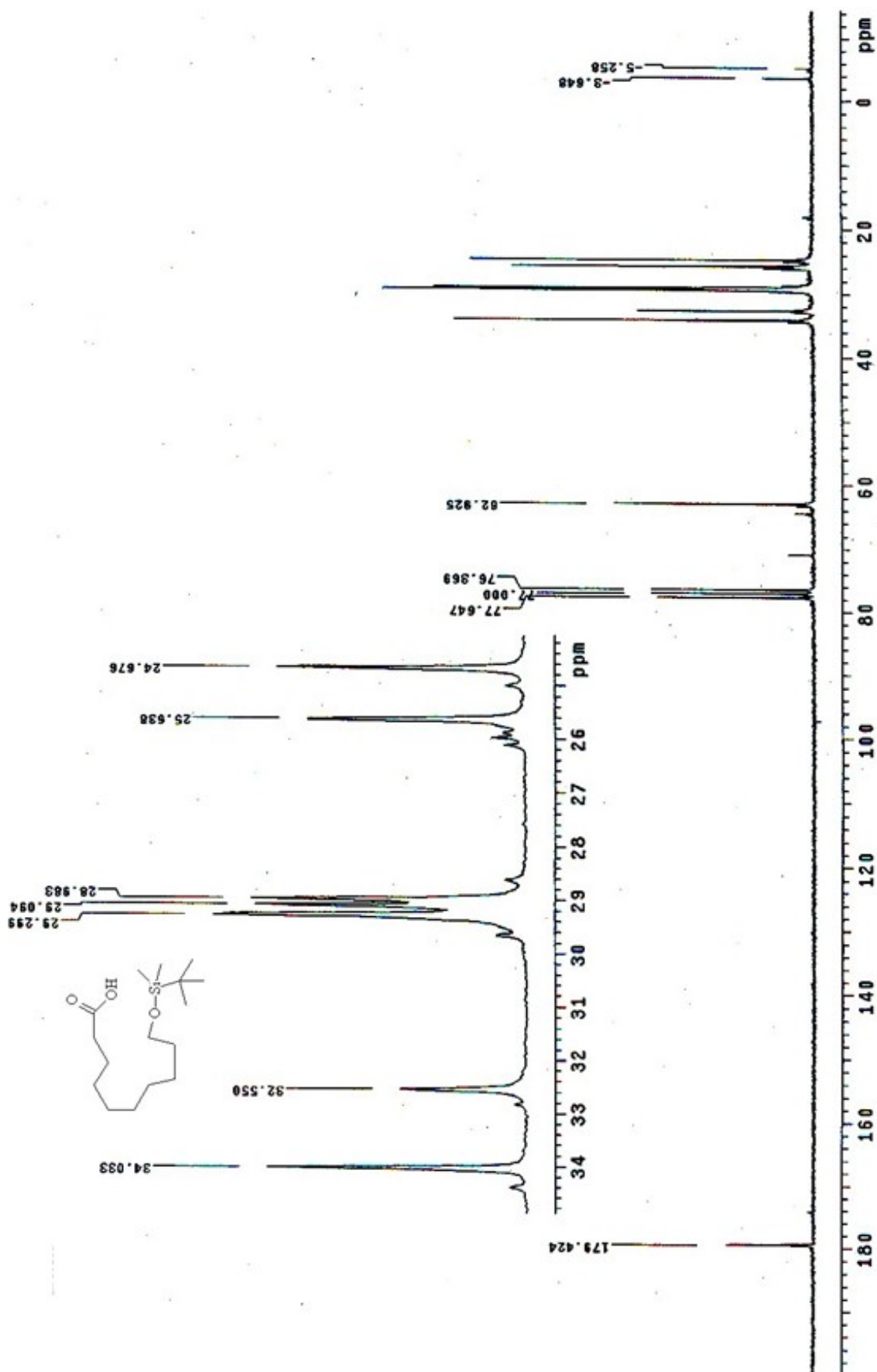


Figure S8.  $^{13}\text{C}$  NMR in  $\text{CDCl}_3$

Supporting Information

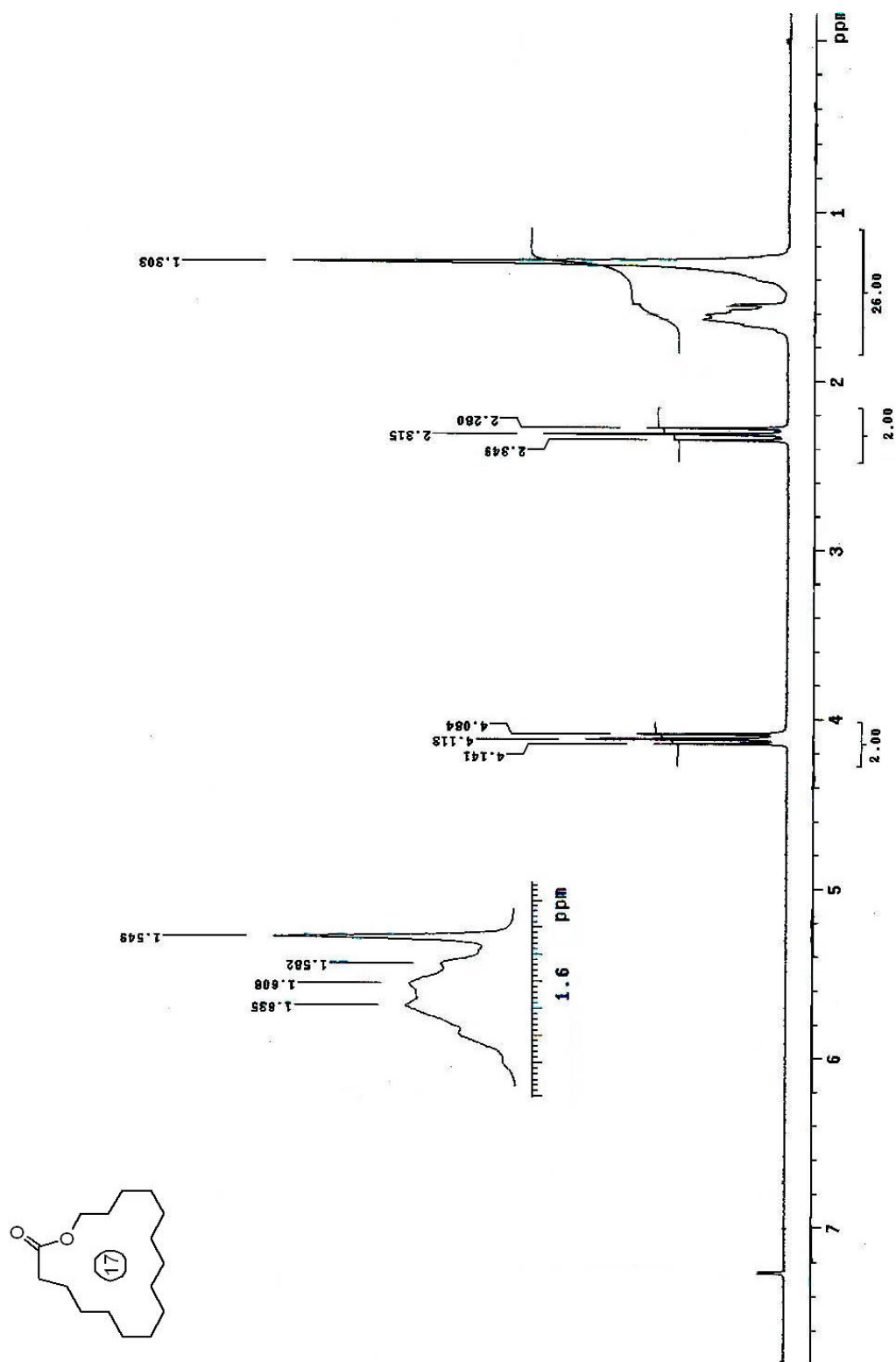


Figure S9. <sup>1</sup>H NMR CDCl<sub>3</sub>

Supporting Information

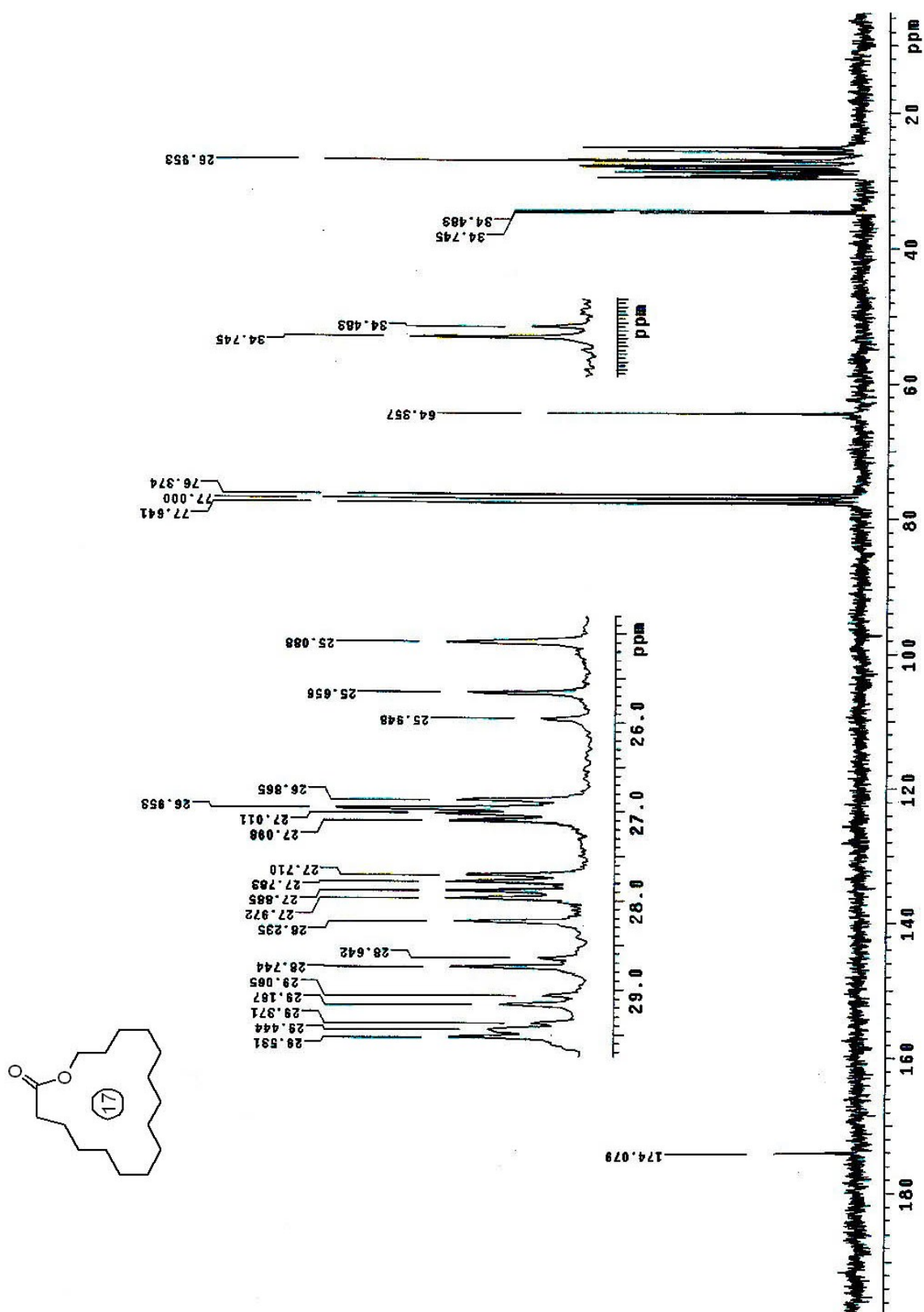
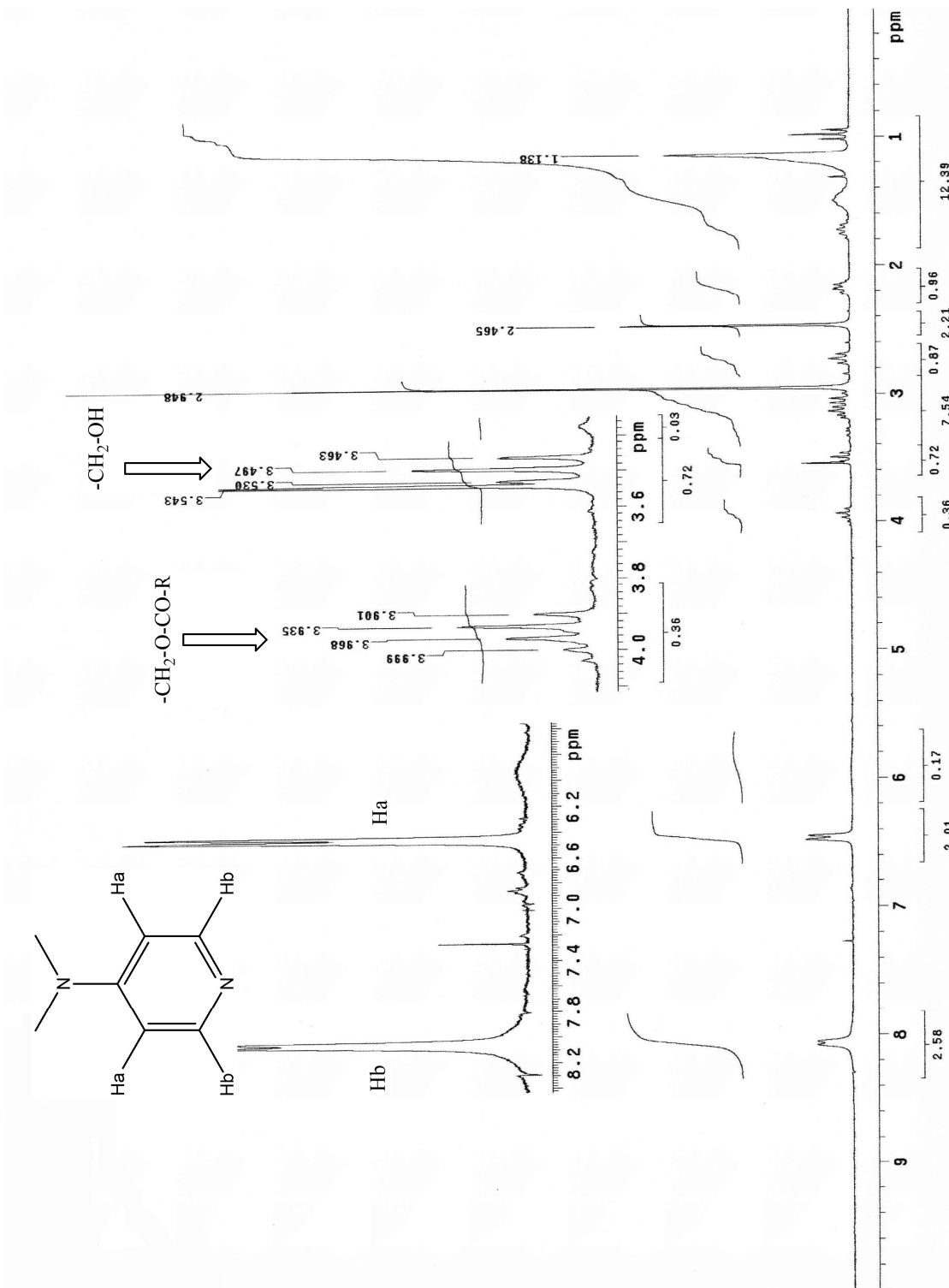


Figure S10.  $^{13}\text{C}$  NMR in  $\text{CDCl}_3$

Supporting Information



**Figure S11.**  $^1\text{H}$  NMR  $\text{CDCl}_3$  of the crude reaction with 16-hydroxyhexadecanoic acid (1 equiv), EDC (1 equiv) and DMAP (2 equiv).

Supporting Information

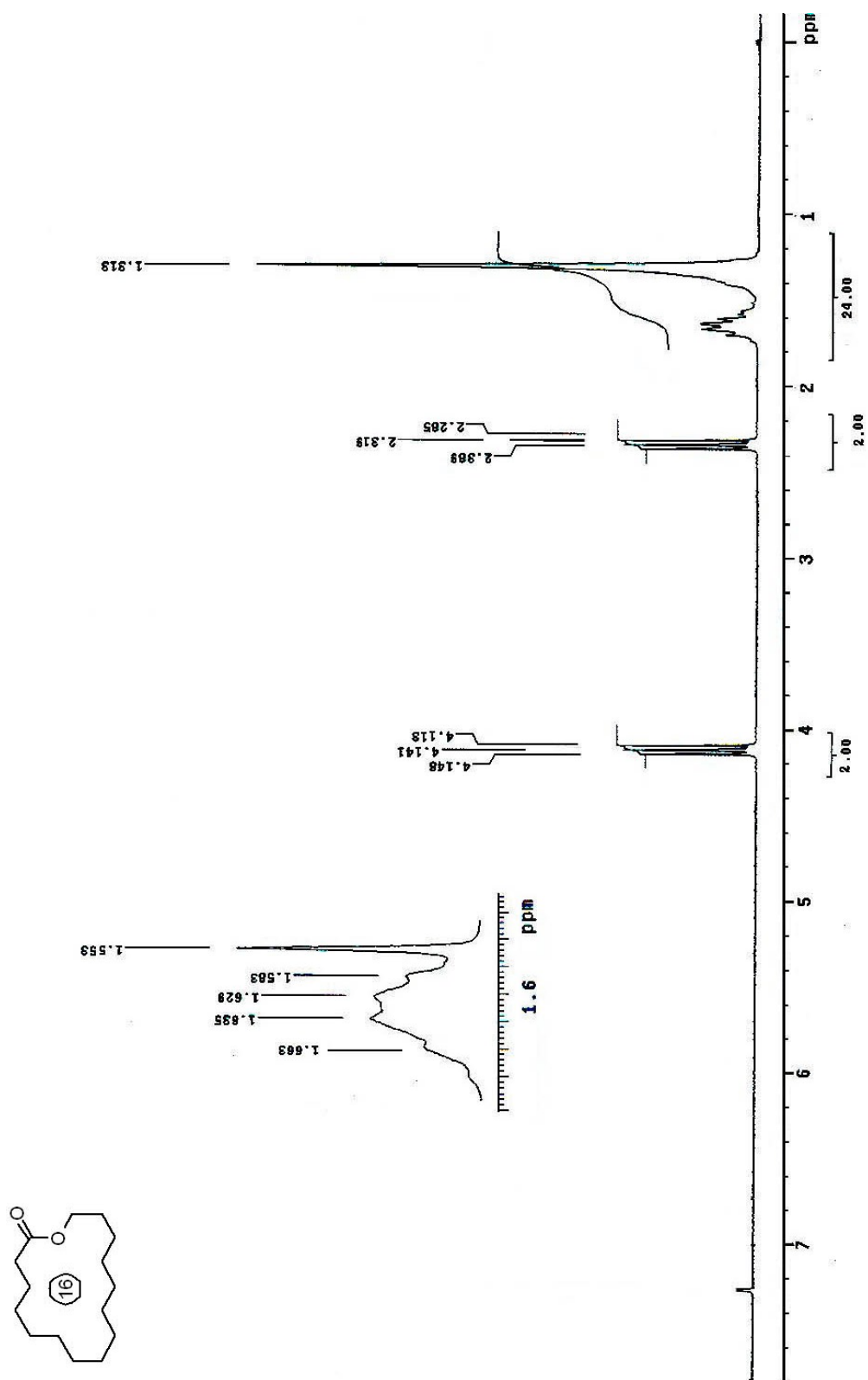


Figure S12.  $^1\text{H}$  NMR in  $\text{CDCl}_3$

Supporting Information

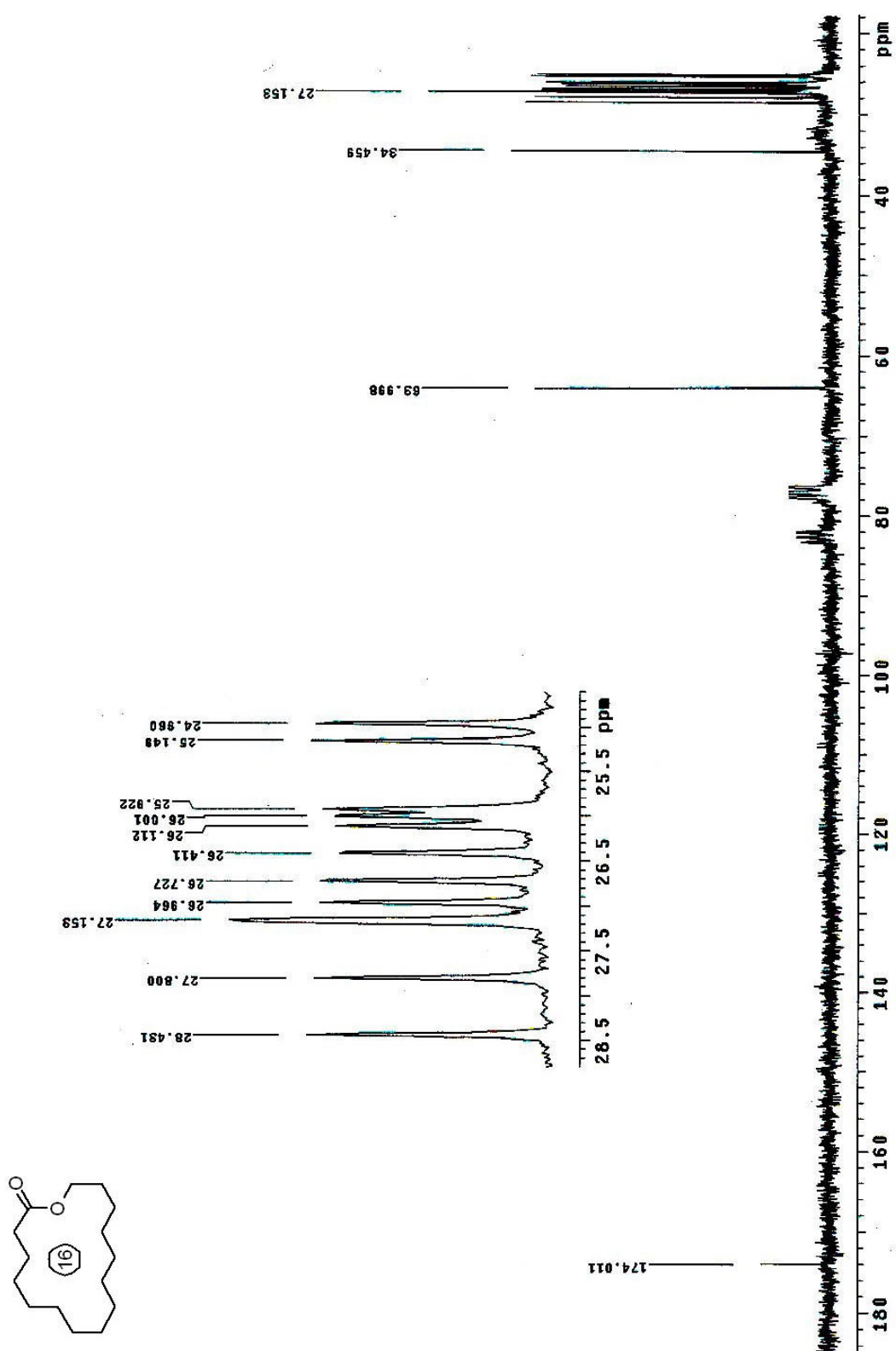


Figure S13.  $^{13}\text{C}$  NMR in  $\text{CDCl}_3$

Supporting Information

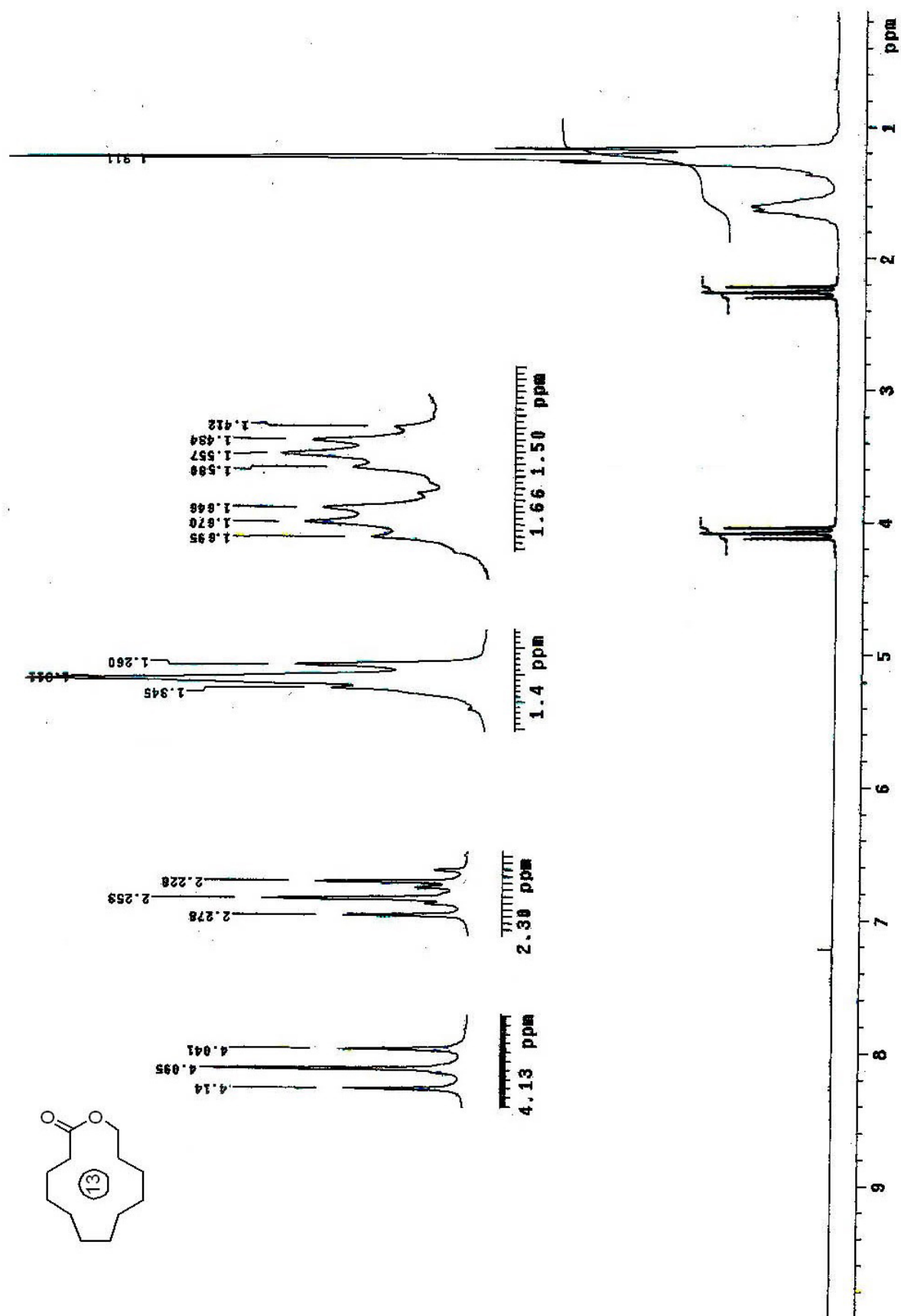


Figure S14.  $^1\text{H}$  NMR in  $\text{CDCl}_3$

Supporting Information

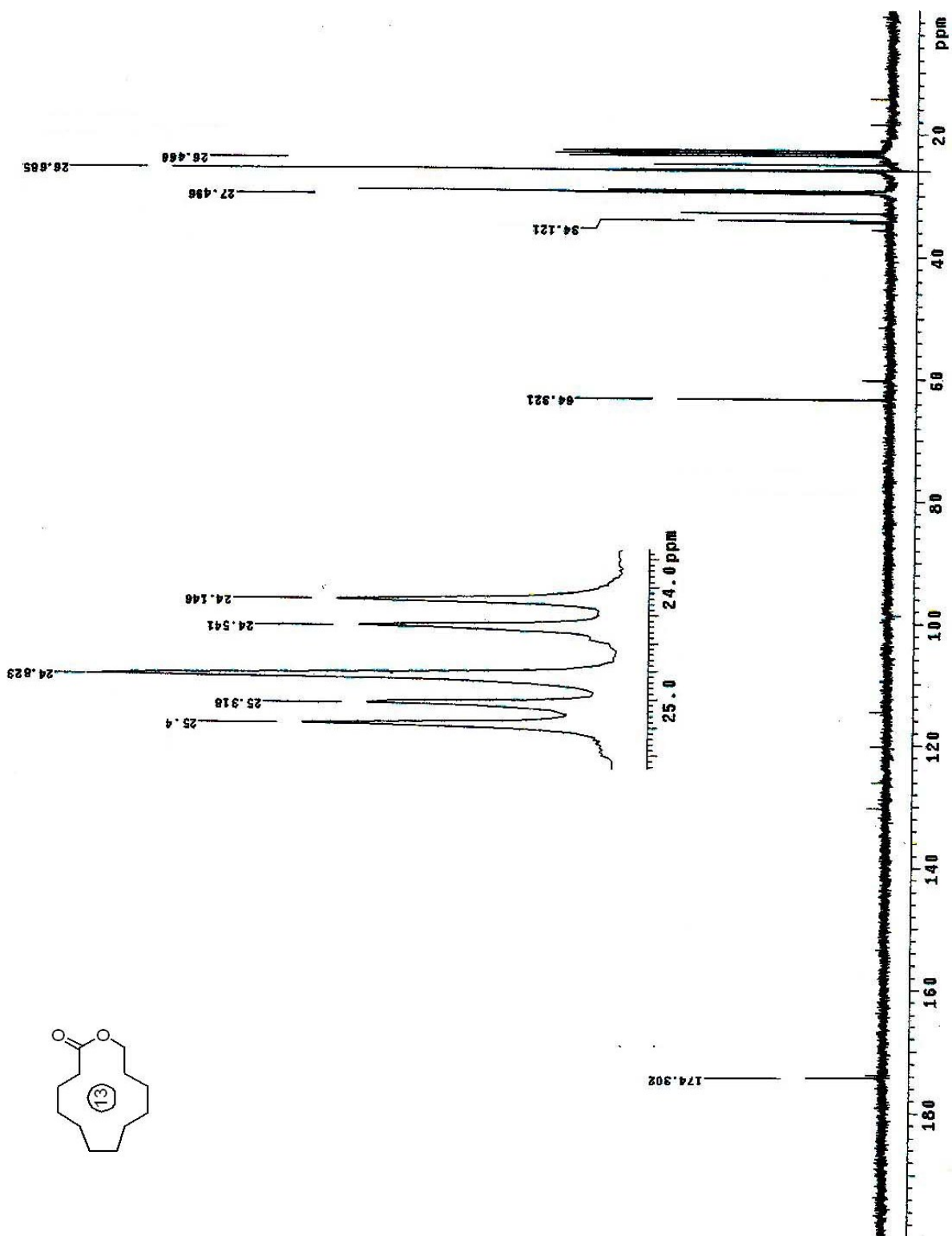


Figure S15.  $^{13}\text{C}$  NMR in  $\text{CDCl}_3$



Supporting Information

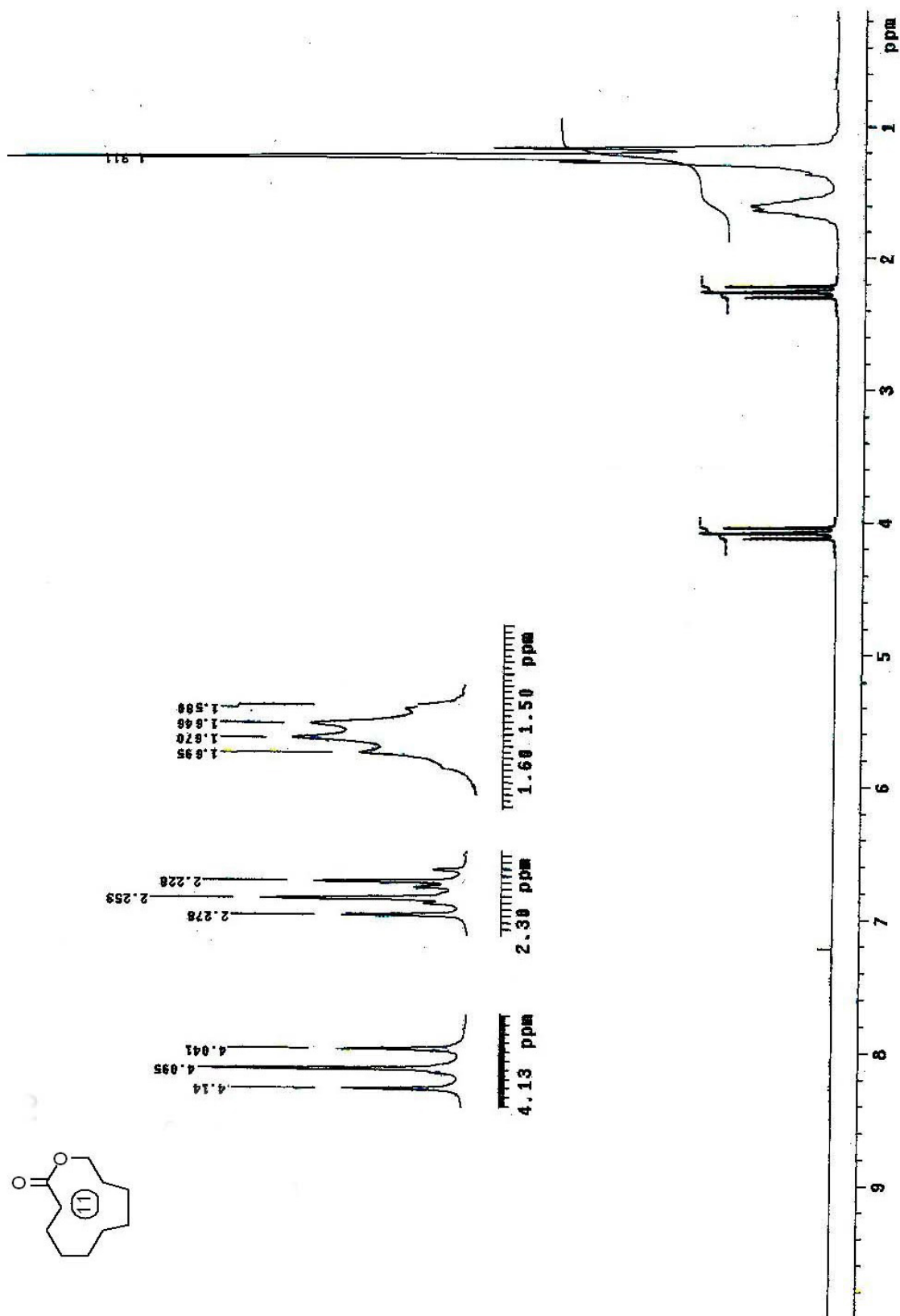


Figure S16.  $^1\text{H NMR}$  in  $\text{CDCl}_3$

Supporting Information

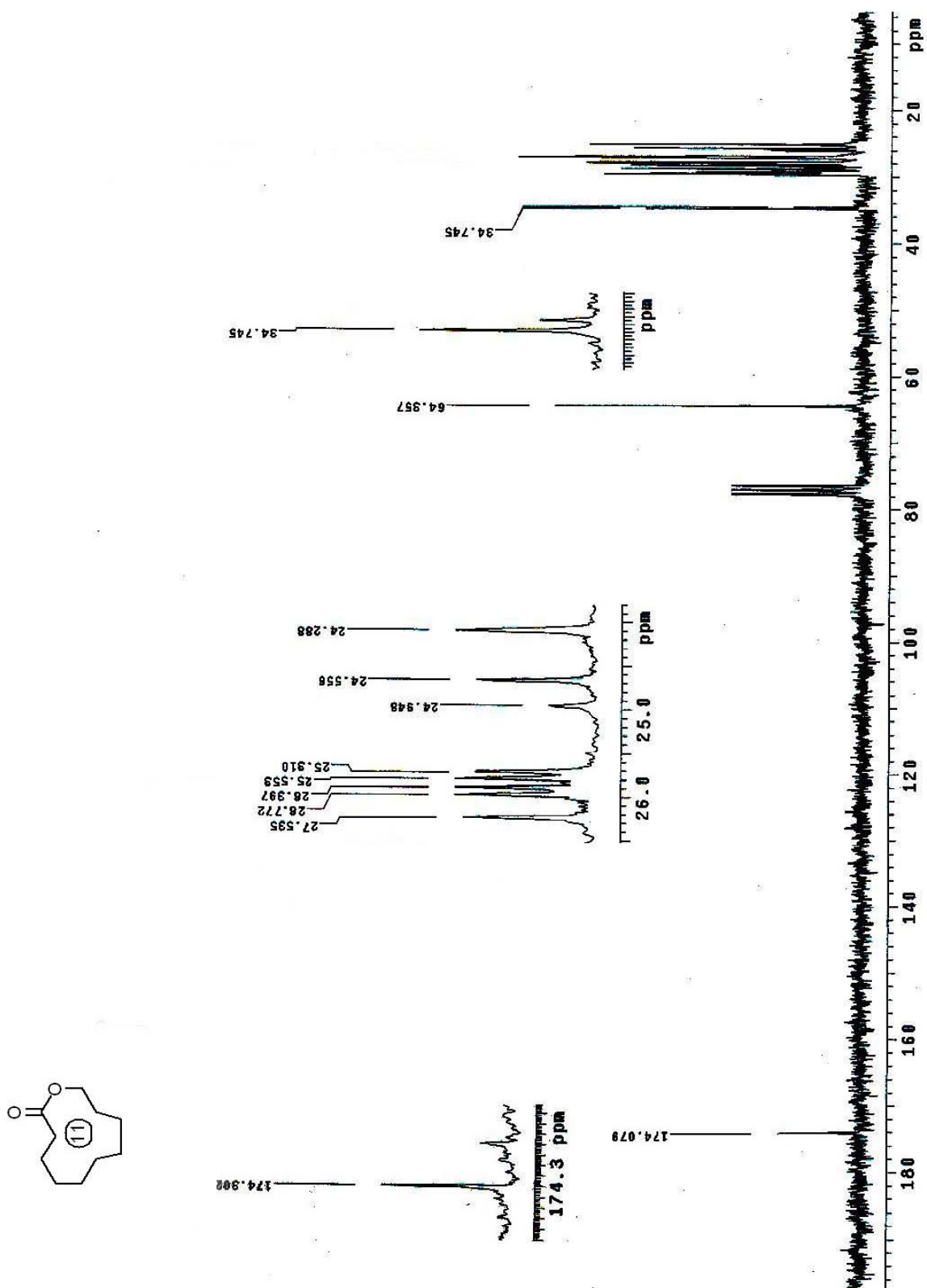


Figure S17.  $^{13}\text{C}$  NMR in  $\text{CDCl}_3$

Supporting Information

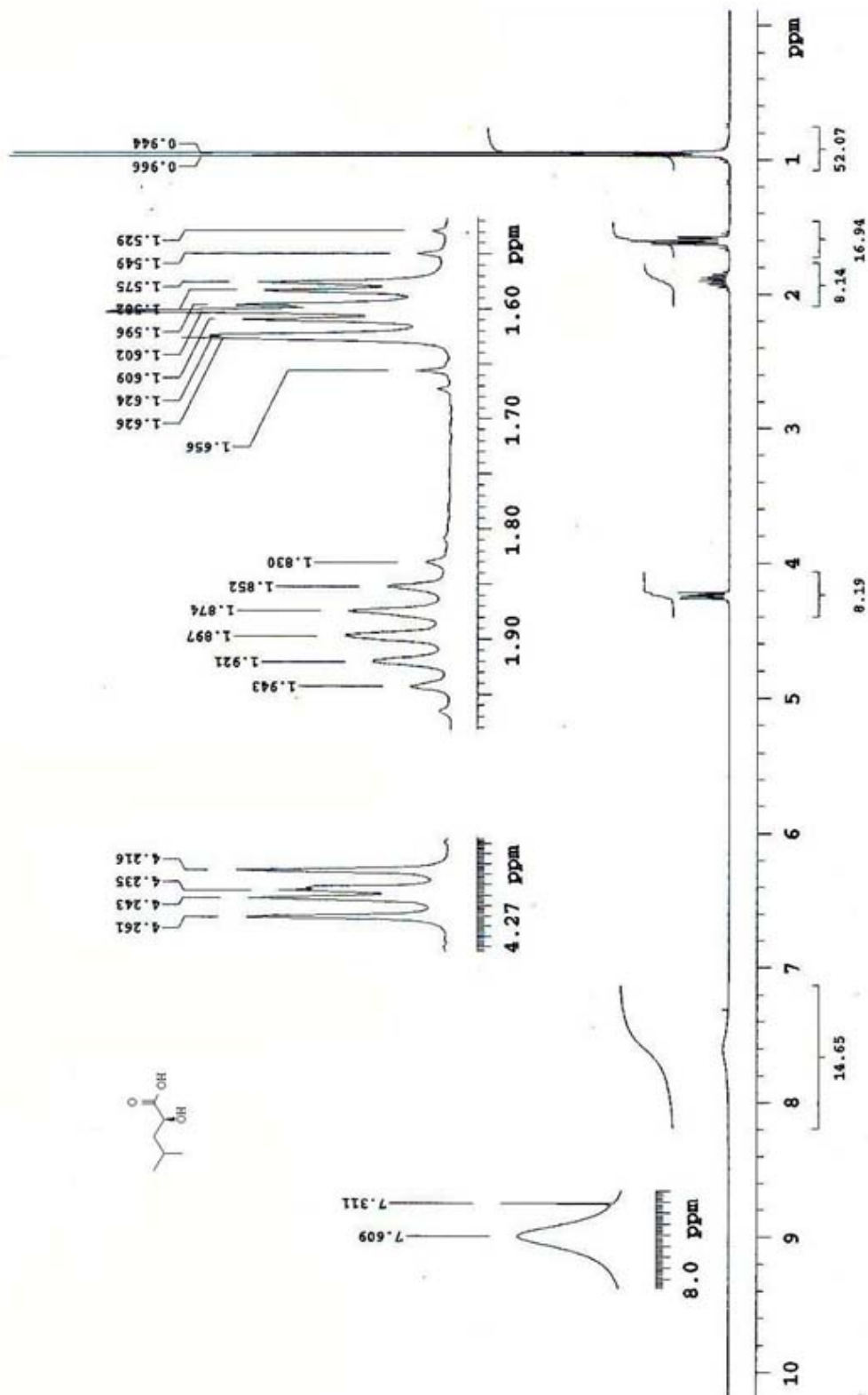


Figure S18.  $^1\text{H}$  NMR of **2** in  $\text{CDCl}_3$

Supporting Information

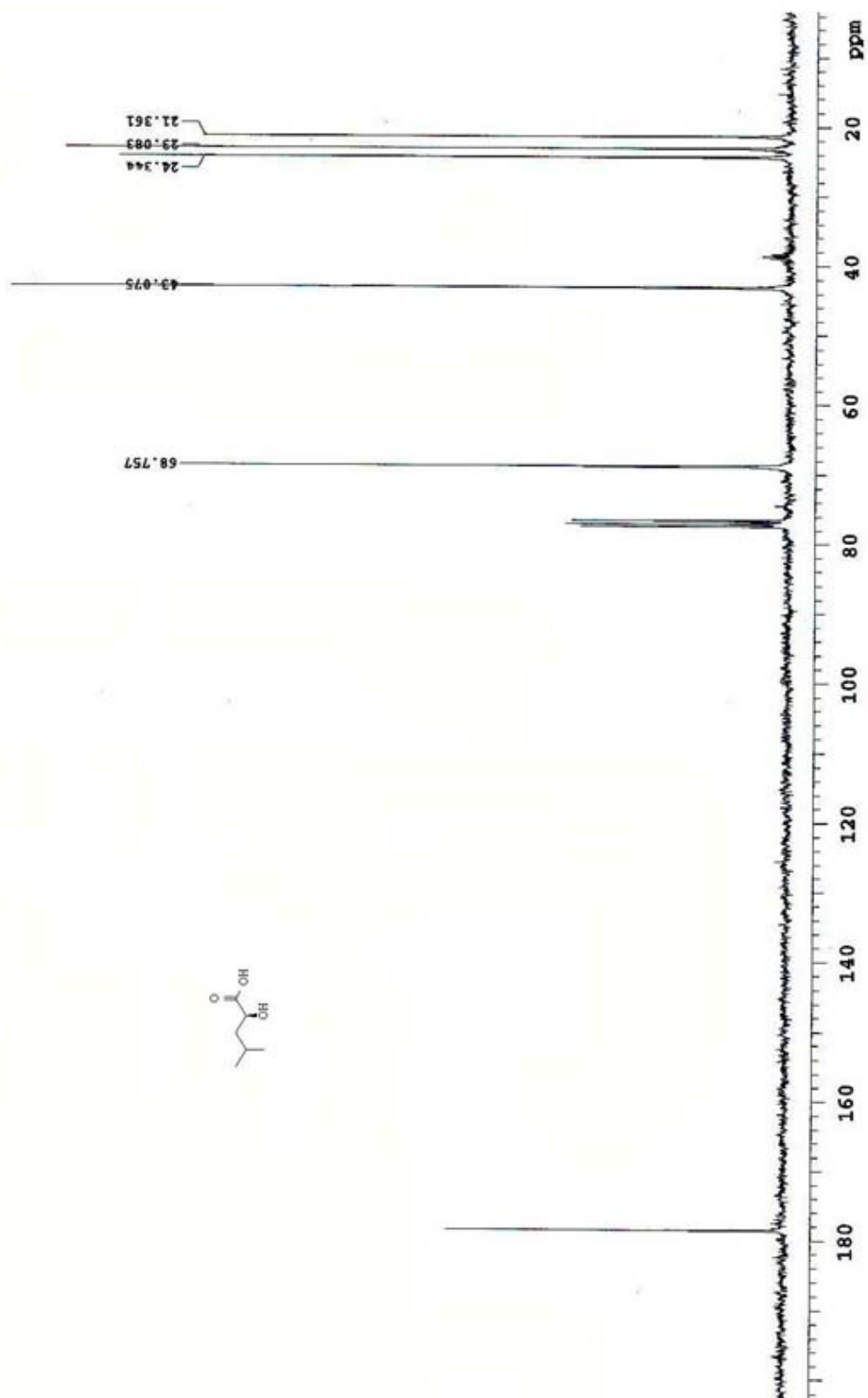


Figure S19. <sup>13</sup>C NMR of 2 in CDCl<sub>3</sub>

Supporting Information

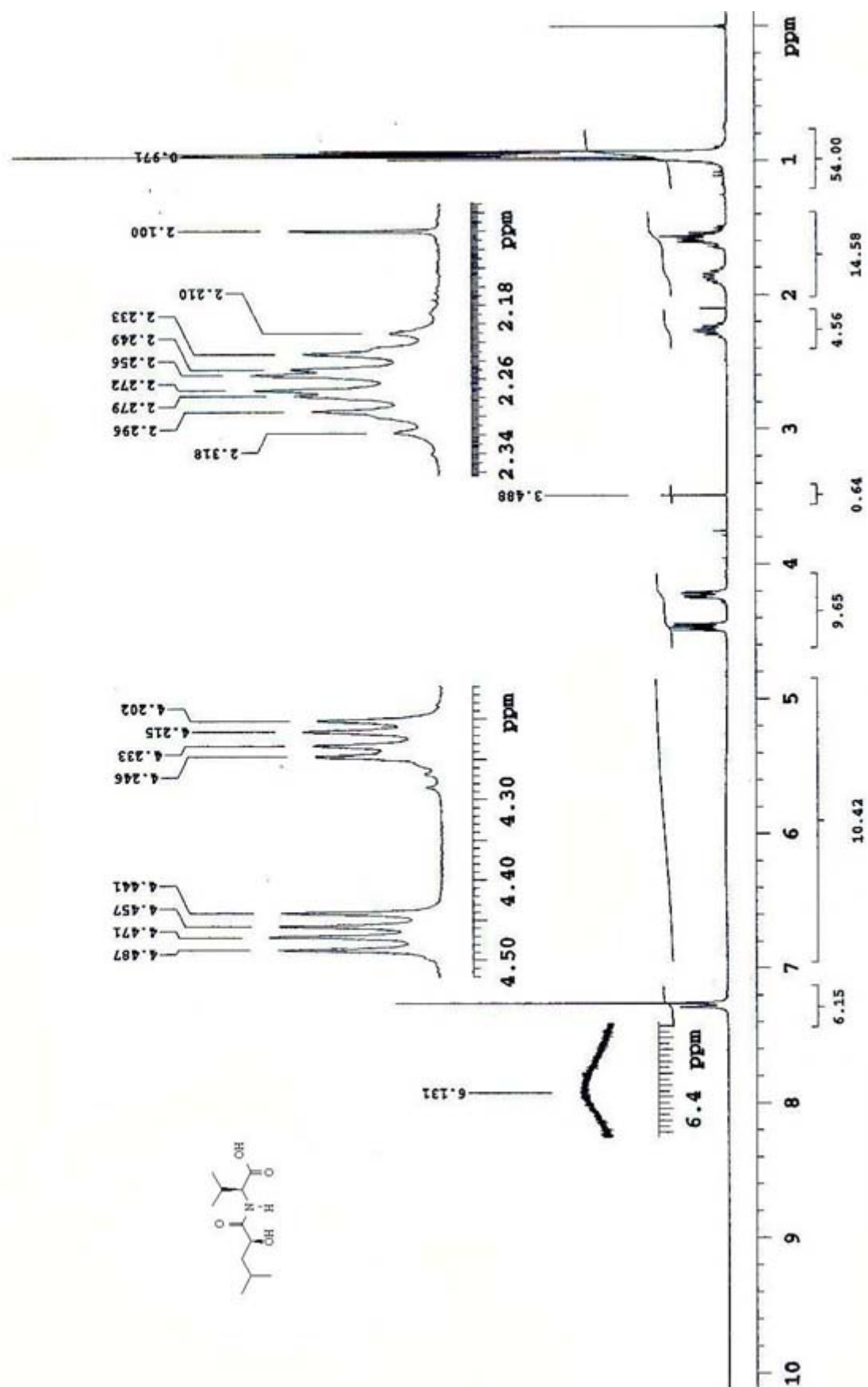


Figure S20.  $^1\text{H}$  NMR of 3 in  $\text{CDCl}_3$

Supporting Information

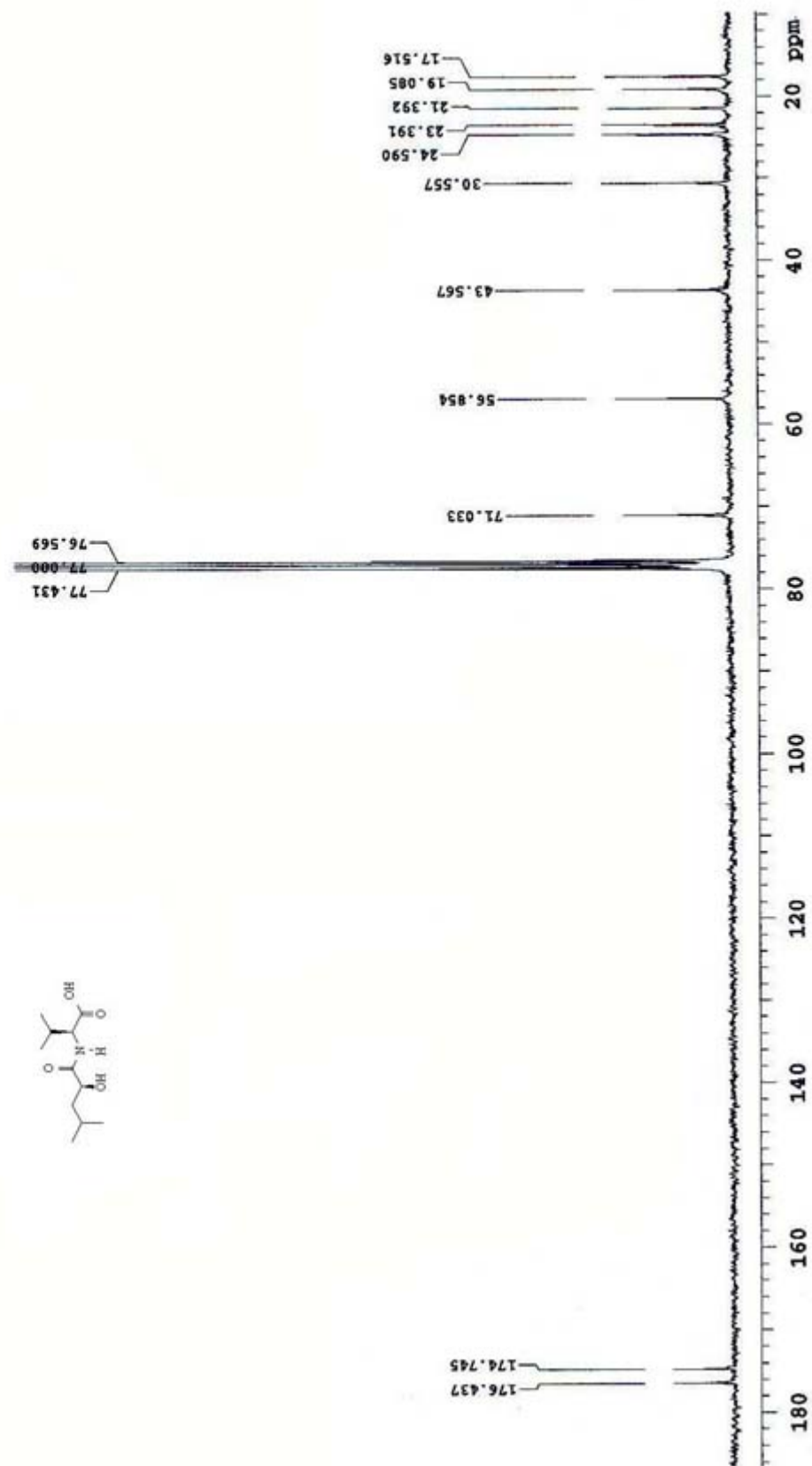


Figure S21.  $^{13}\text{C}$  NMR of 3 in  $\text{CDCl}_3$

Supporting Information

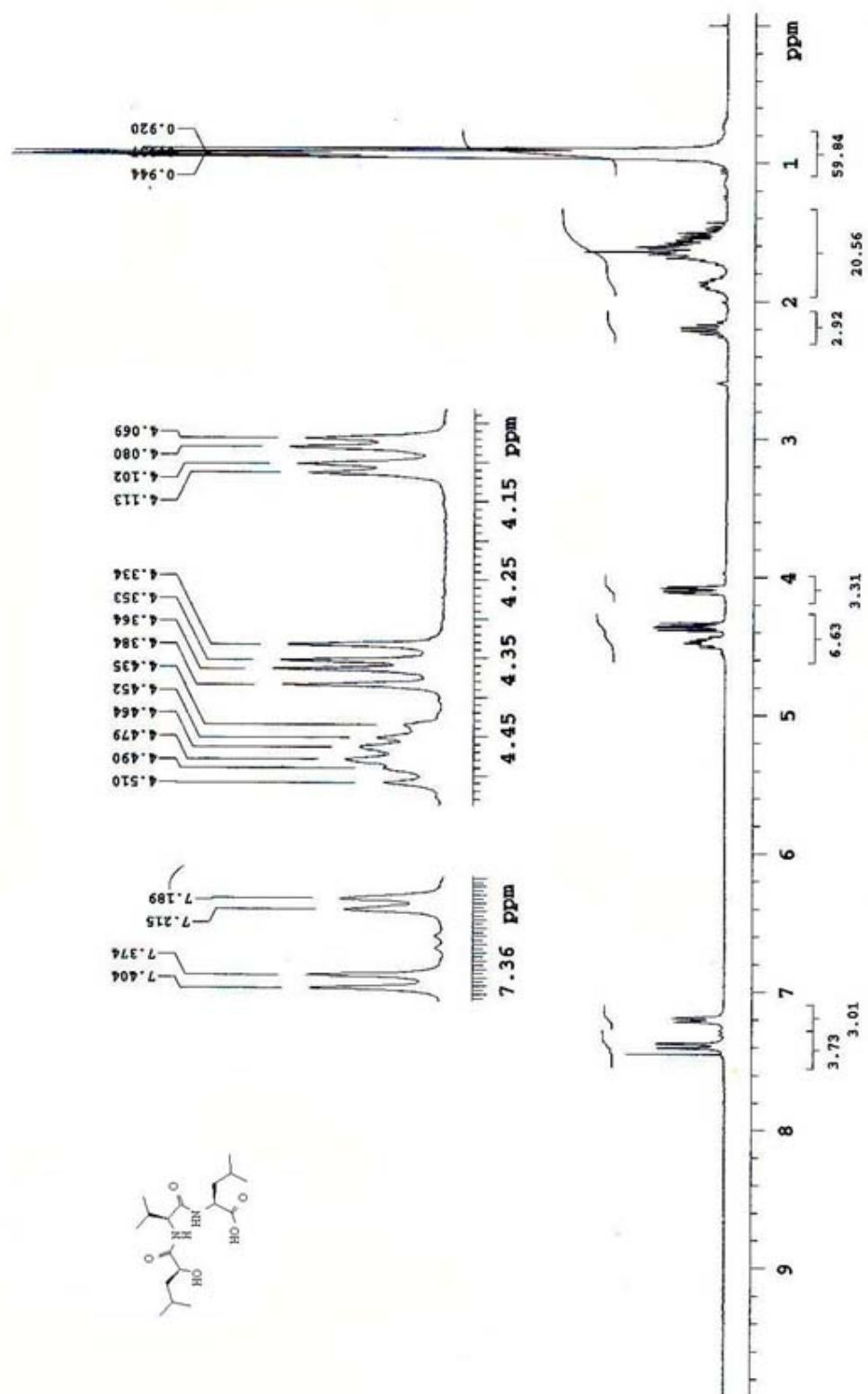


Figure S22.  $^1\text{H}$  NMR of 4 in  $\text{CDCl}_3$

### Supporting Information

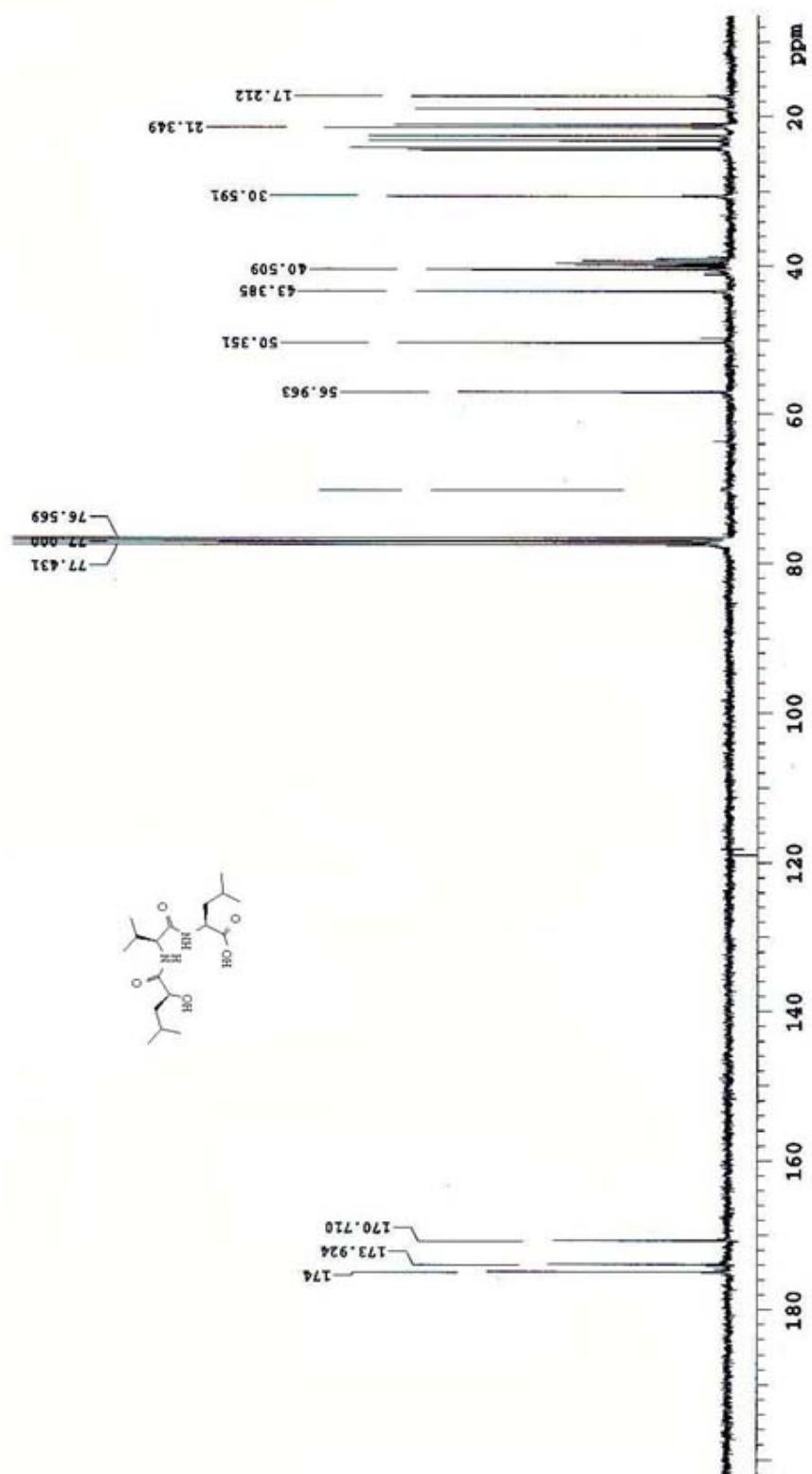


Figure S23.  $^{13}\text{C}$  NMR of 4 in  $\text{CDCl}_3$



Supporting Information

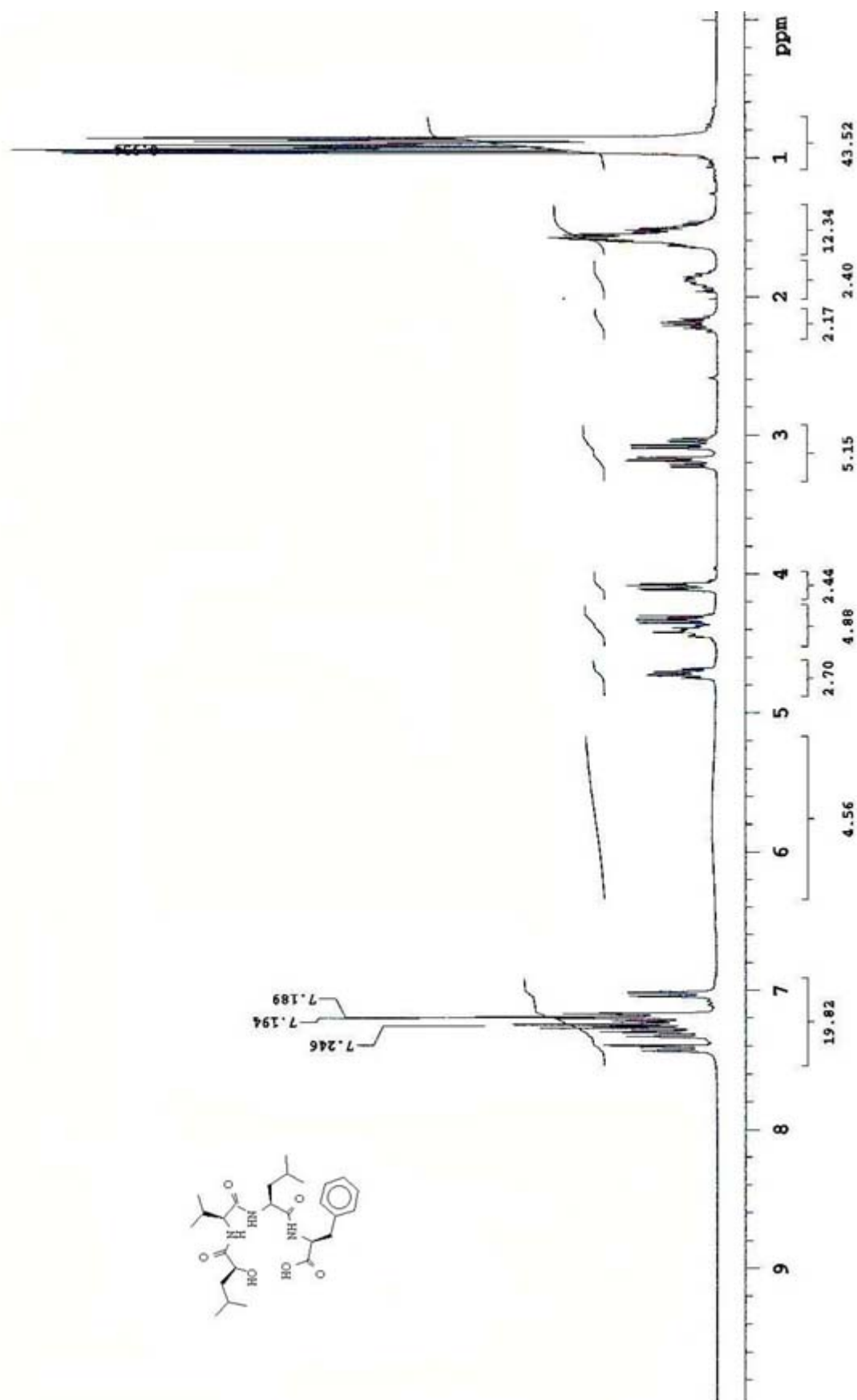


Figure S24.  $^1\text{H}$  NMR of **5** in  $\text{CDCl}_3$

Supporting Information

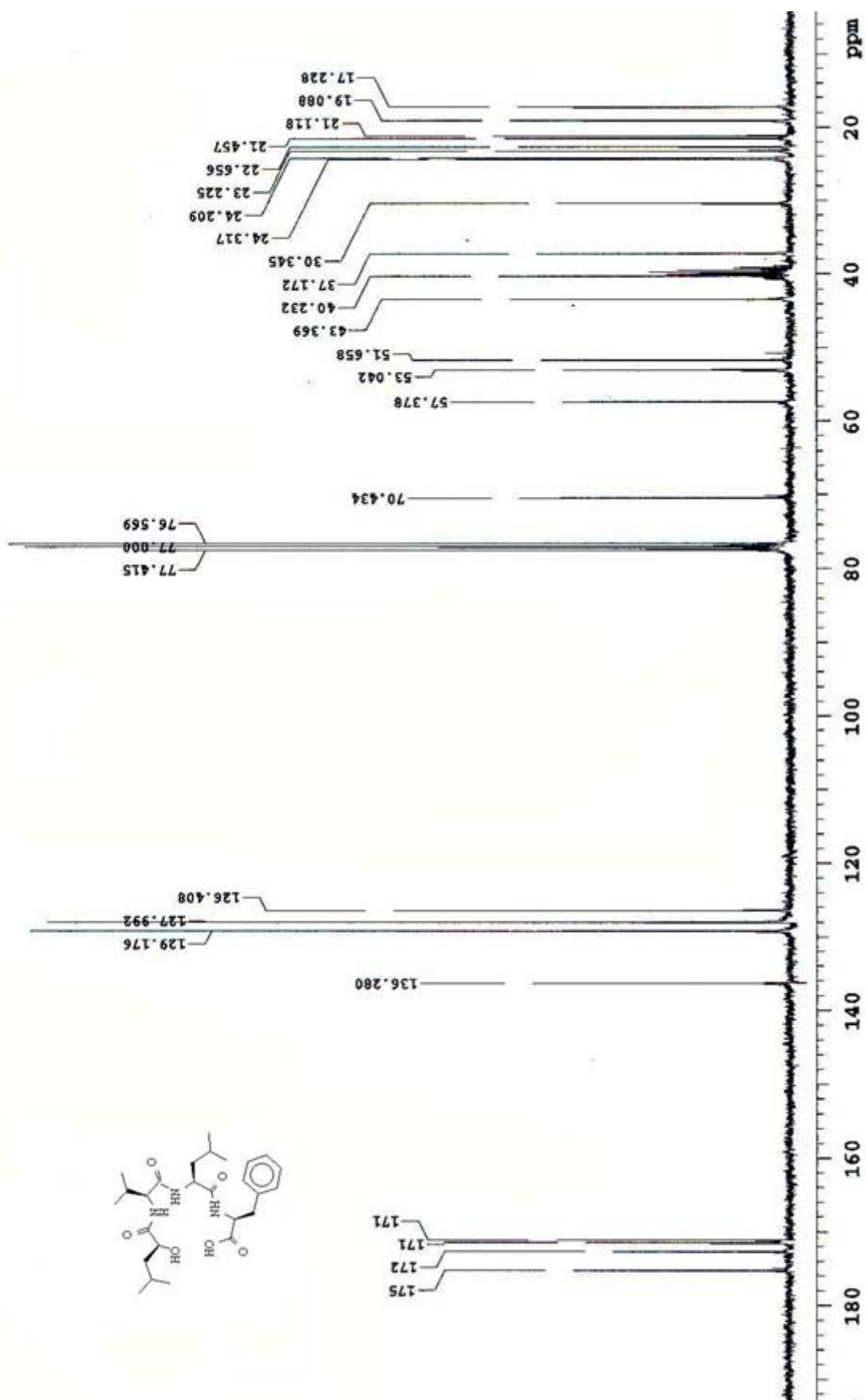


Figure S25.  $^{13}\text{C}$  NMR of 5 in  $\text{CDCl}_3$

Supporting Information

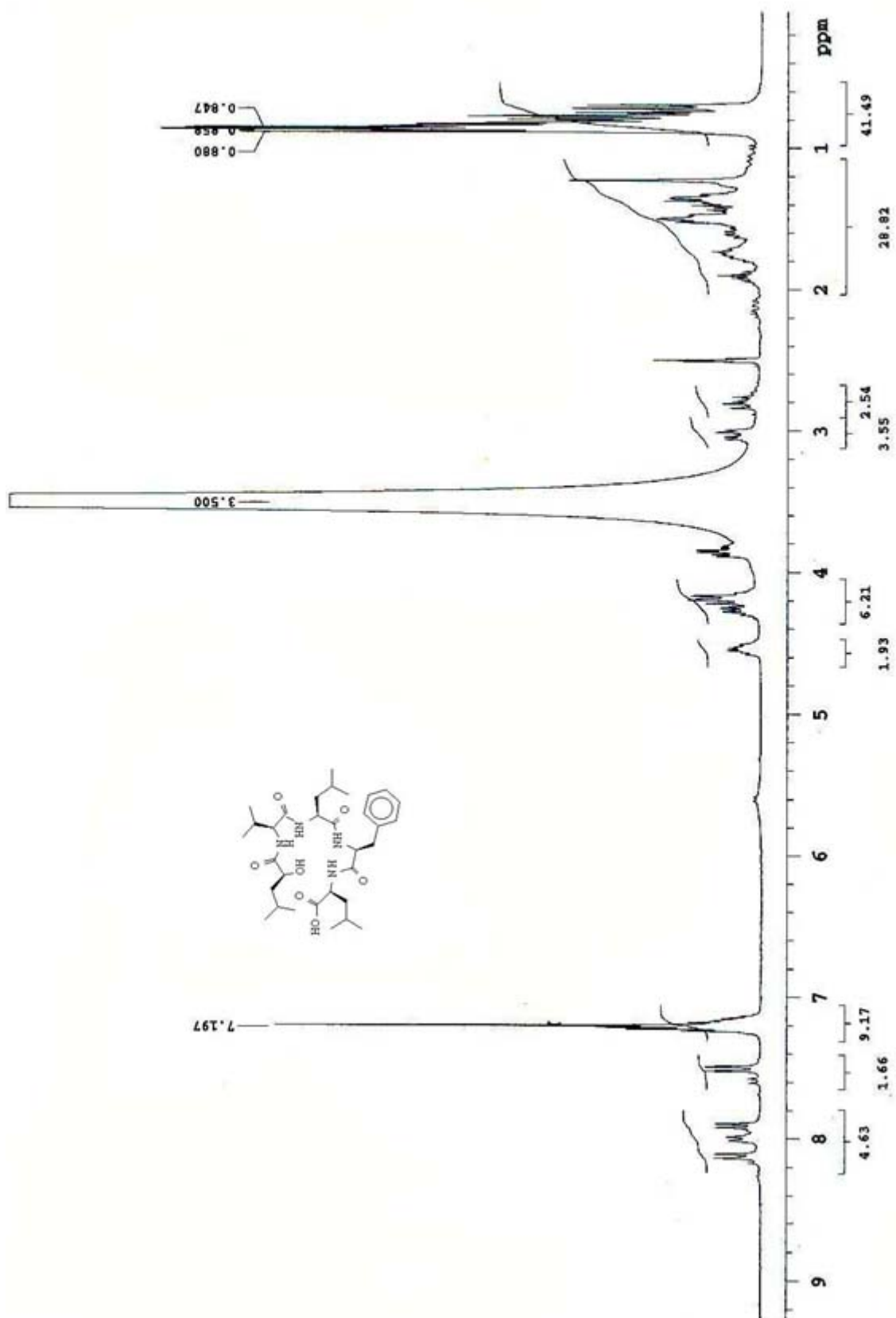


Figure S26. <sup>1</sup>H NMR of **6** in CD<sub>3</sub>OD

Supporting Information

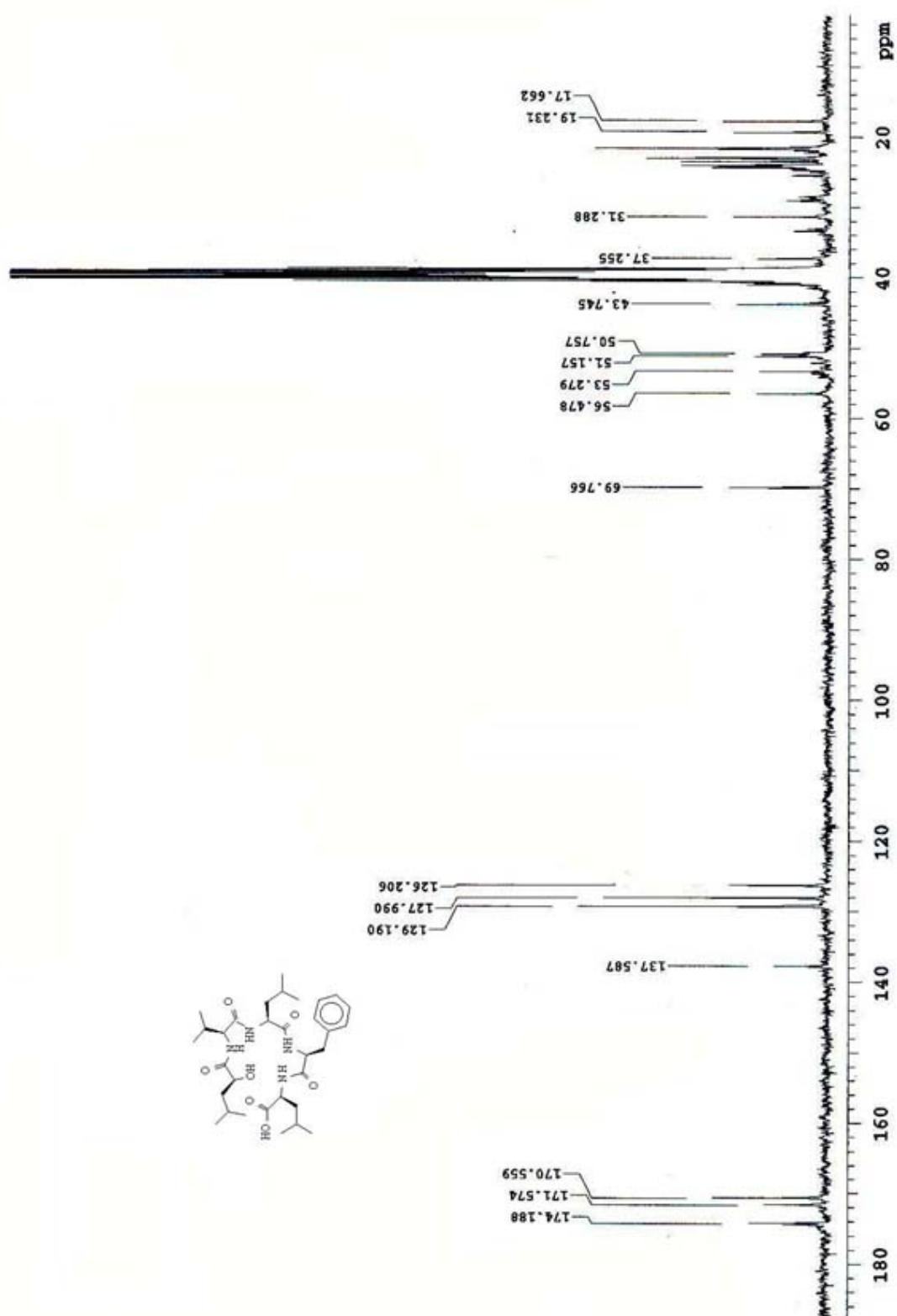


Figure S27. <sup>13</sup>C NMR of 6 in CD<sub>3</sub>OD

Supporting Information

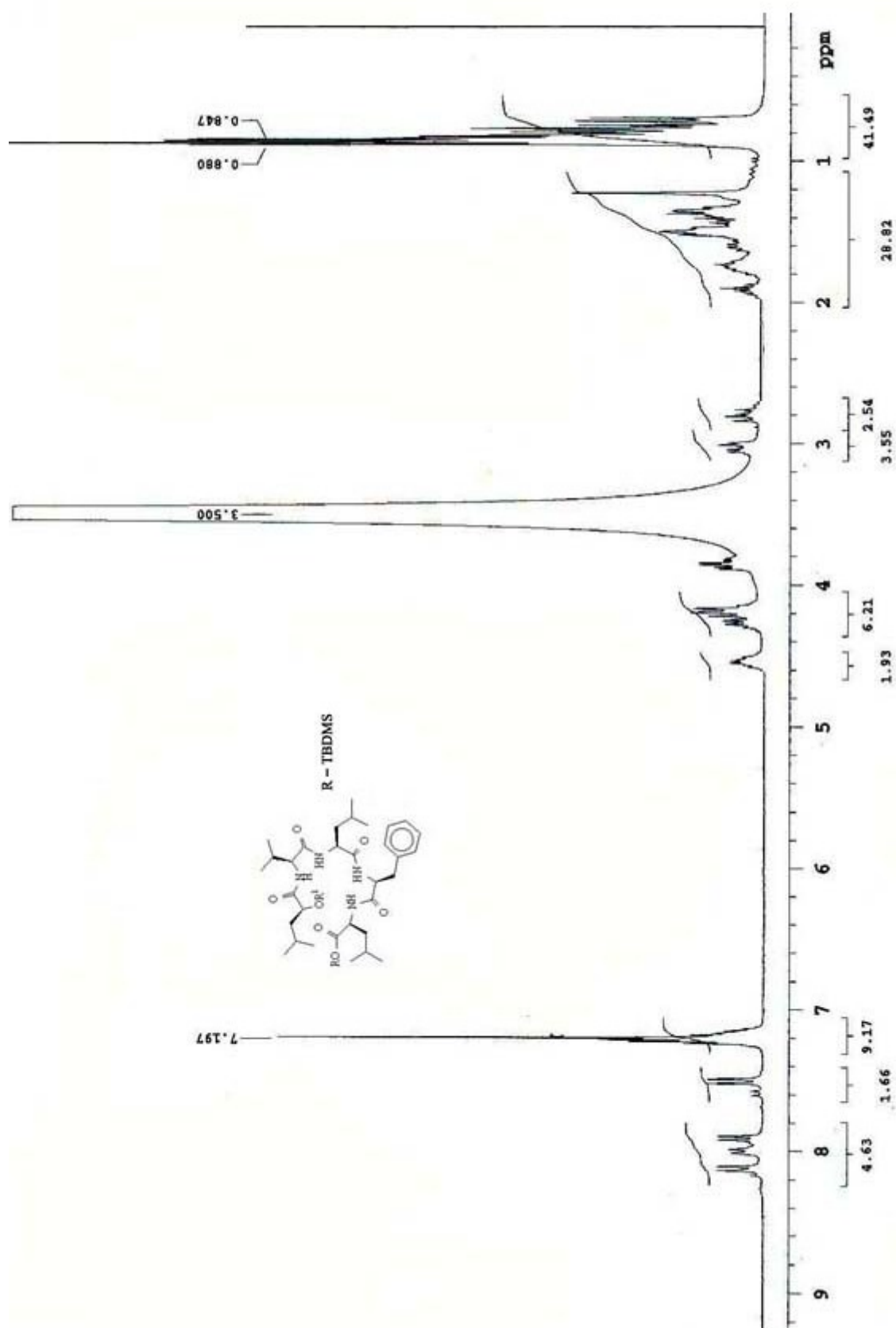


Figure S28.  $^1\text{H}$  NMR of **8** in  $\text{CD}_3\text{OD}$

Supporting Information

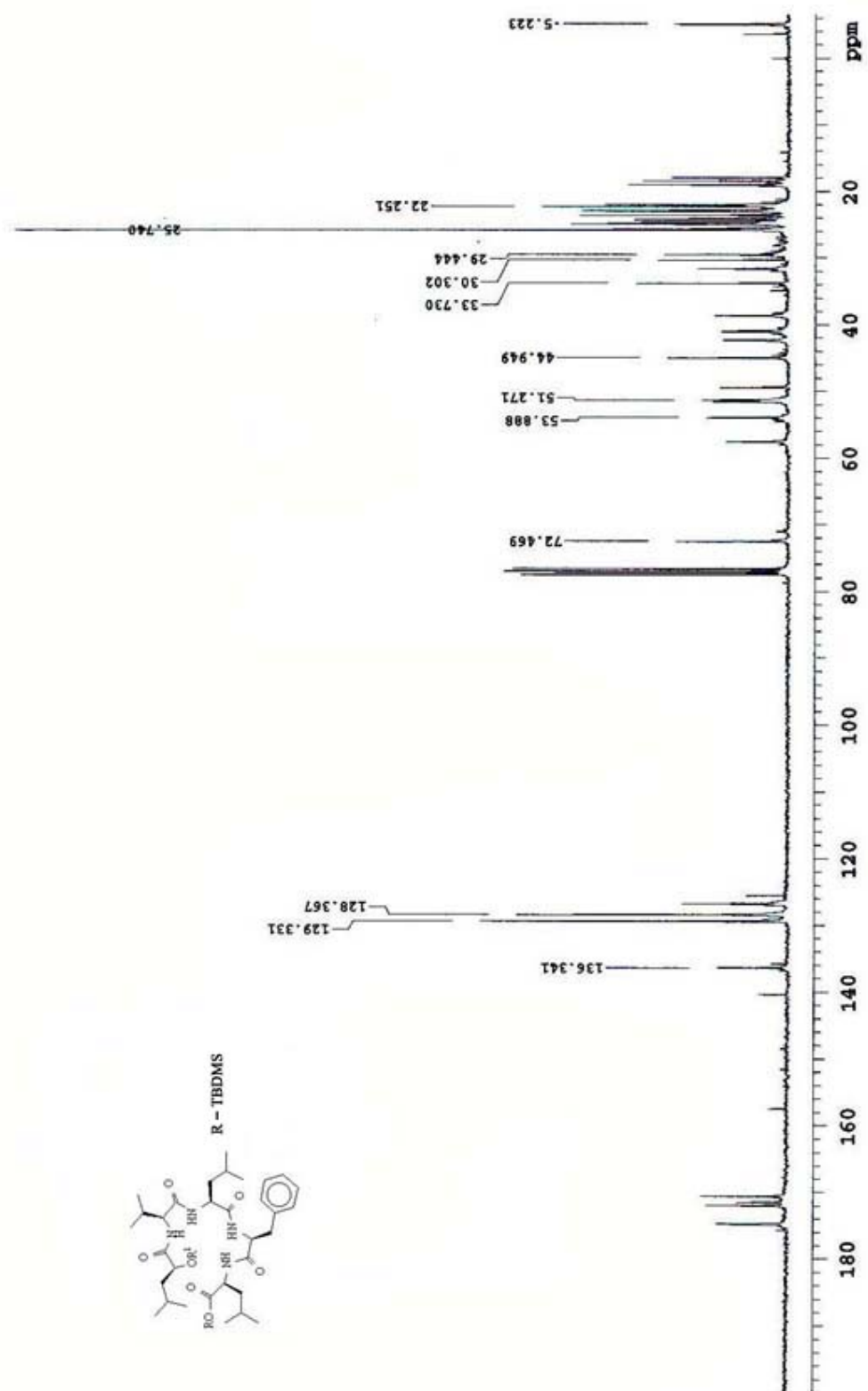


Figure S29.  $^{13}\text{C}$  NMR of **8** in  $\text{CD}_3\text{OD}$

Supporting Information

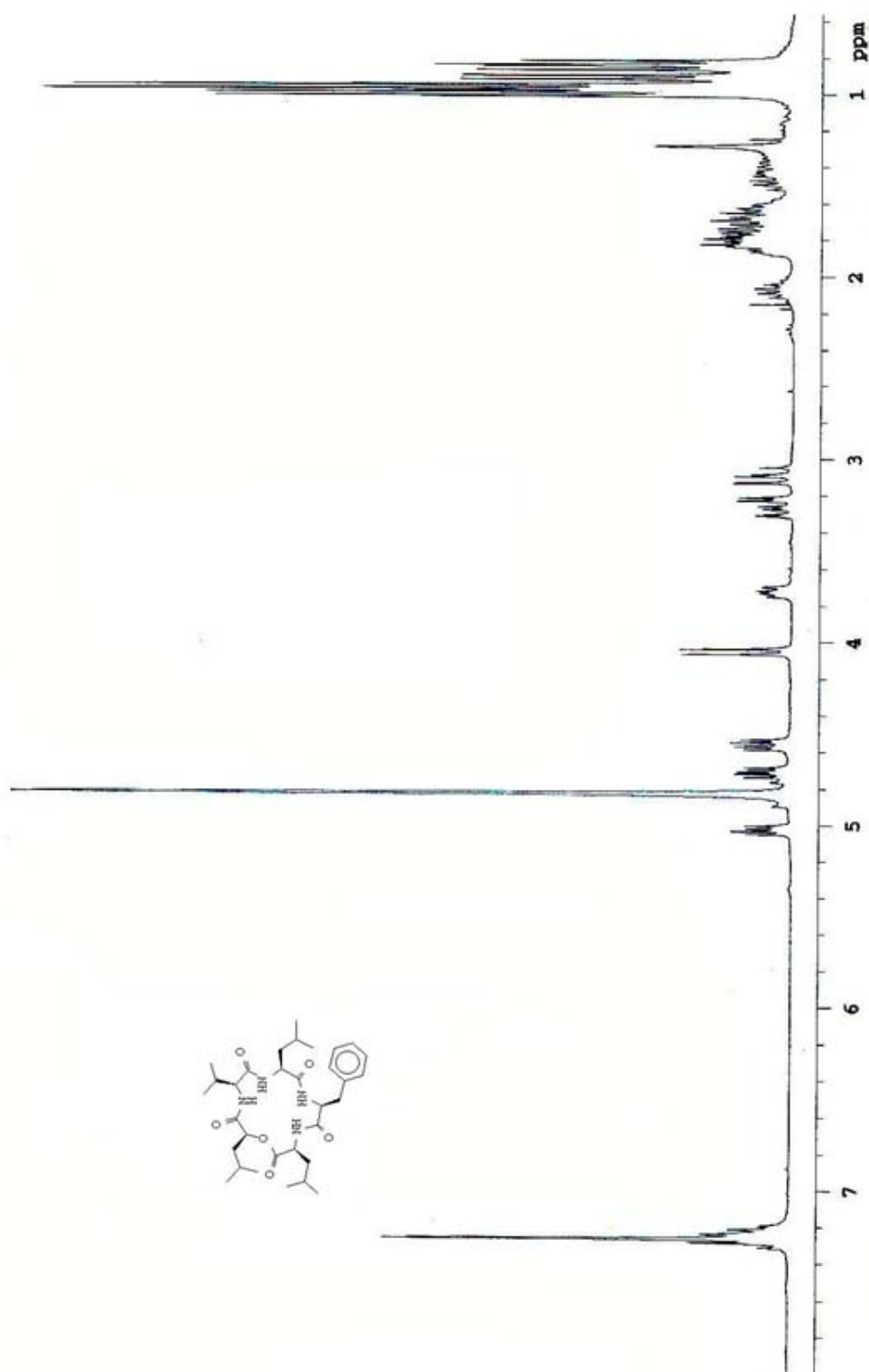


Figure S30.  $^1\text{H}$  NMR of 7 in  $\text{CD}_3\text{OD}$

Supporting Information

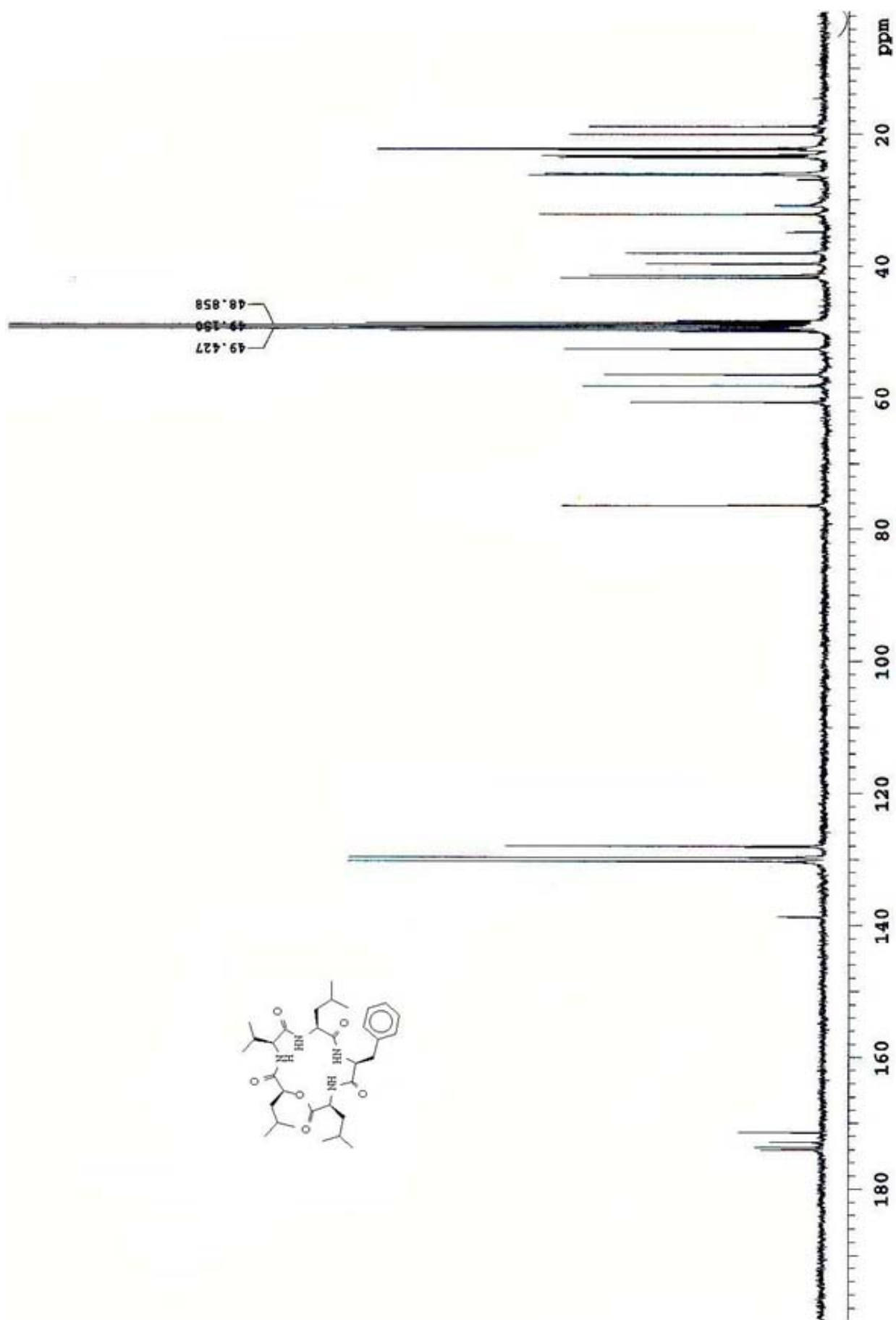
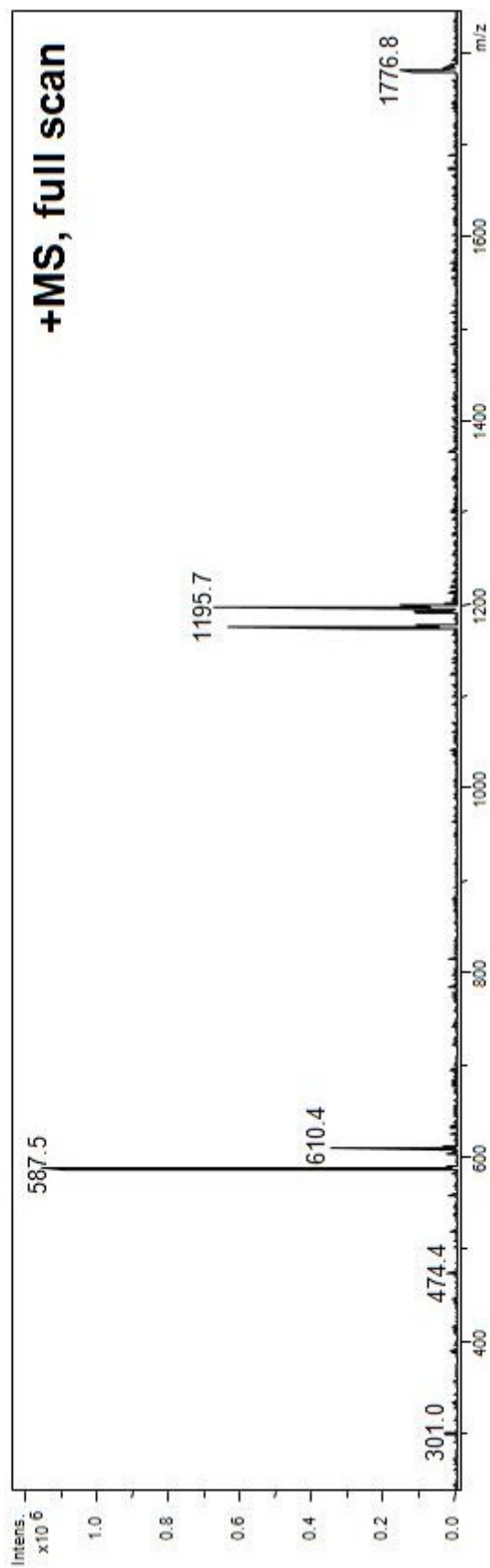


Figure S31.  $^{13}\text{C}$  NMR of **7** in  $\text{CD}_3\text{OD}$



## Esquire3000plus Data

## Cyclopentapeptide



**587.4 is M+H, then**

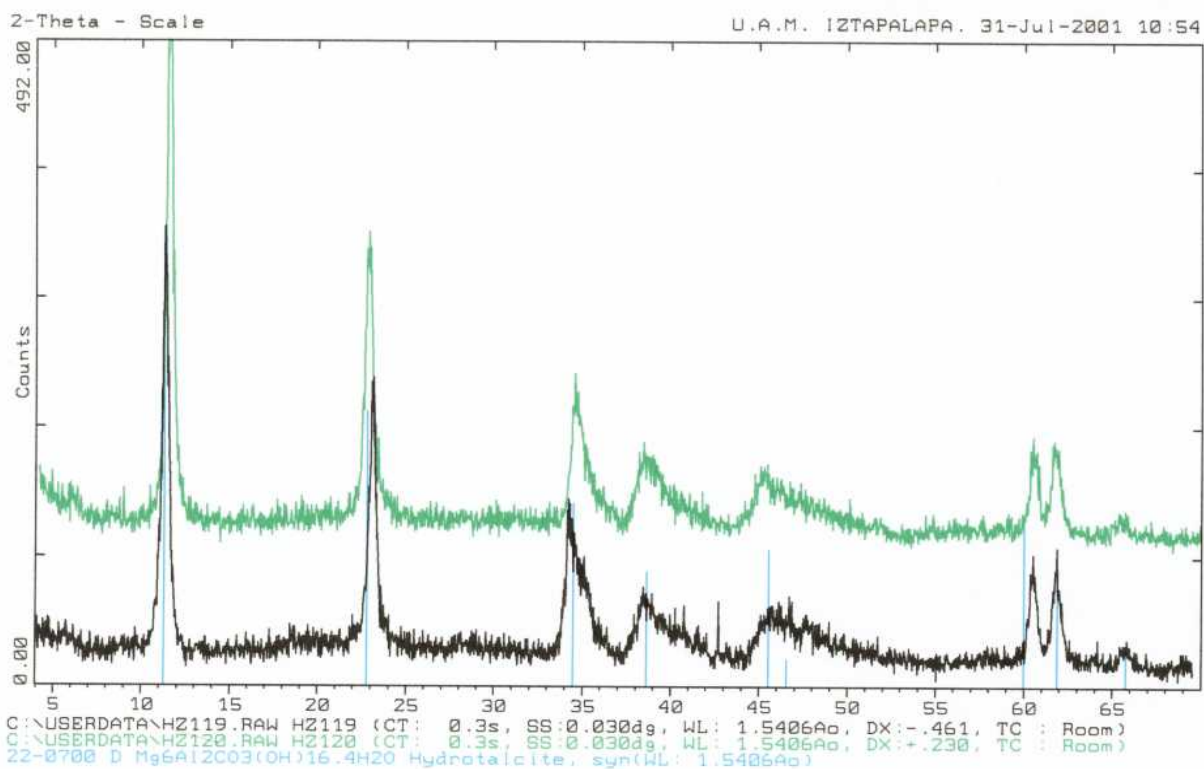
**609.4 is M+Na**

**1173.4 is 2M+H**

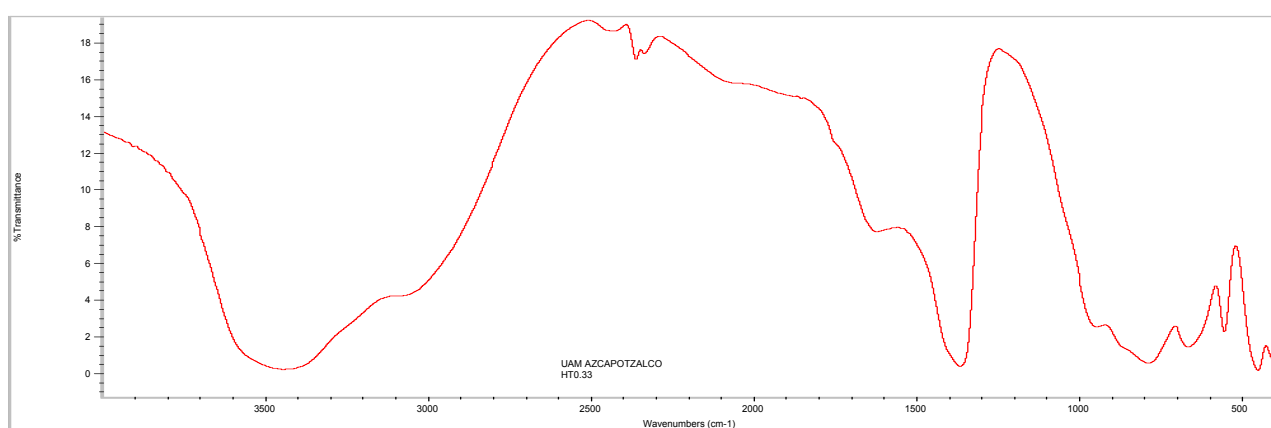
**1195.7 is 2M+Na**

Figure S32. ESI MS of 7

### Supporting Information



**Figure S33.** X-ray diffraction patterns of hydrotalcite Mg-Al with  $x = \text{Al} / (\text{Al} + \text{Mg}) = 0.33$  are shown; these materials show a crystalline hydrotalcite pattern, indicating the formation of these compounds.



**Figure S34.** FT-IR of hydrotalcite Mg-Al with  $x = \text{Al} / (\text{Al} + \text{Mg}) = 0.33$