

**Stereochemical assignment of four diastereoisomers of 3,4-dimethylpyroglutamic acid,  
a moiety of callipeltin B**

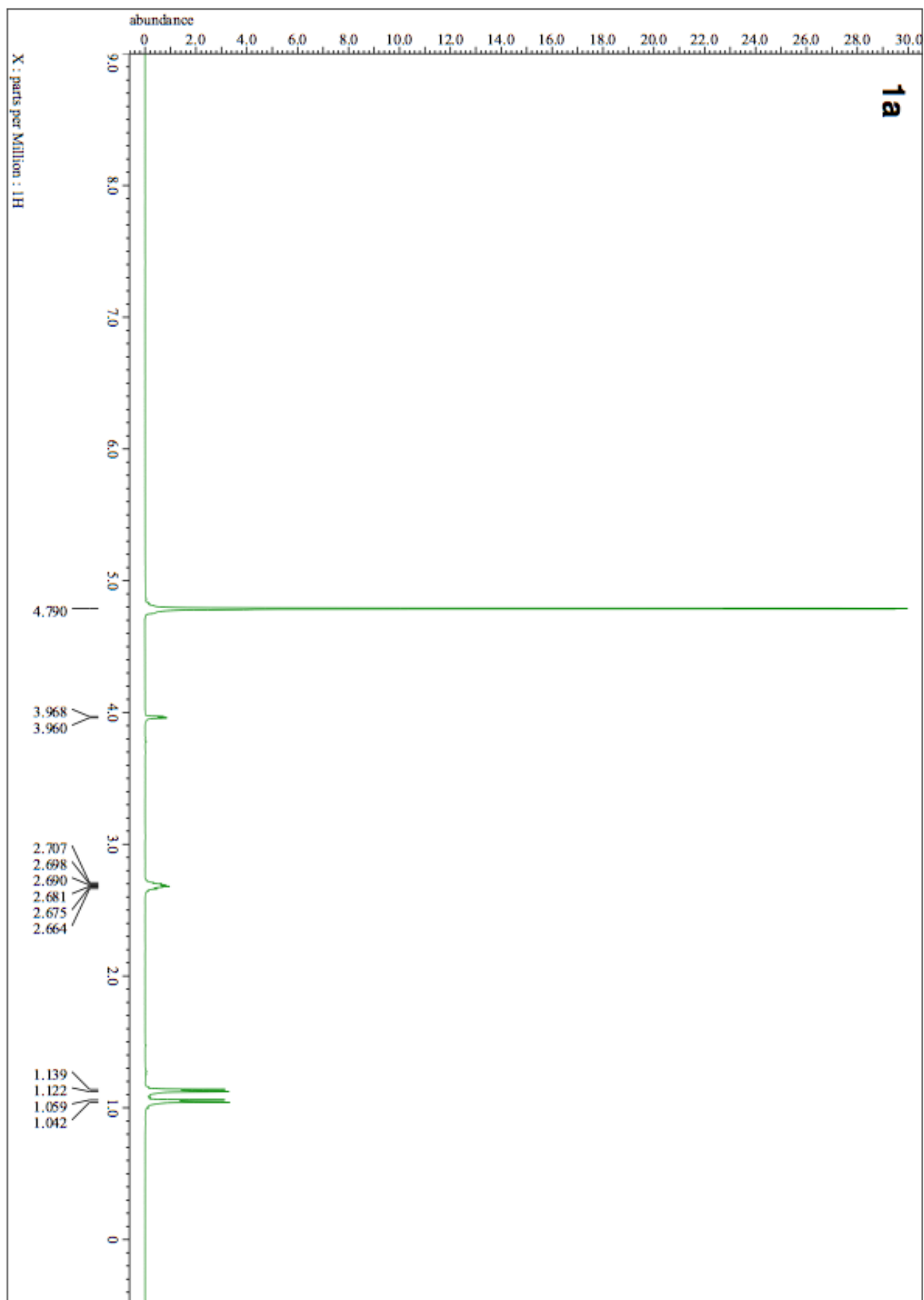
**Supporting Information**

Mari Kikuchi and Hiroyuki Konno\*

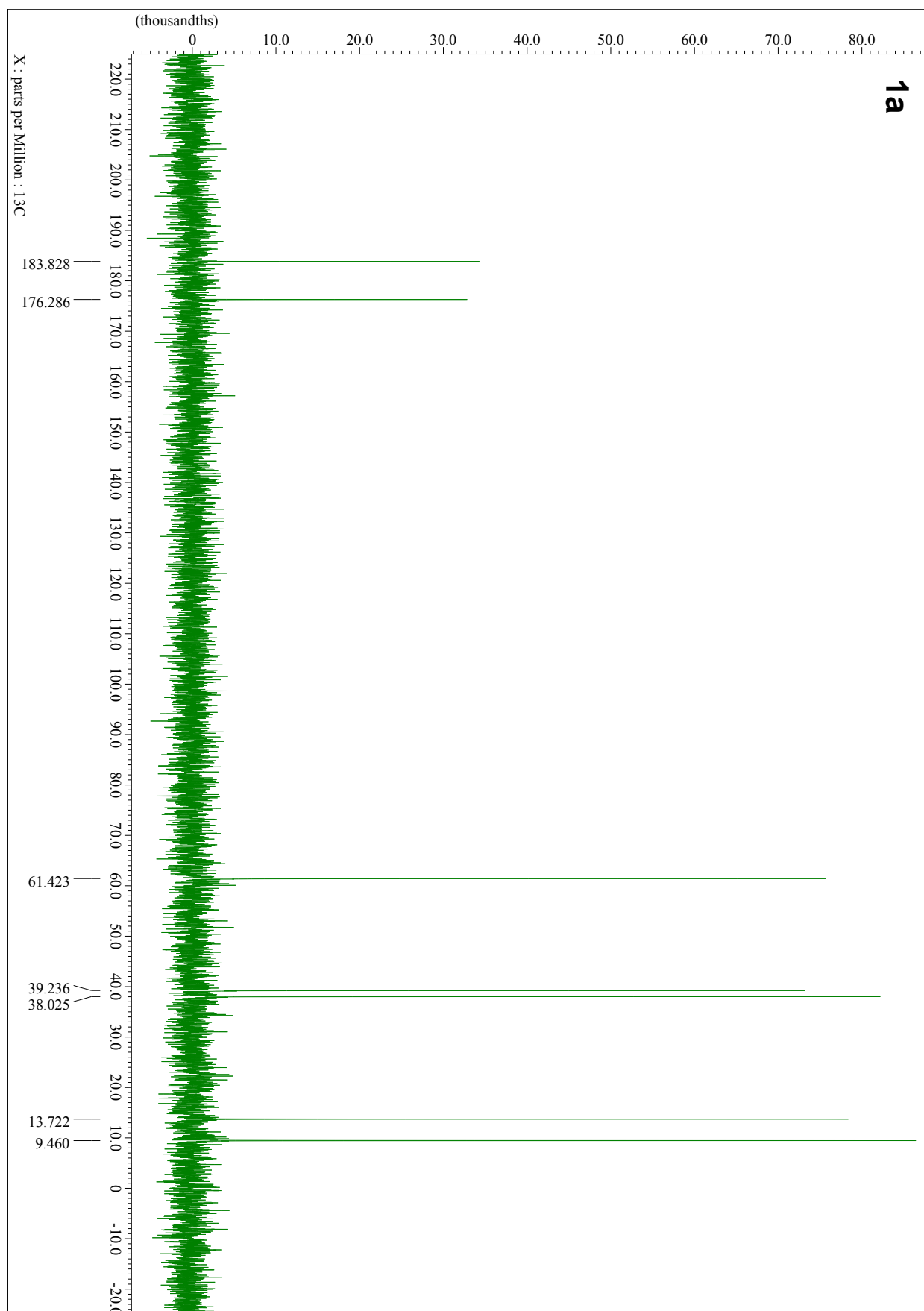
*Department of Biochemical Engineering, Graduate School of Science and Technology, Yamagata  
University, Yonezawa, Yamagata 992-8510, Japan*

konno@yz.yamagata-u.ac.jp

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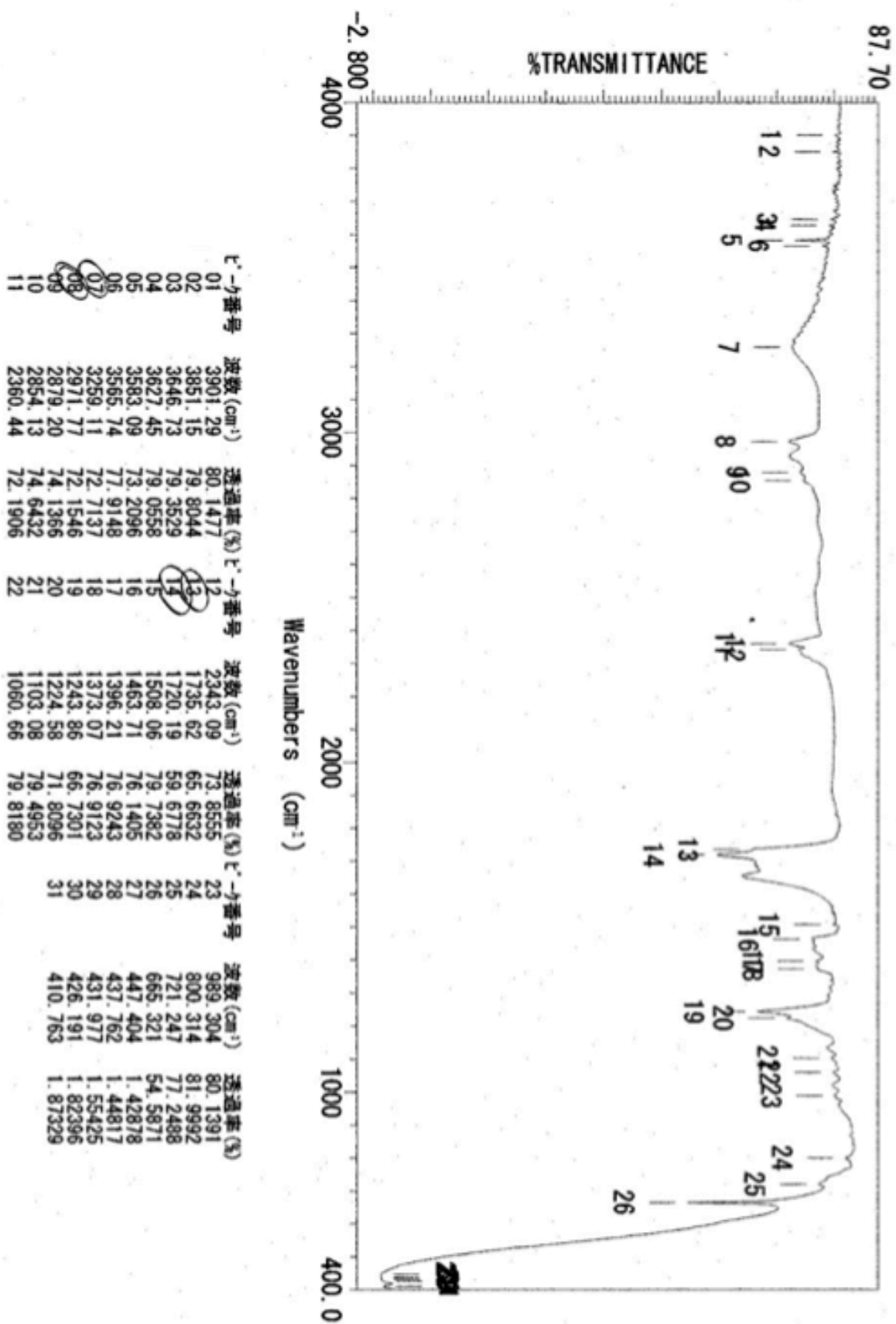


Supplementary Figure S1.  $^1\text{H}$  NMR spectrum of **1a**.

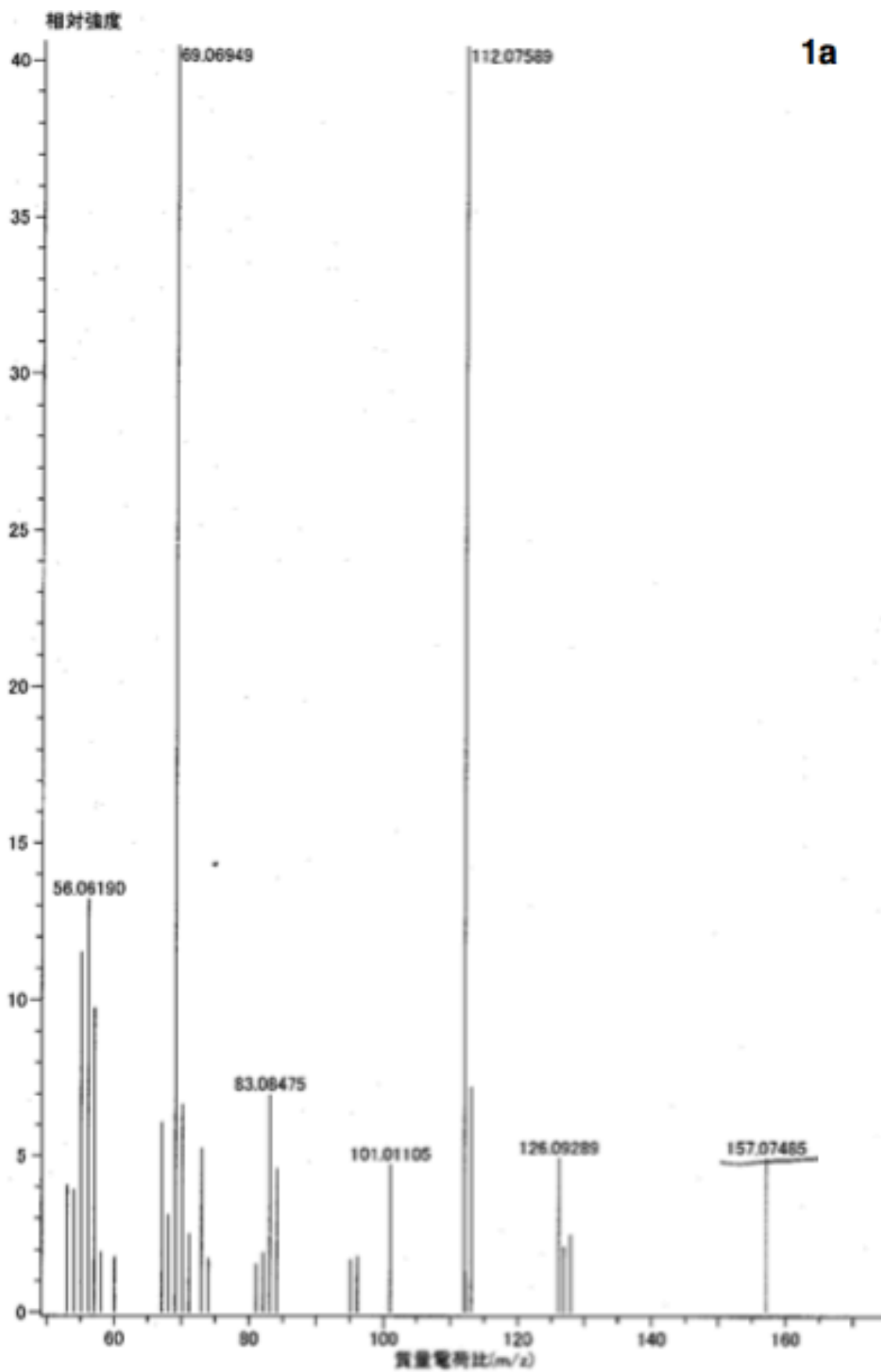


Supplementary Figure S2.  $^{13}\text{C}$  NMR spectrum of **1a**.

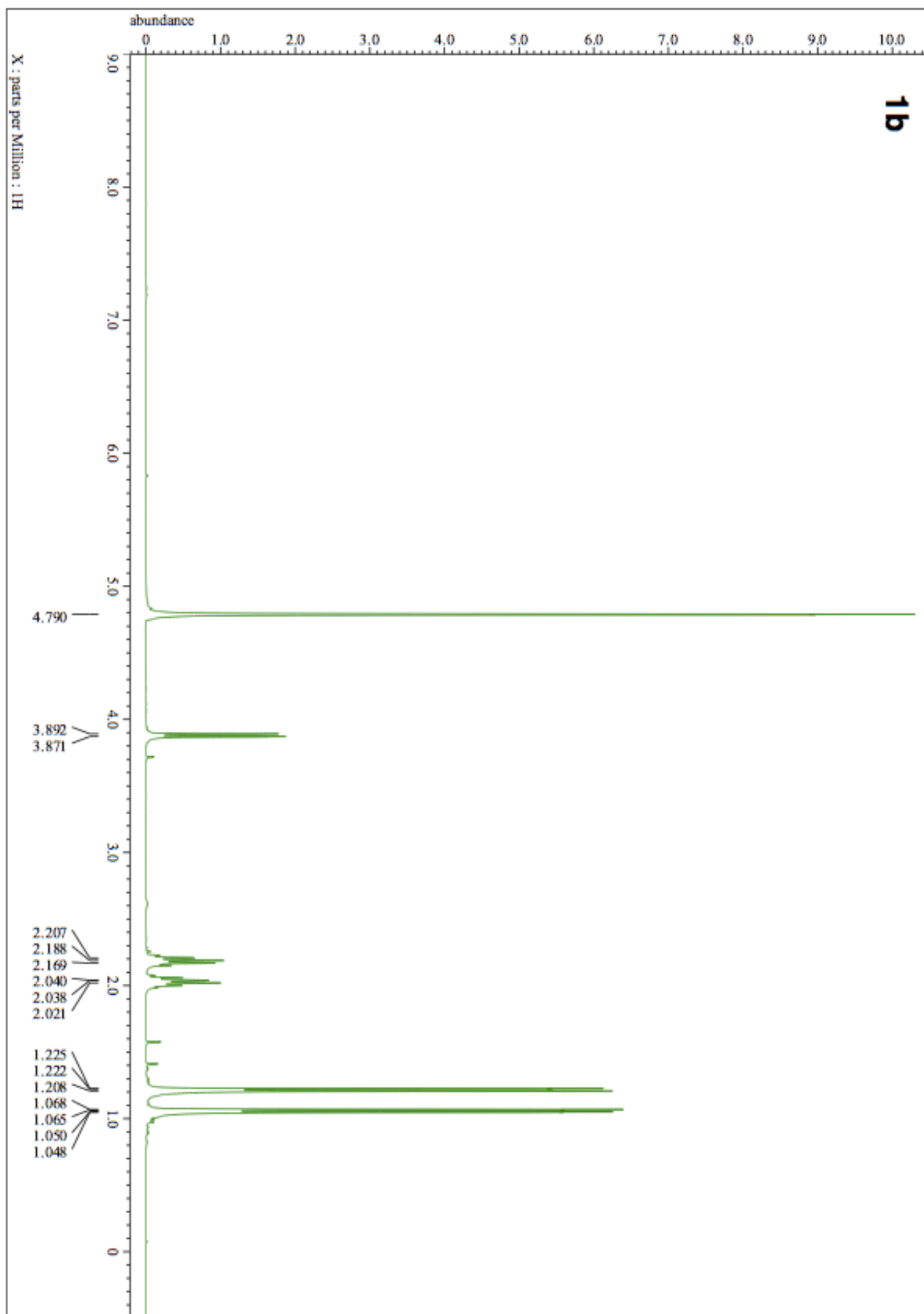
1a



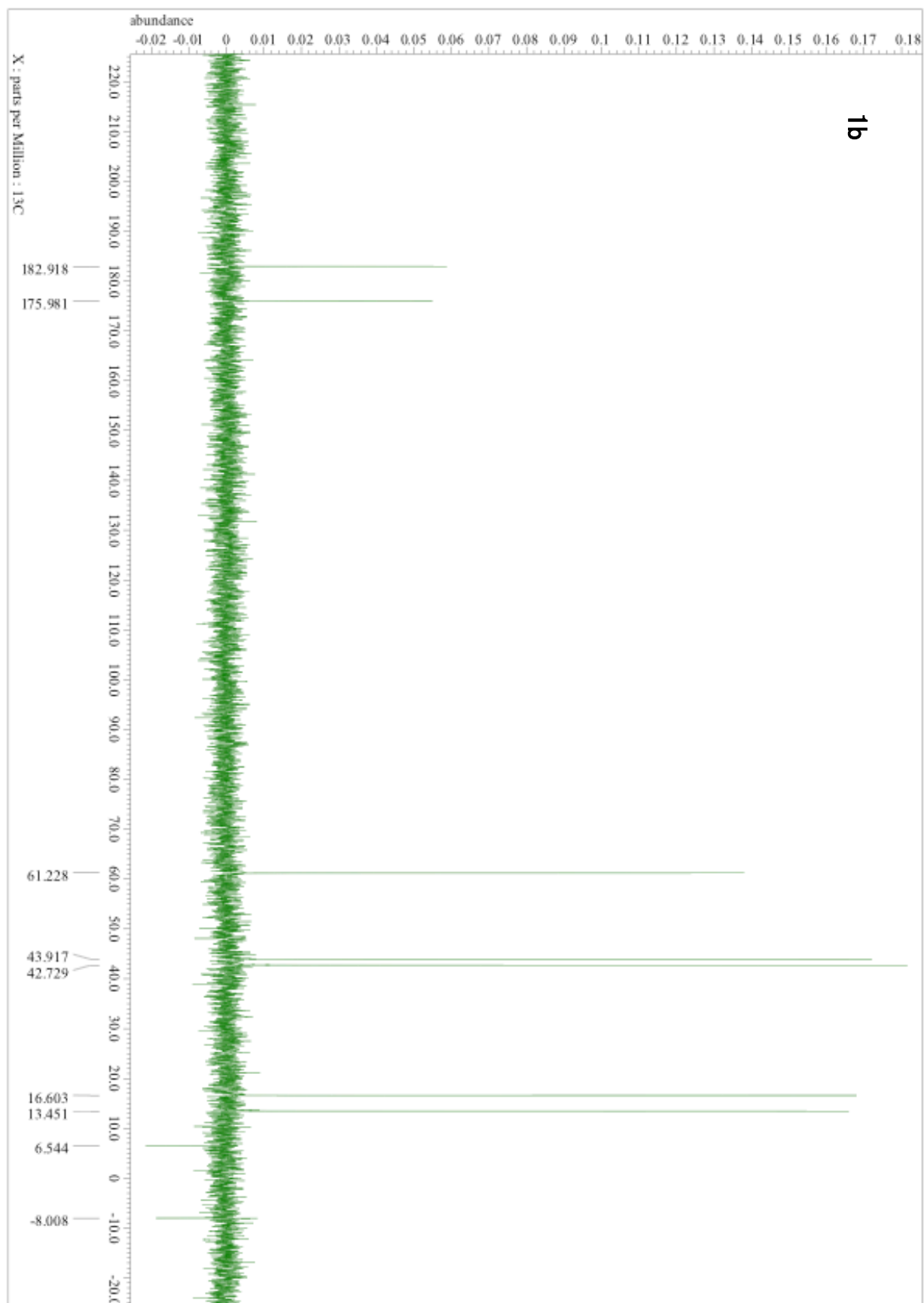
Supplementary Figure S3. IR spectrum of 1a.



Supplementary Figure S4. MS spectrum of 1a.

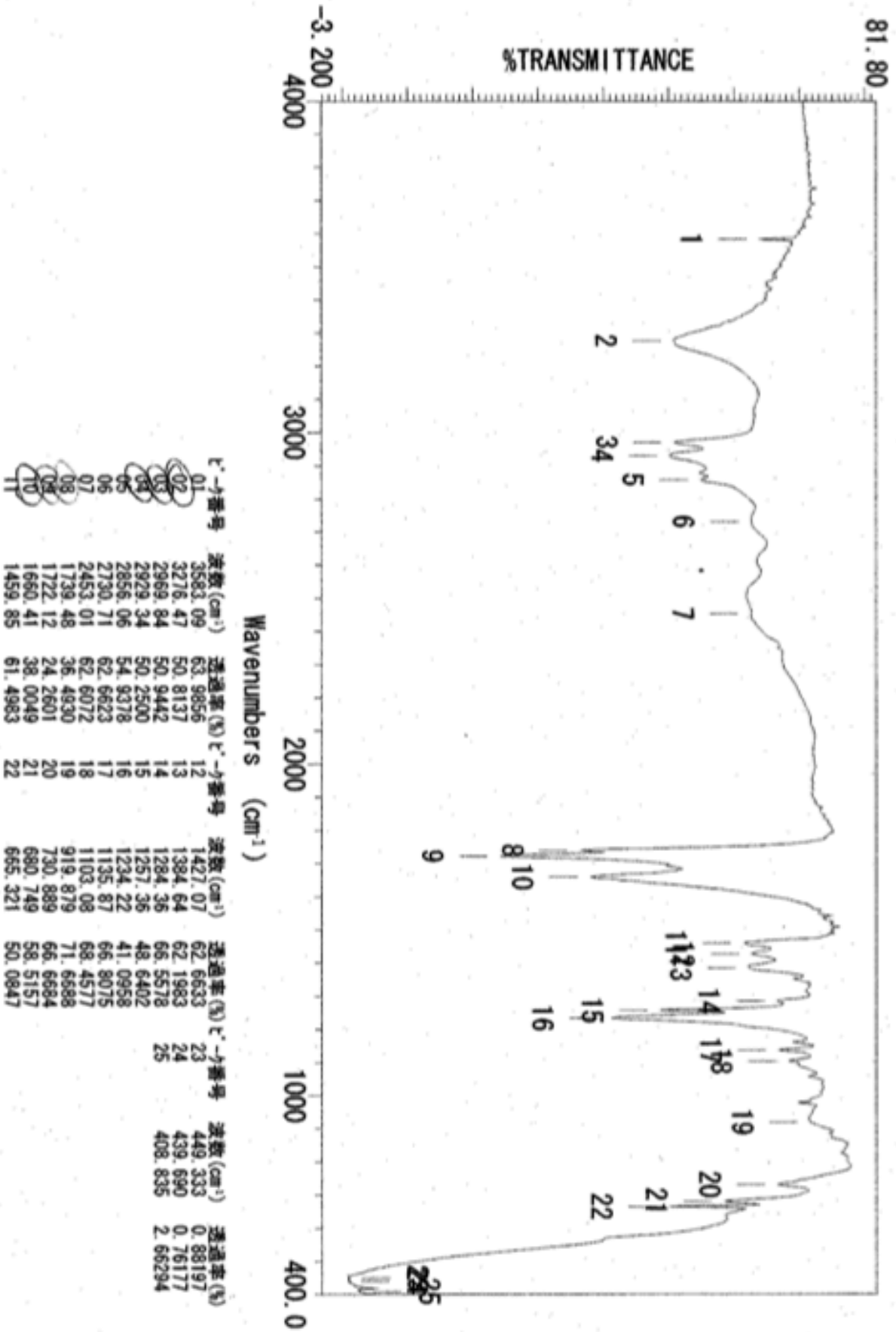


Supplementary Figure S5.  $^1\text{H}$  NMR spectrum of **1b**.



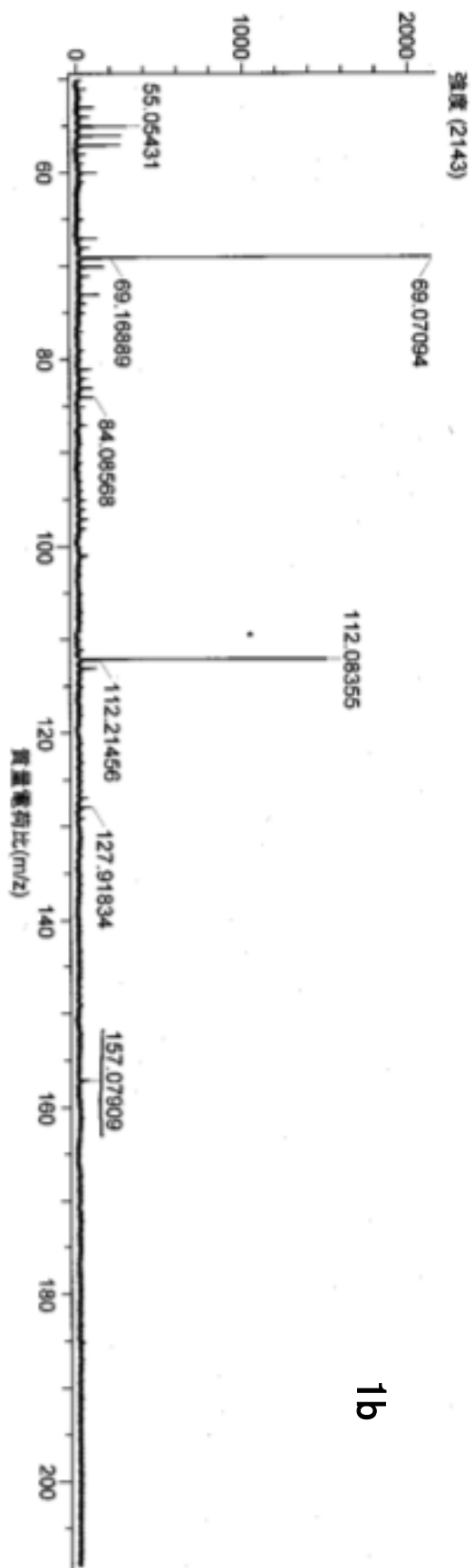
Supplementary Figure S6.  $^{13}\text{C}$  NMR spectrum of **1b**.

1b



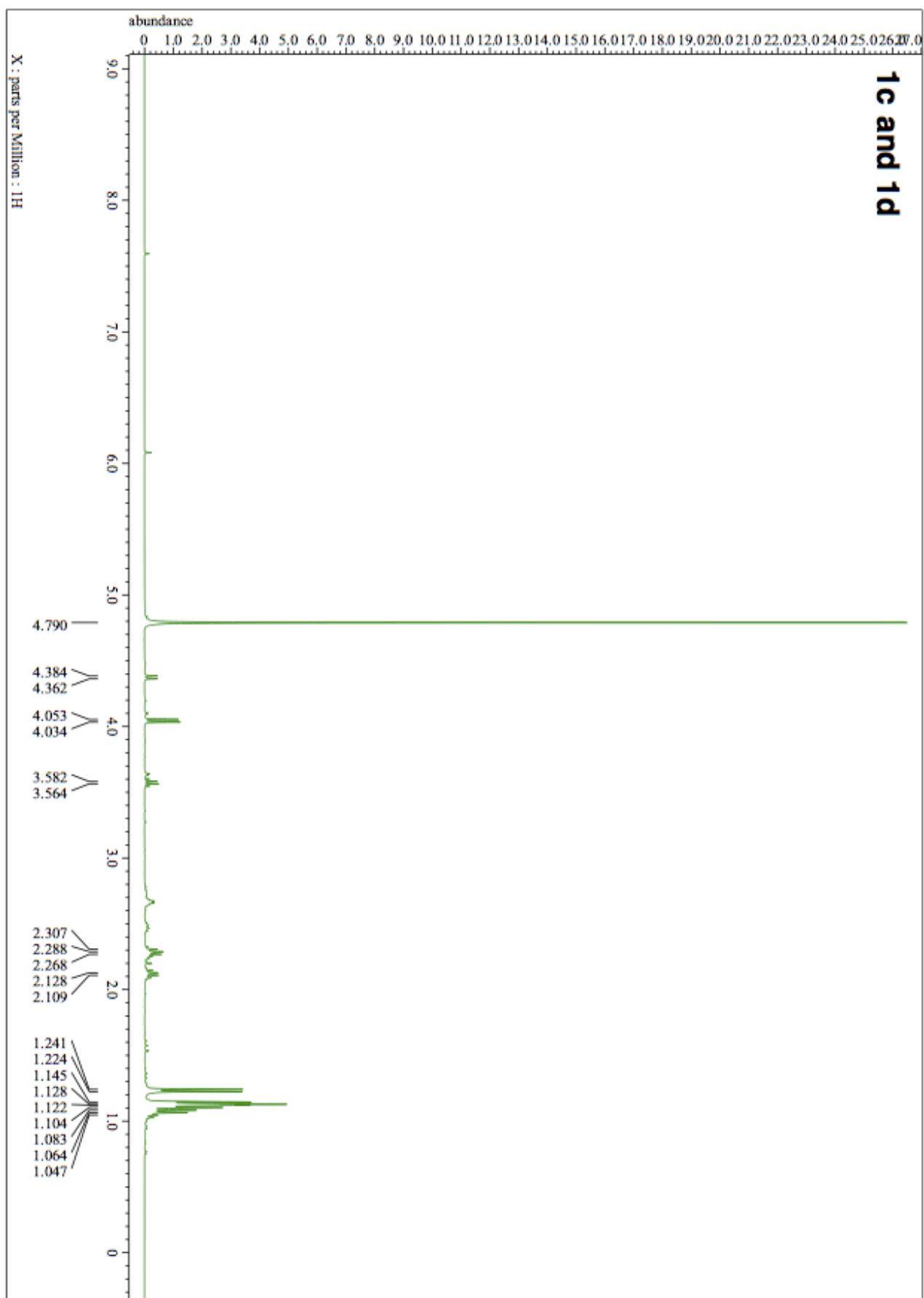
Supplementary Figure S7. IR spectrum of 1b.



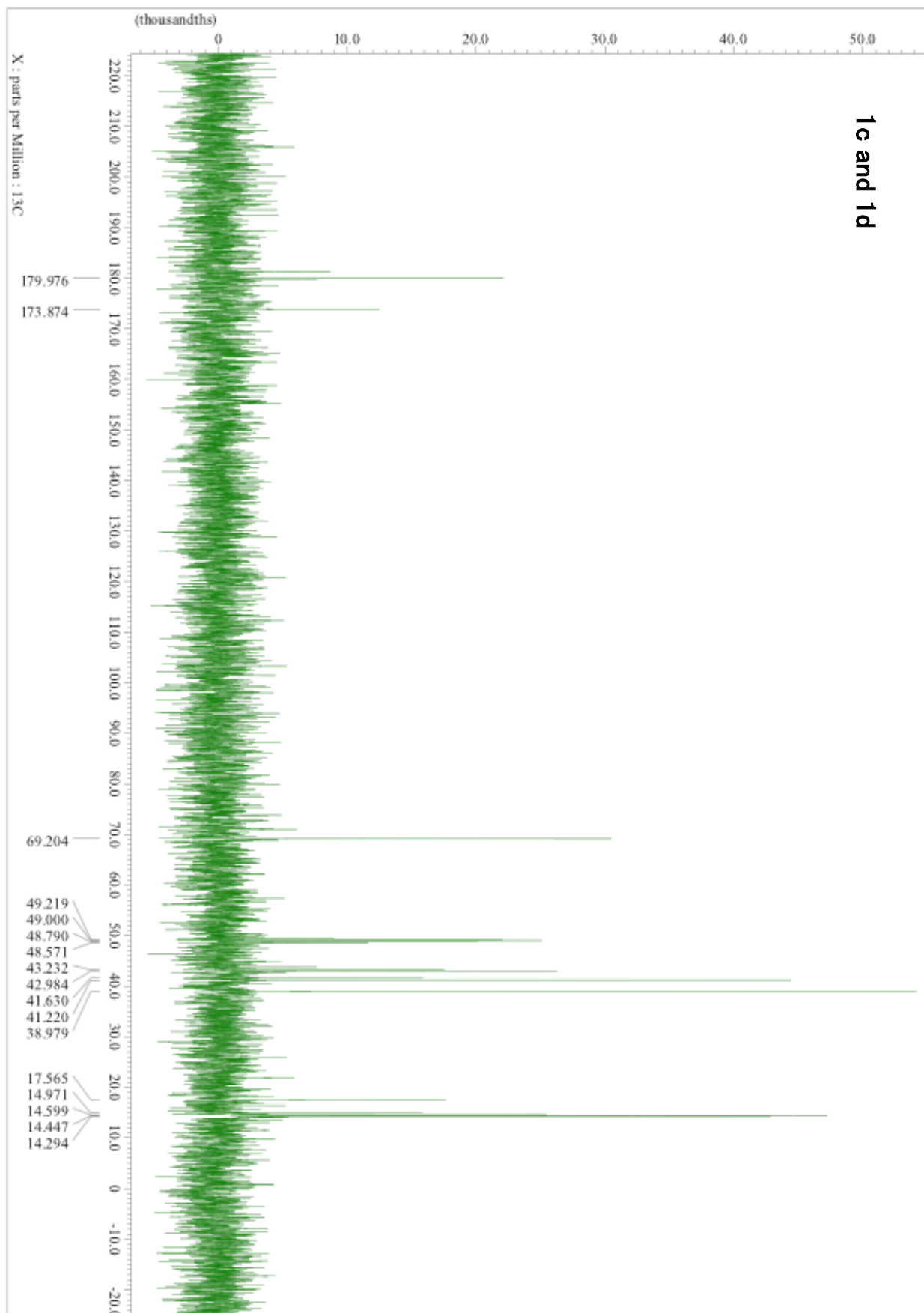


**1b**

Supplementary Figure S8. MS spectrum of **1b**.

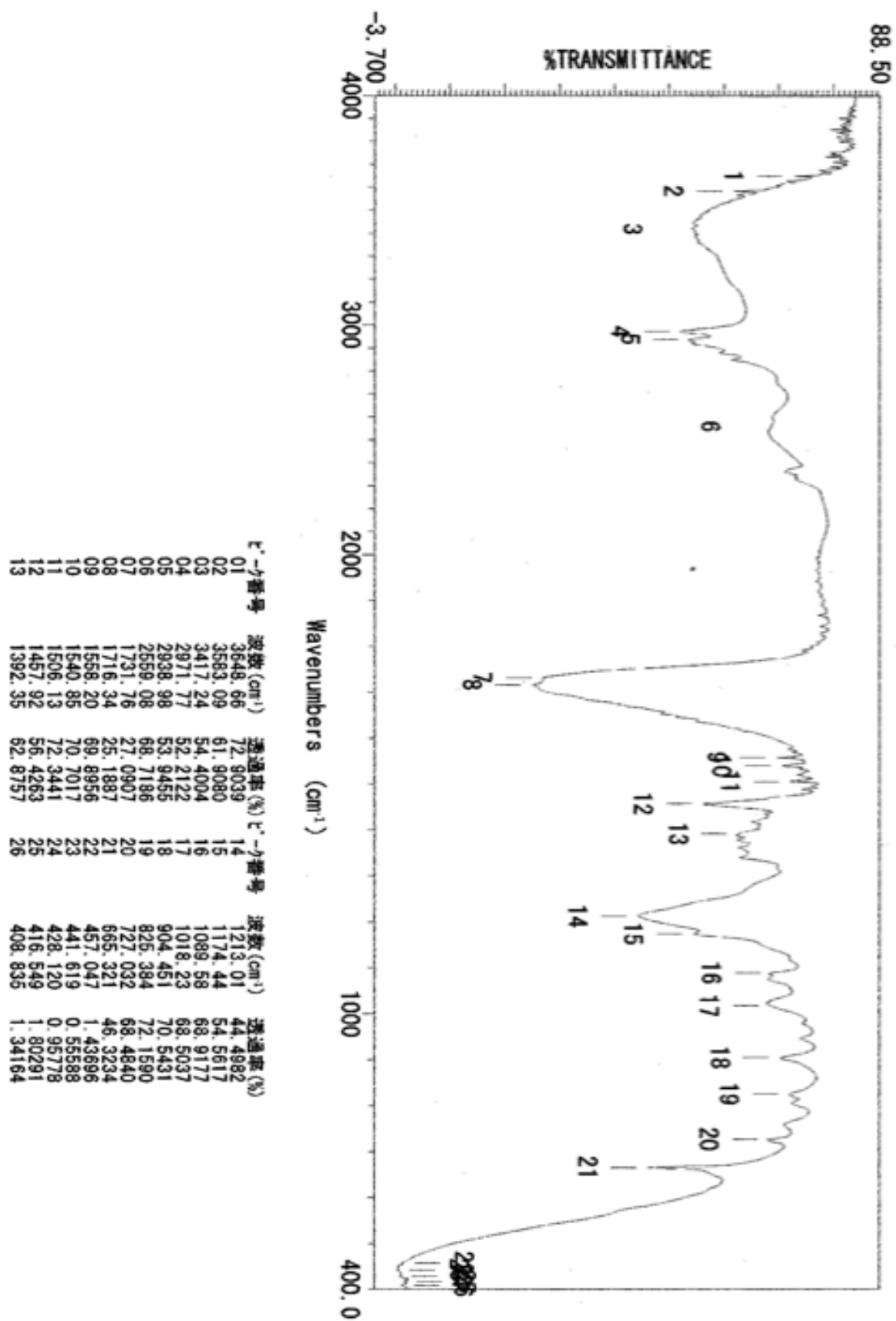


Supplementary Figure S9.  $^1\text{H}$  NMR spectrum of mixture of **1c** and **1d**.

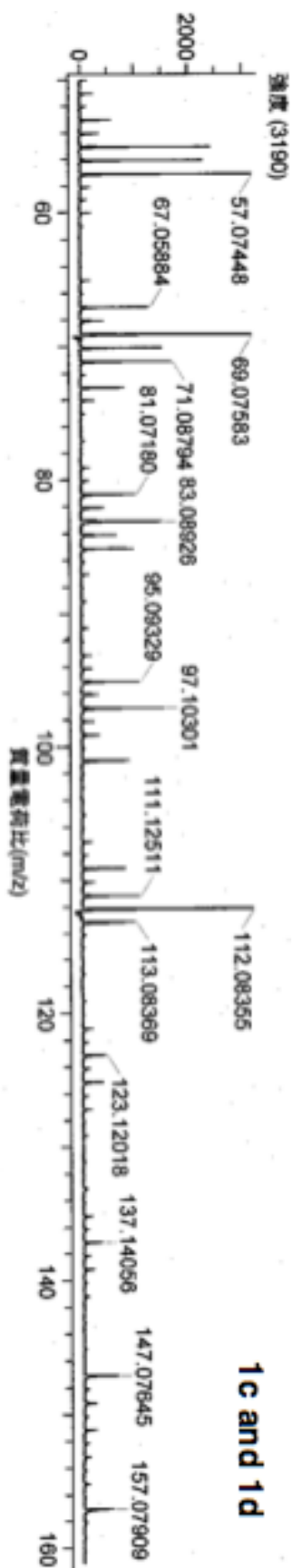


Supplementary Figure S10.  $^{13}\text{C}$  NMR spectrum of mixture of **1c** and **1d**.

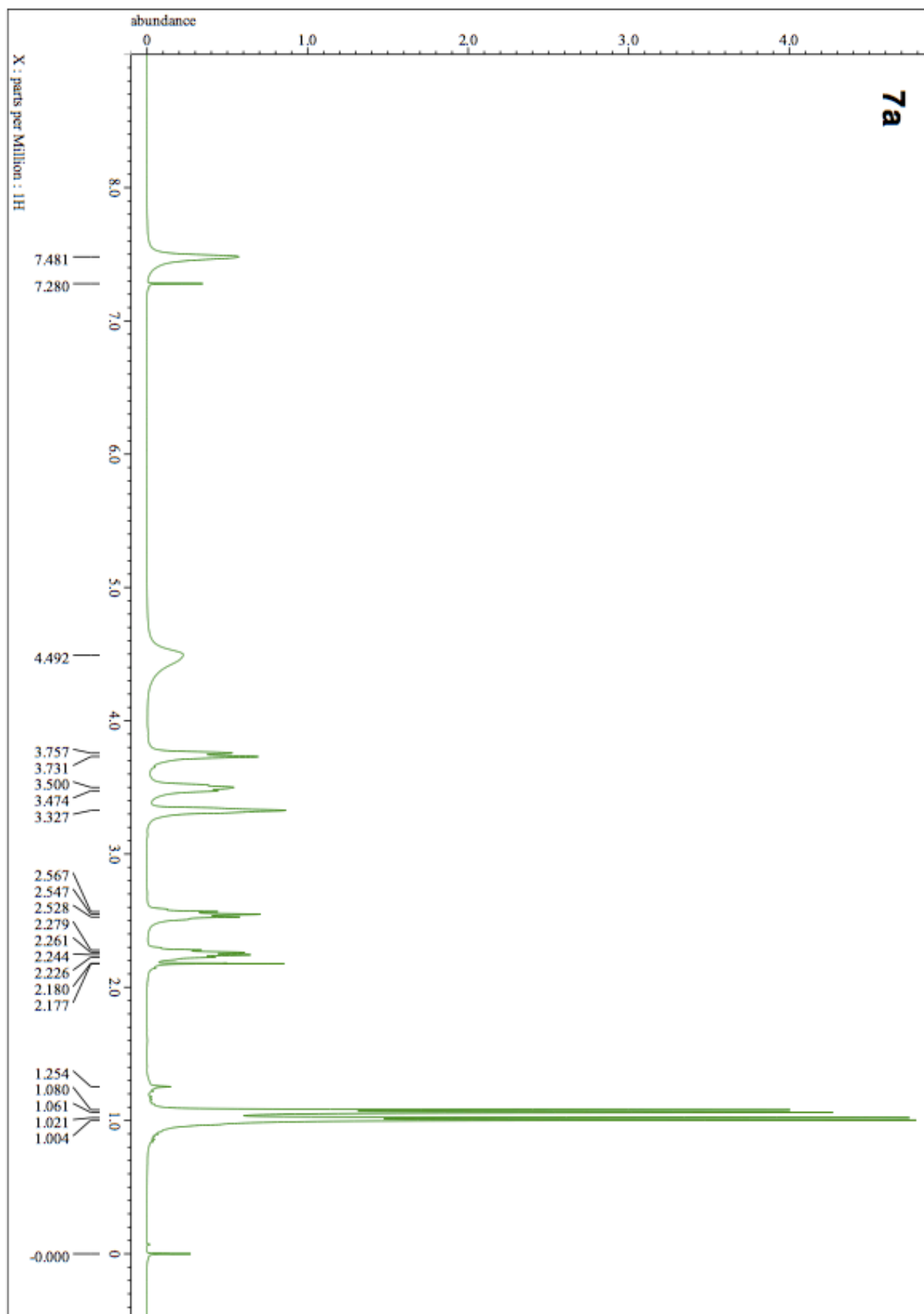
1c and 1d



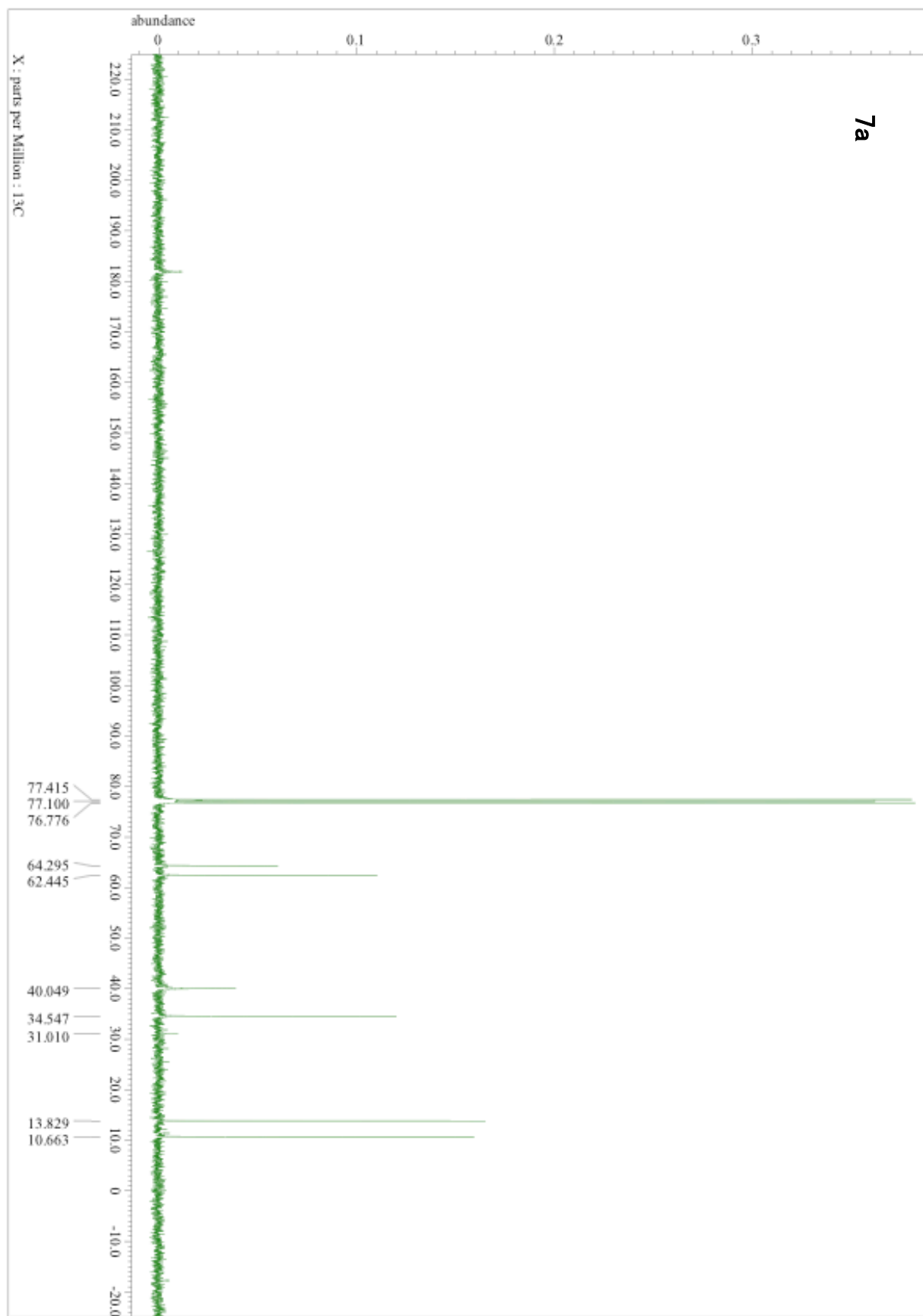
Supplementary Figure S11. IR spectrum of mixture of 1c and 1d.



Supplementary Figure S12. MS spectrum of mixture of **1c** and **1d**.

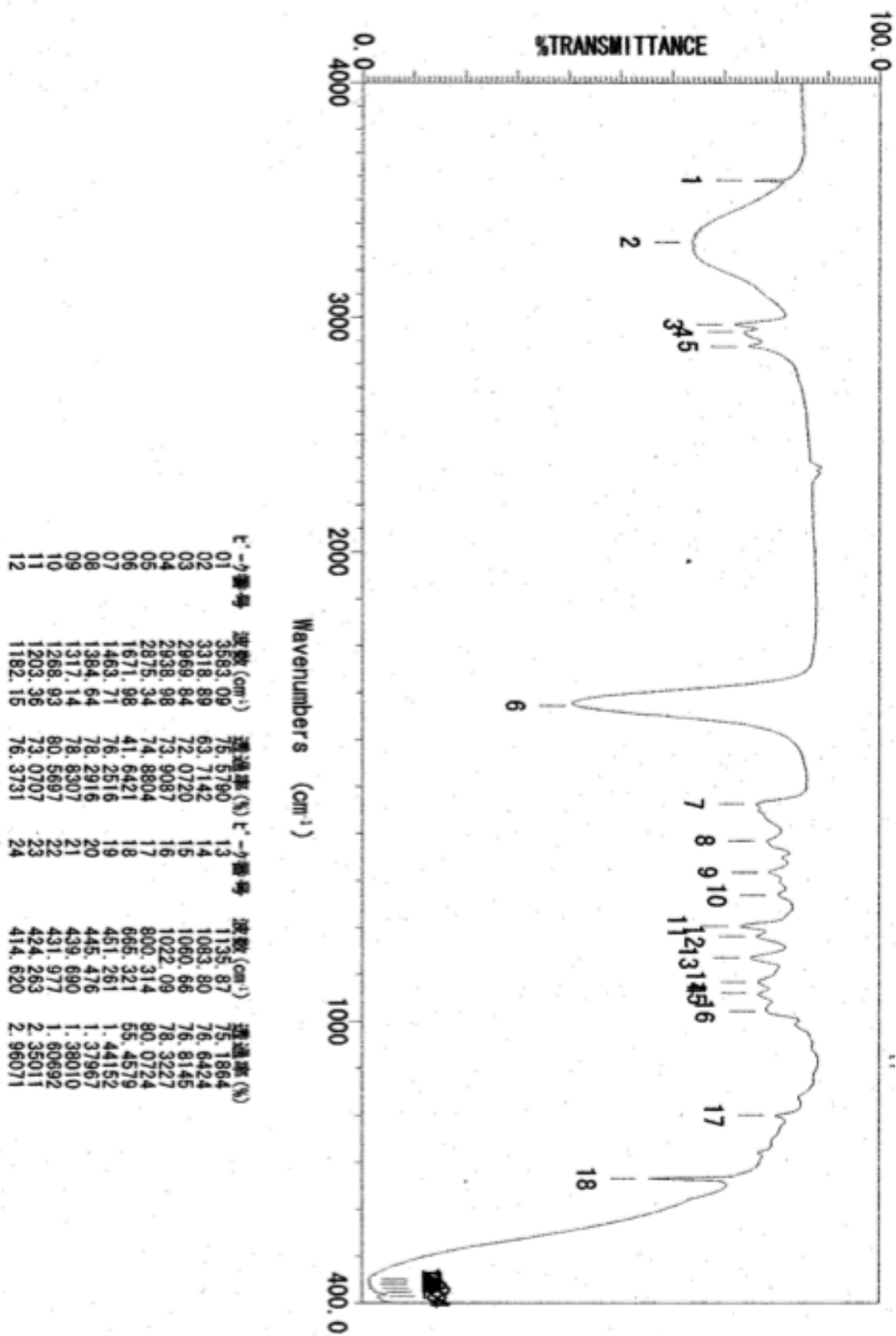


Supplementary Figure S13.  $^1\text{H}$  NMR spectrum of **7a**.



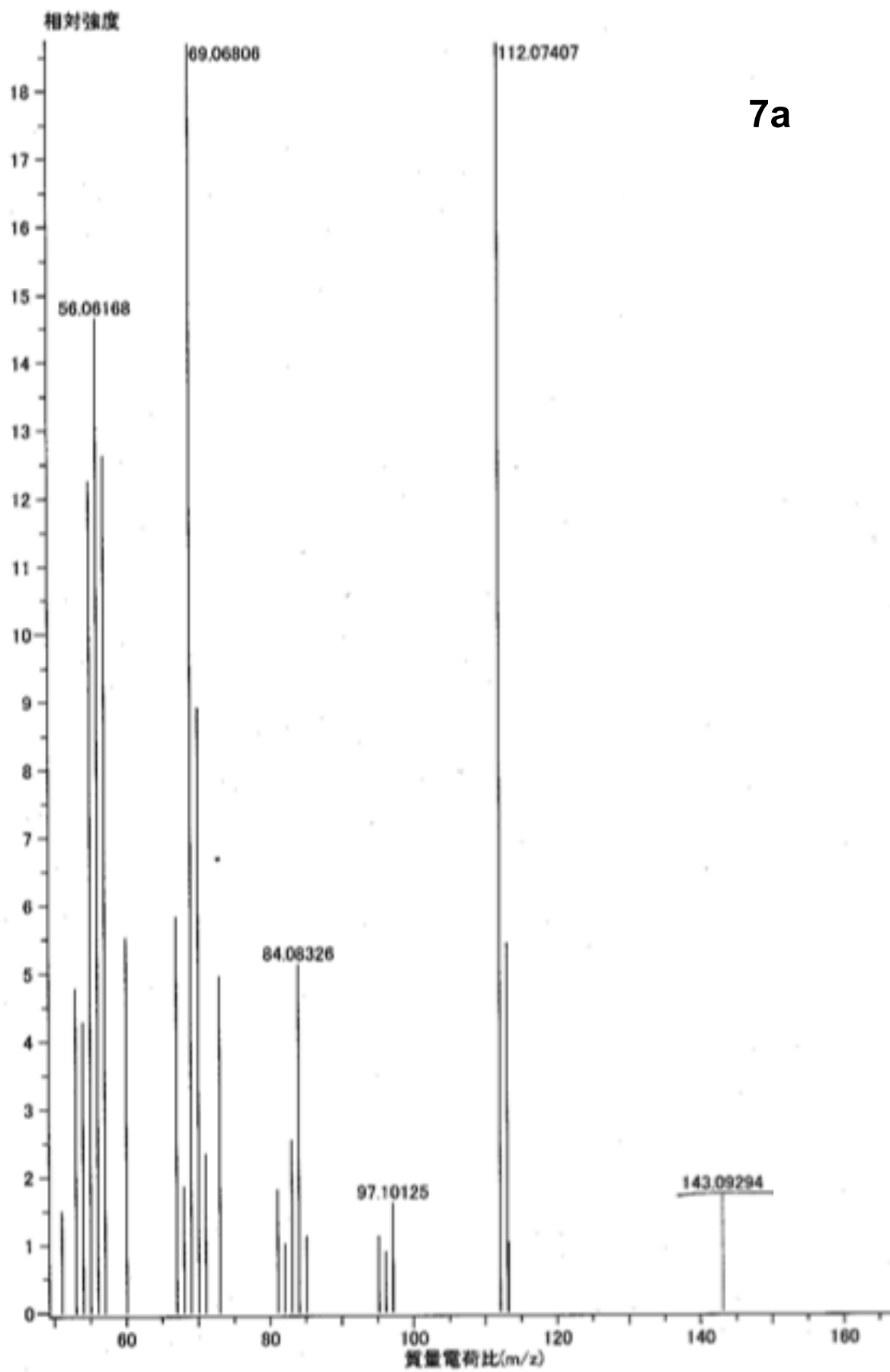
Supplementary Figure S14.  $^{13}\text{C}$  NMR spectrum of **7a**.

7a



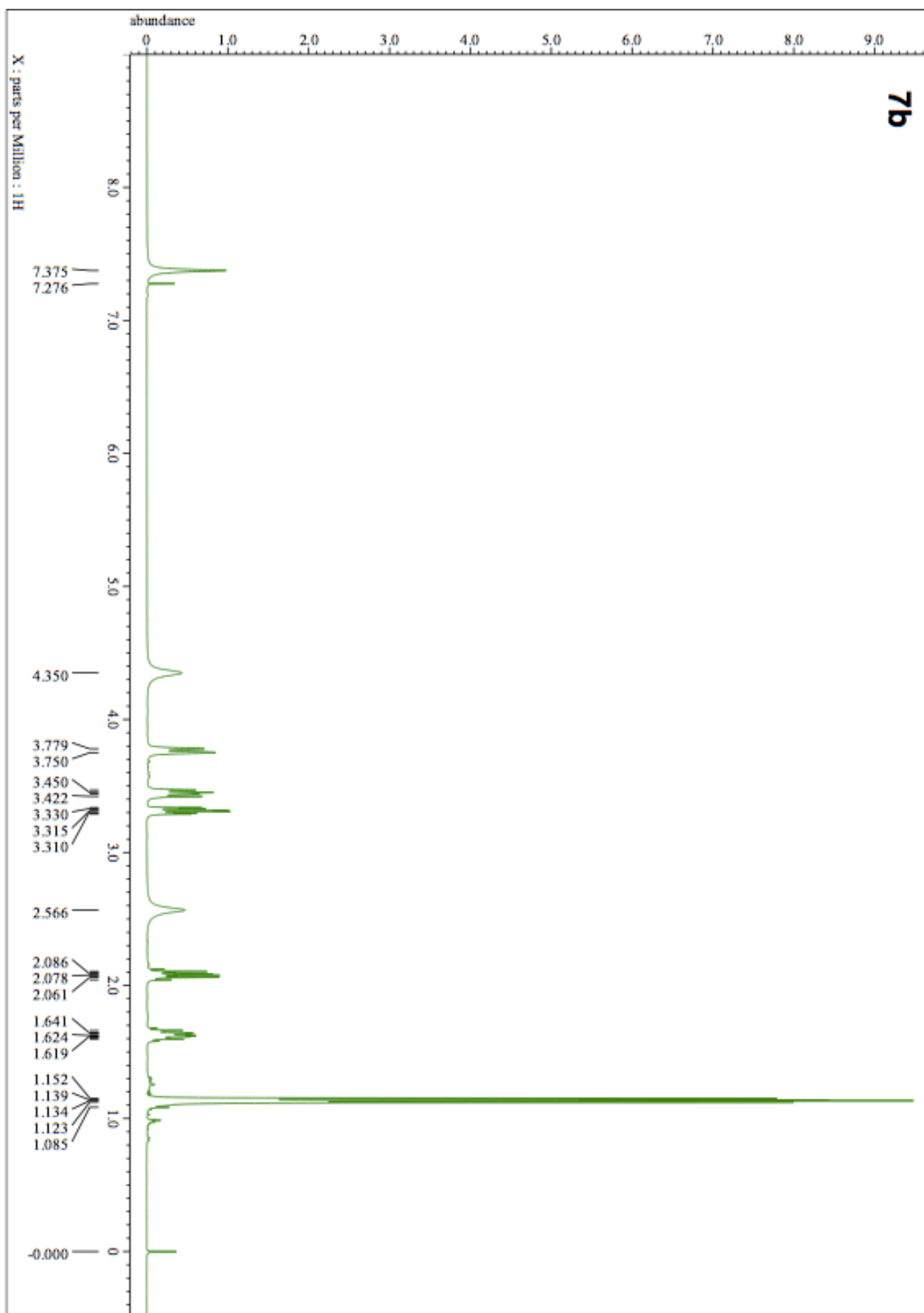
Supplementary Figure S15. IR spectrum of 7a.



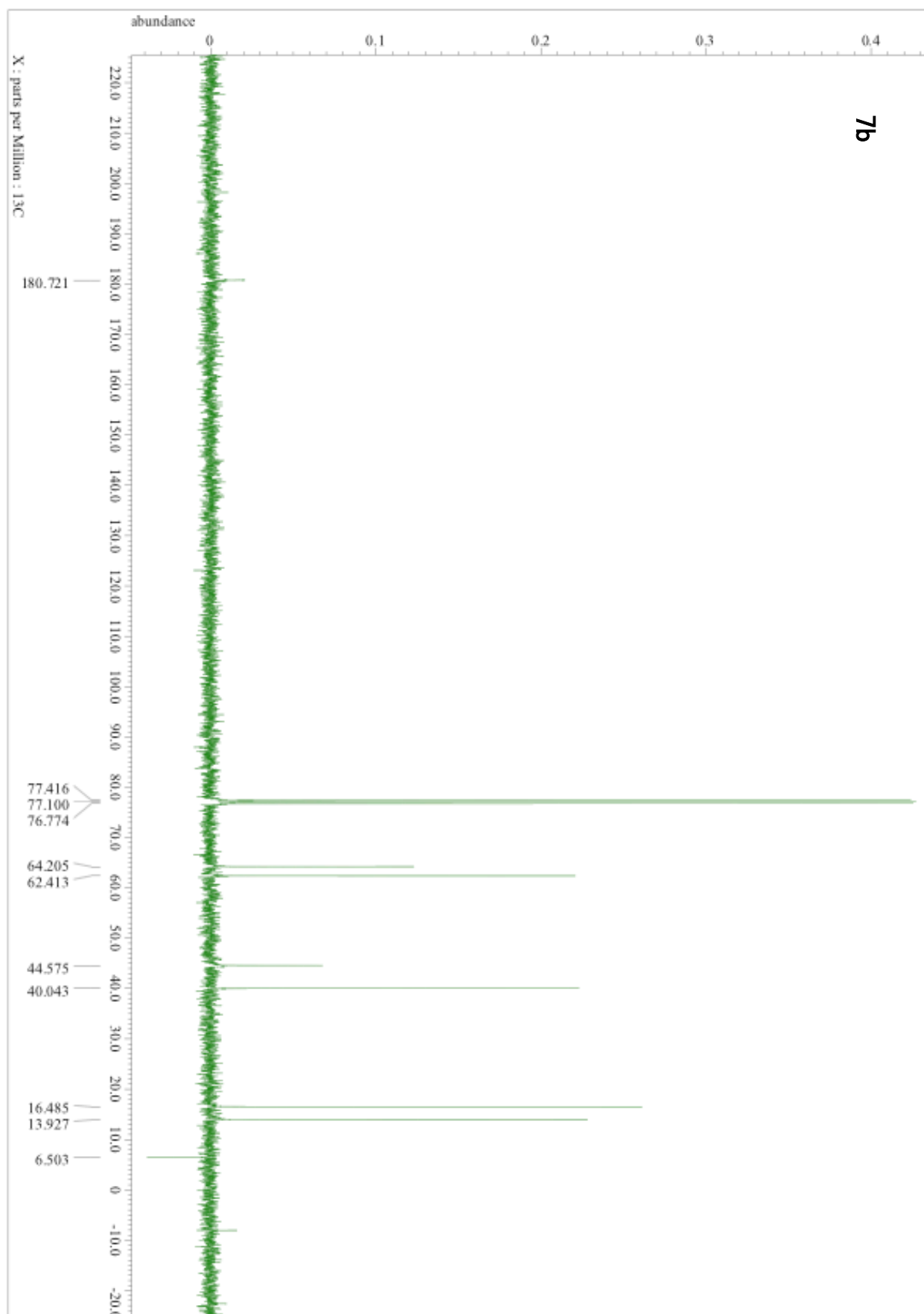


7a

Supplementary Figure S16. MS spectrum of 7a.

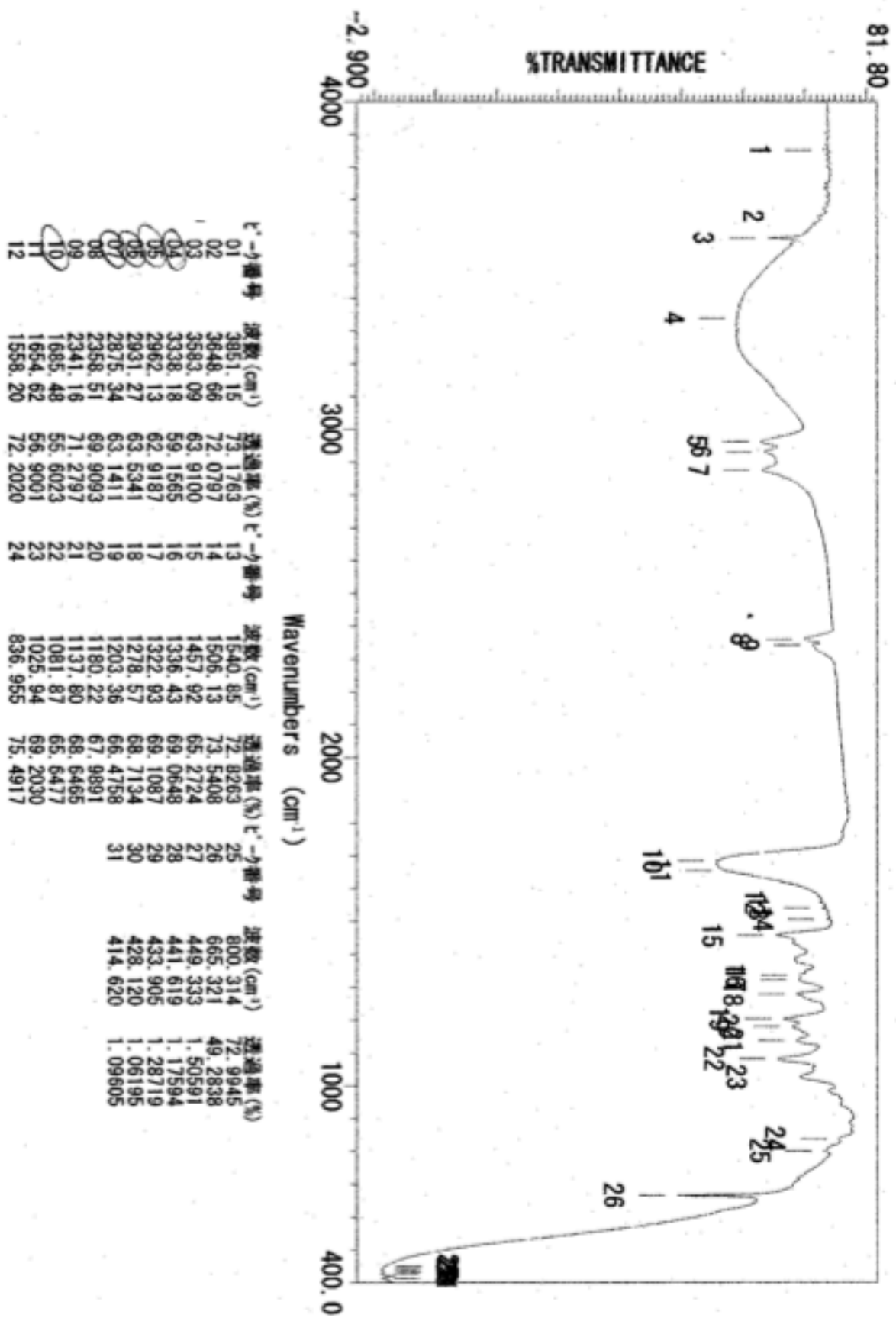


Supplementary Figure S17.  $^1\text{H}$  NMR spectrum of **7b**.



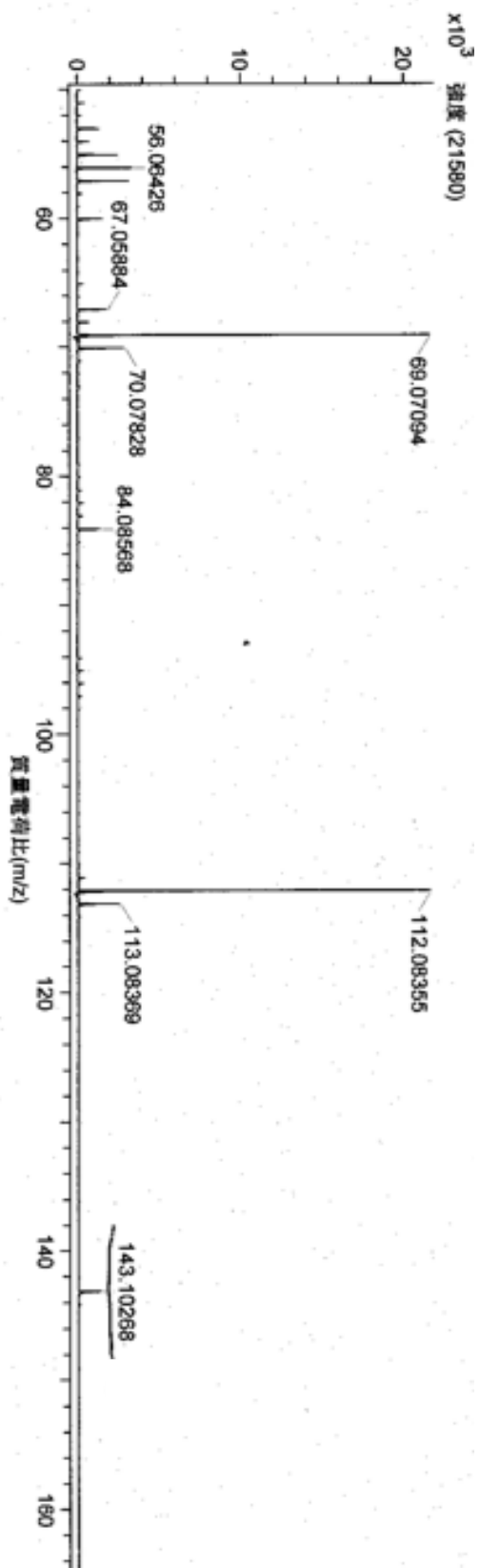
Supplementary Figure S18.  $^{13}\text{C}$  NMR spectrum of **7b**.

7b

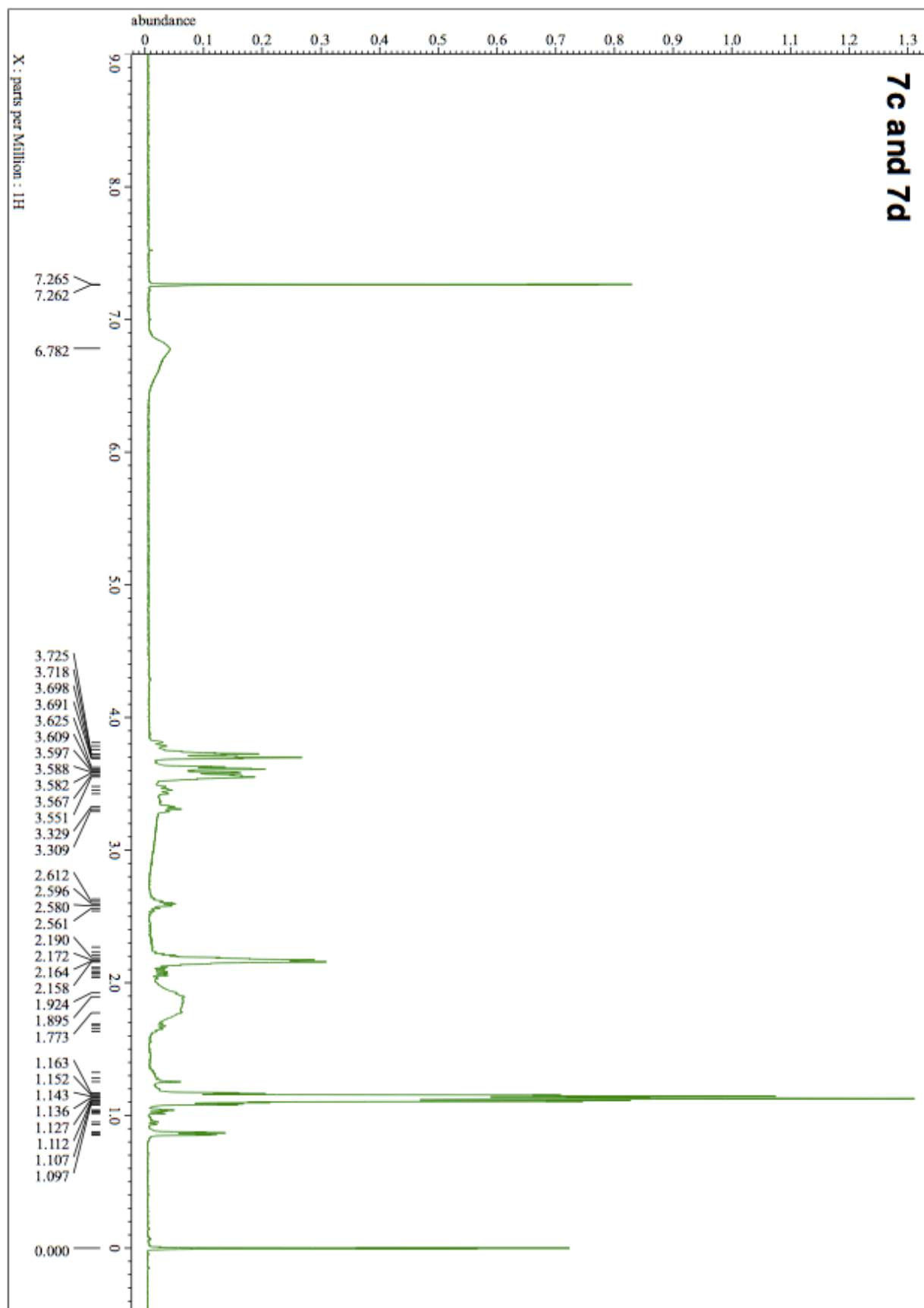


Supplementary Figure S19. IR spectrum of 7b.

7b

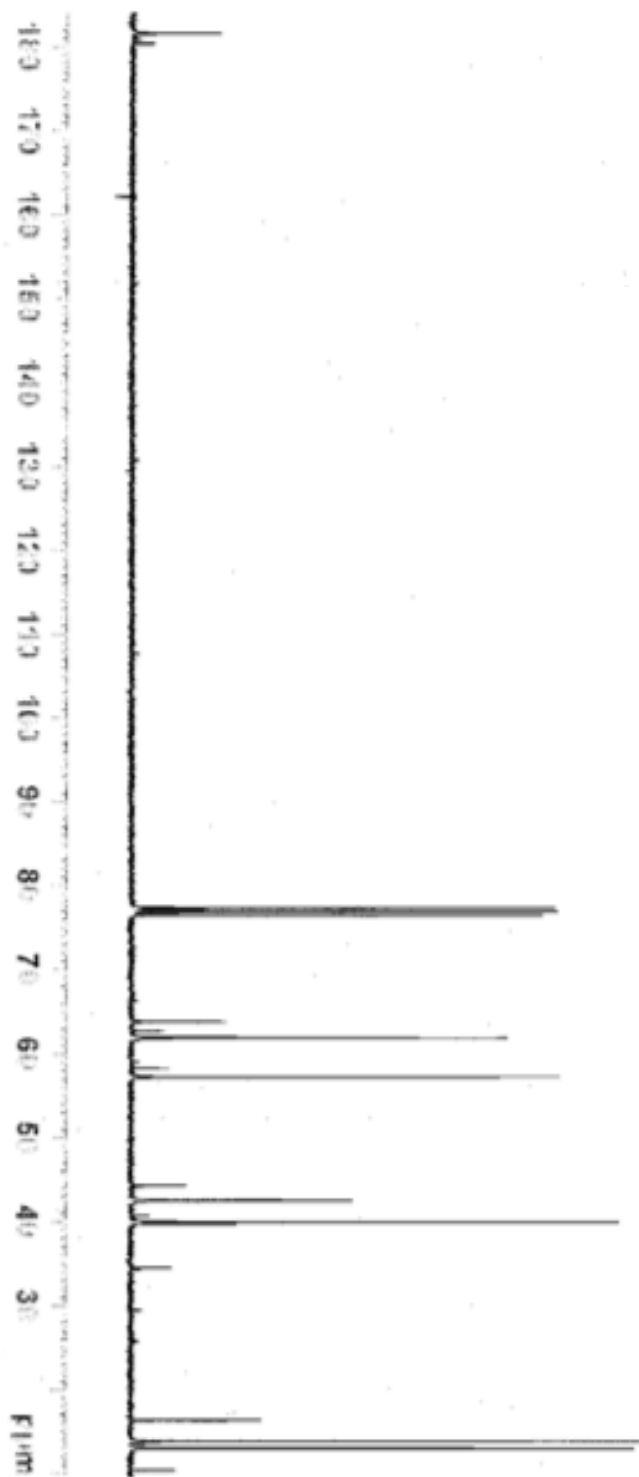


Supplementary Figure S20. MS spectrum of 7b.



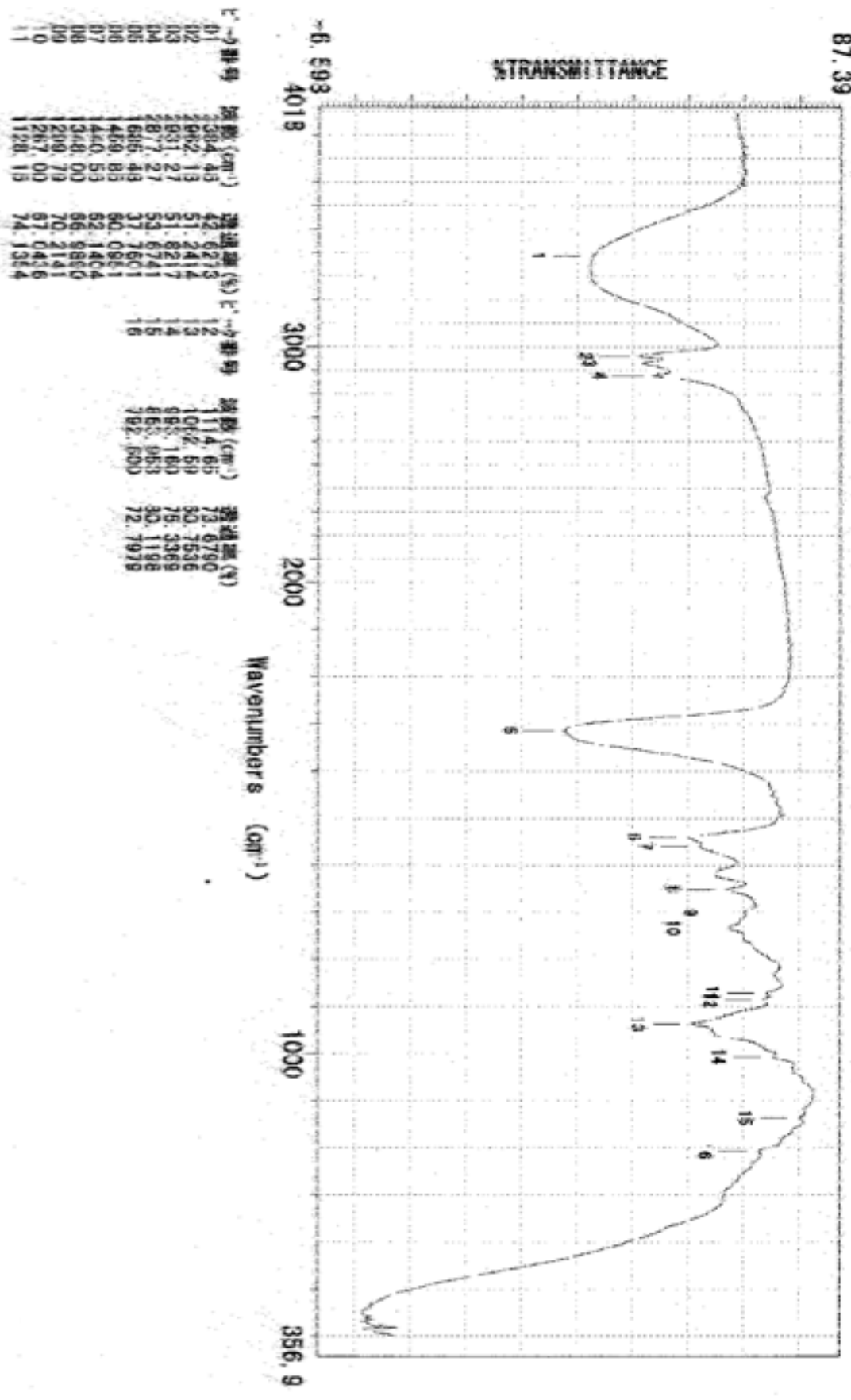
Supplementary Figure S21.  $^1\text{H}$  NMR spectrum of mixture of **7c** and **7d**.

# 7c and 7d



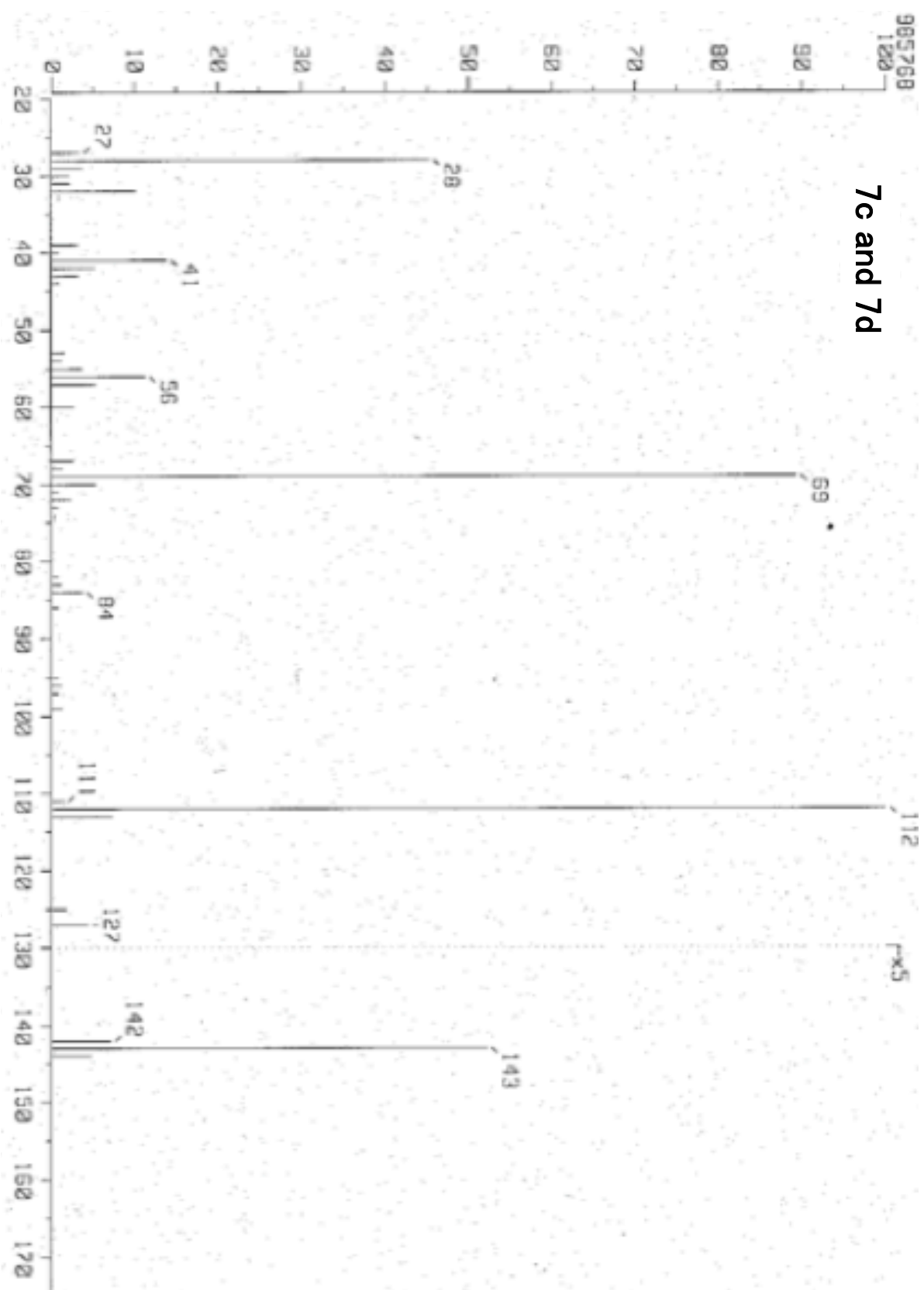
Supplementary Figure S22.  $^{13}\text{C}$  NMR spectrum of mixture of 7c and 7d.

87.39  
7c and 7d



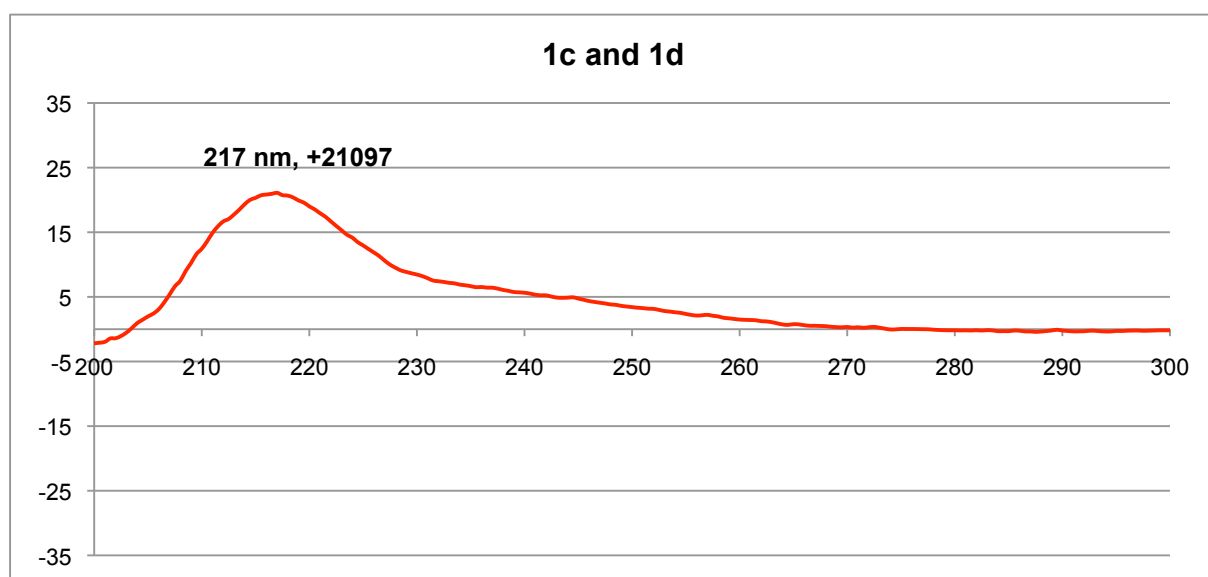
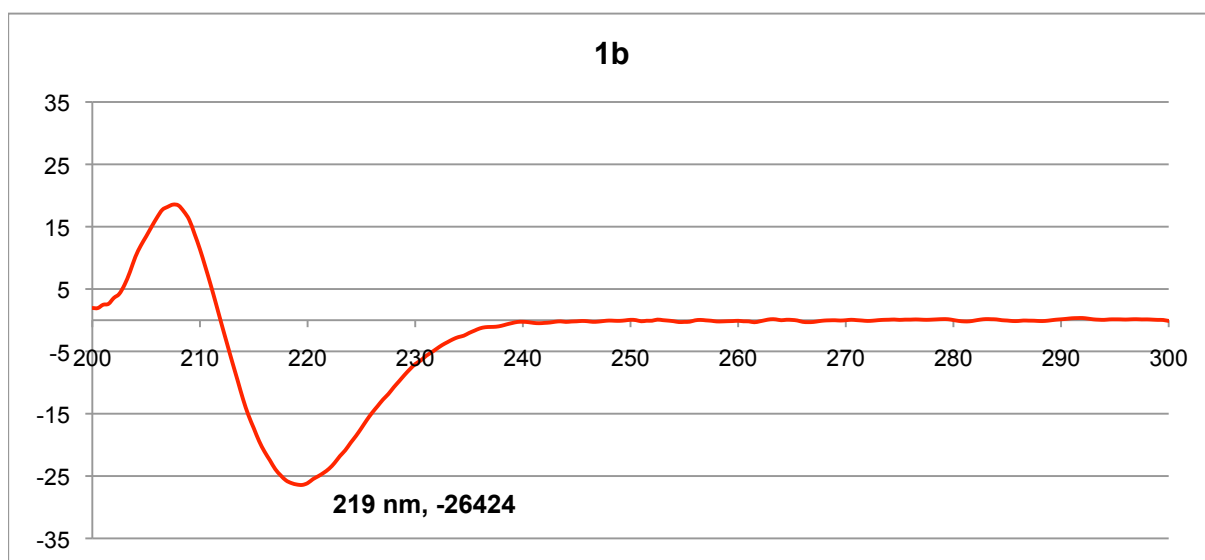
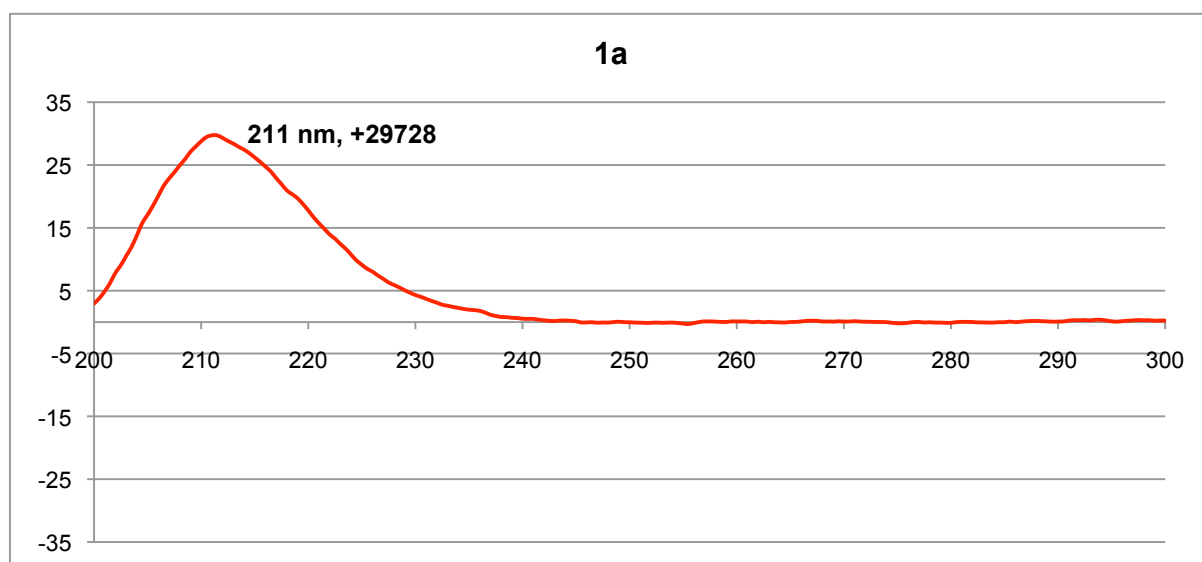
Supplementary Figure S23. IR spectrum of mixture of 7c and 7d.





7c and 7d

Supplementary Figure S24. MS spectrum of mixture of 7c and 7d.



Supplementary Figure S25. CD spectra of **1a**, **1b**, and mixture of **1c** and **1d**.