

Supplementary Material

Antitumor thiodiketopiperazine derivatives from the deep sea-derived fungus *Epicoccum nigrum* SD-388

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Figure S1. ¹H NMR (500 MHz, DMSO-*d*₆) spectrum of compound **1**.

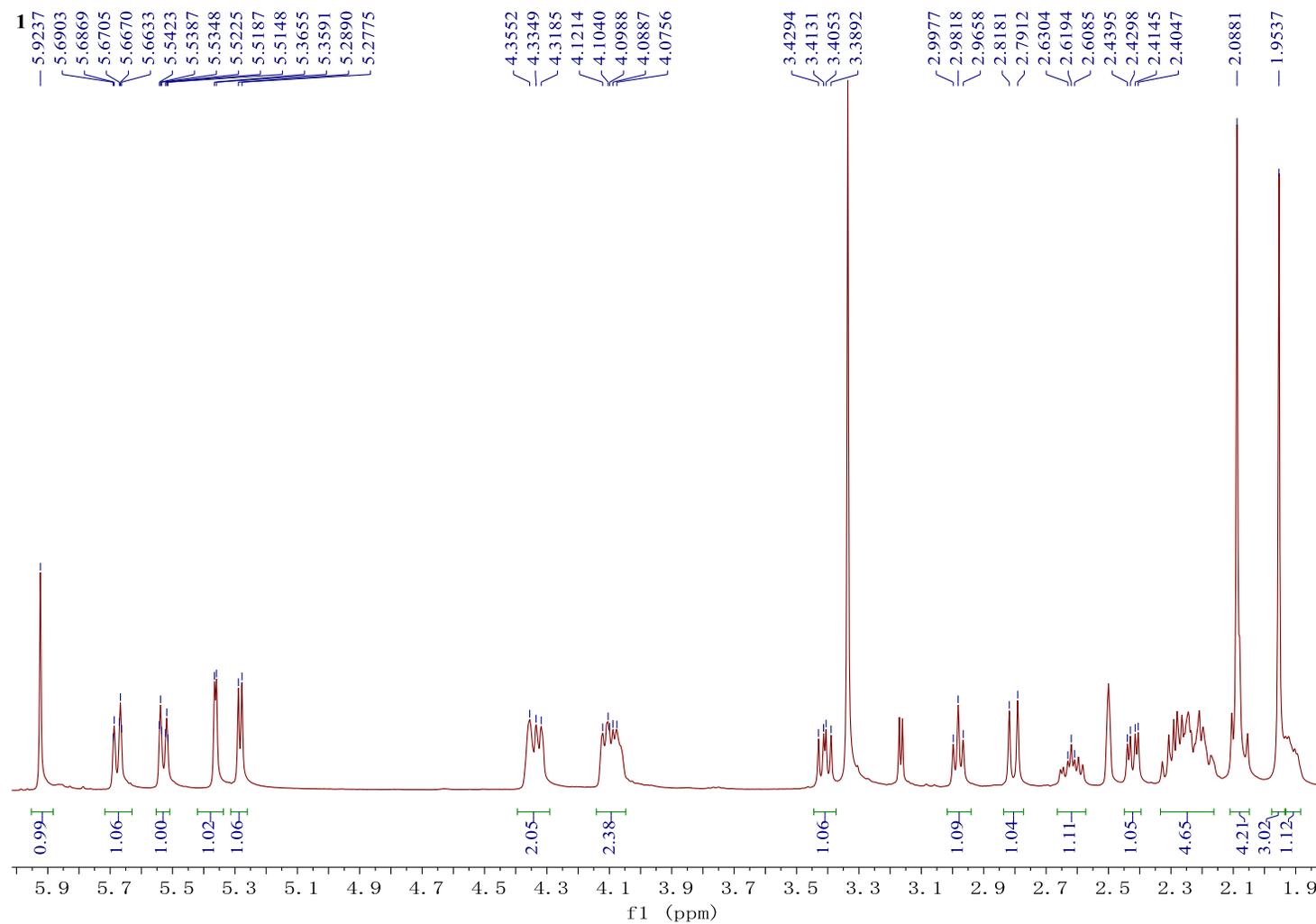


Figure S2. ^{13}C NMR and DEPT (125 MHz, $\text{DMSO}-d_6$) spectrum of compound **1**.

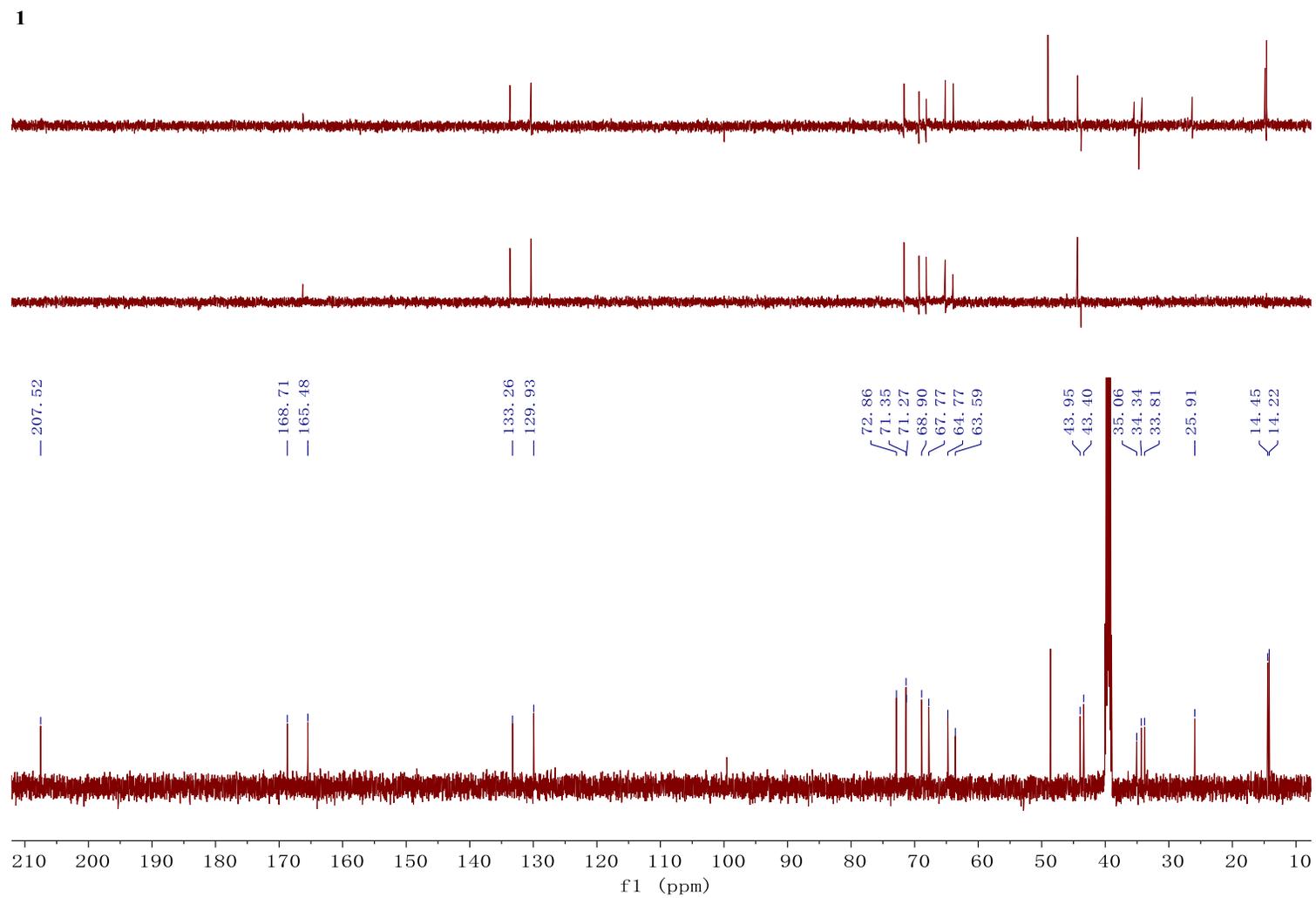


Figure S3. COSY spectrum of compound 1.

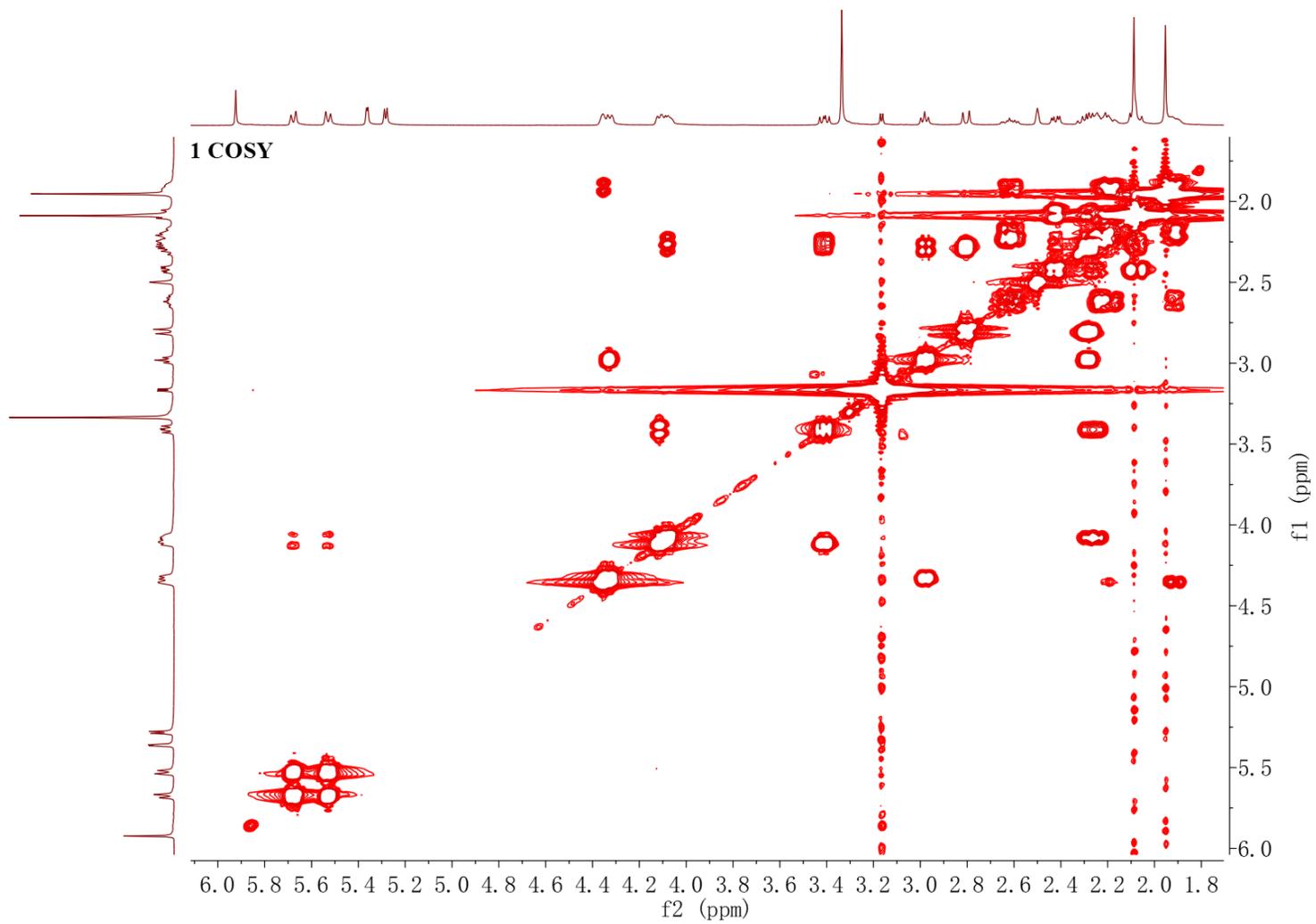


Figure S4. HMBC spectrum of compound **1**.

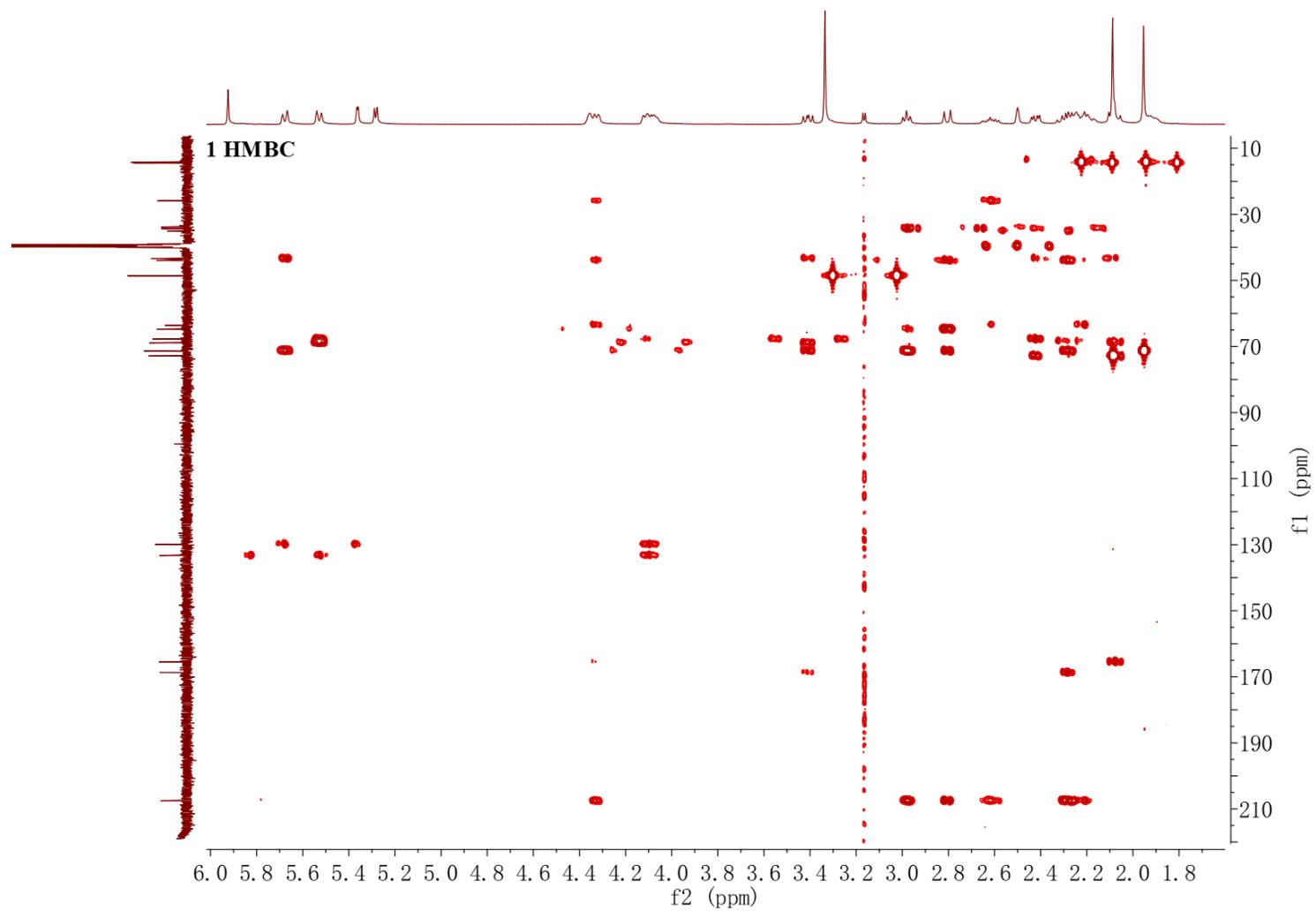


Figure S5. NOESY spectrum of compound 1.

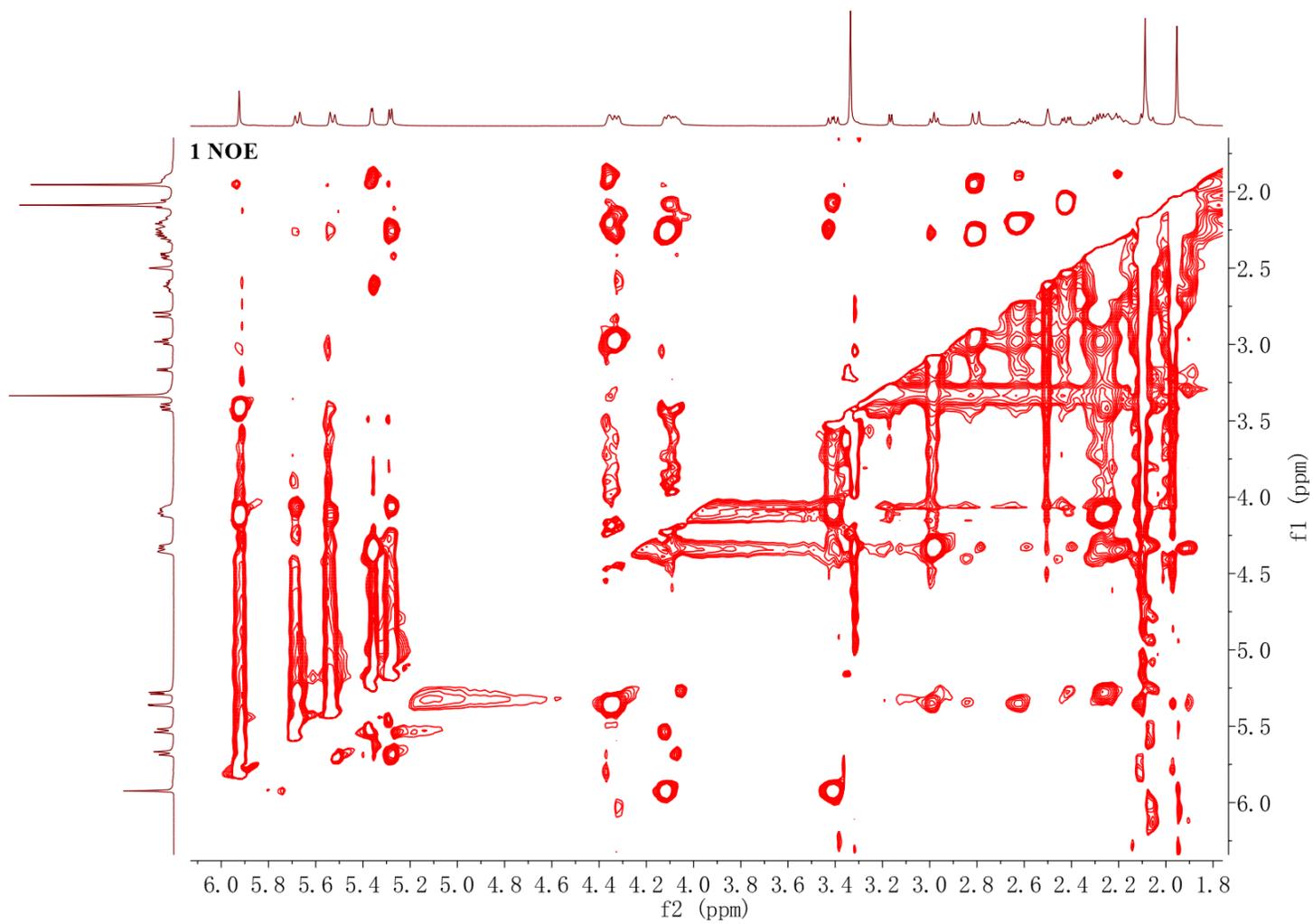


Figure S6. HR-ESI-MS of compound 1.

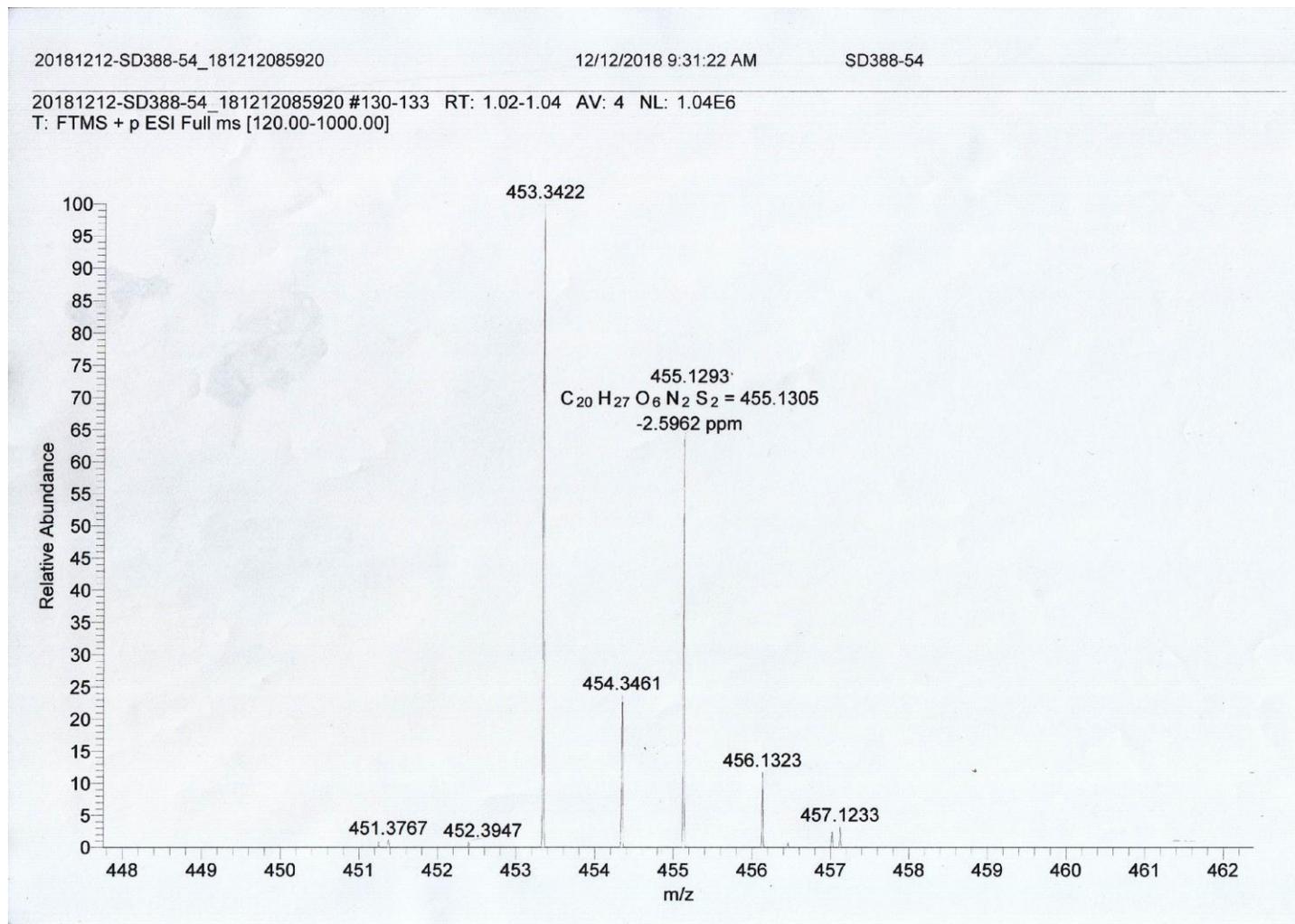


Figure S7. Crystal packing of compound 1 at 293(2) K;

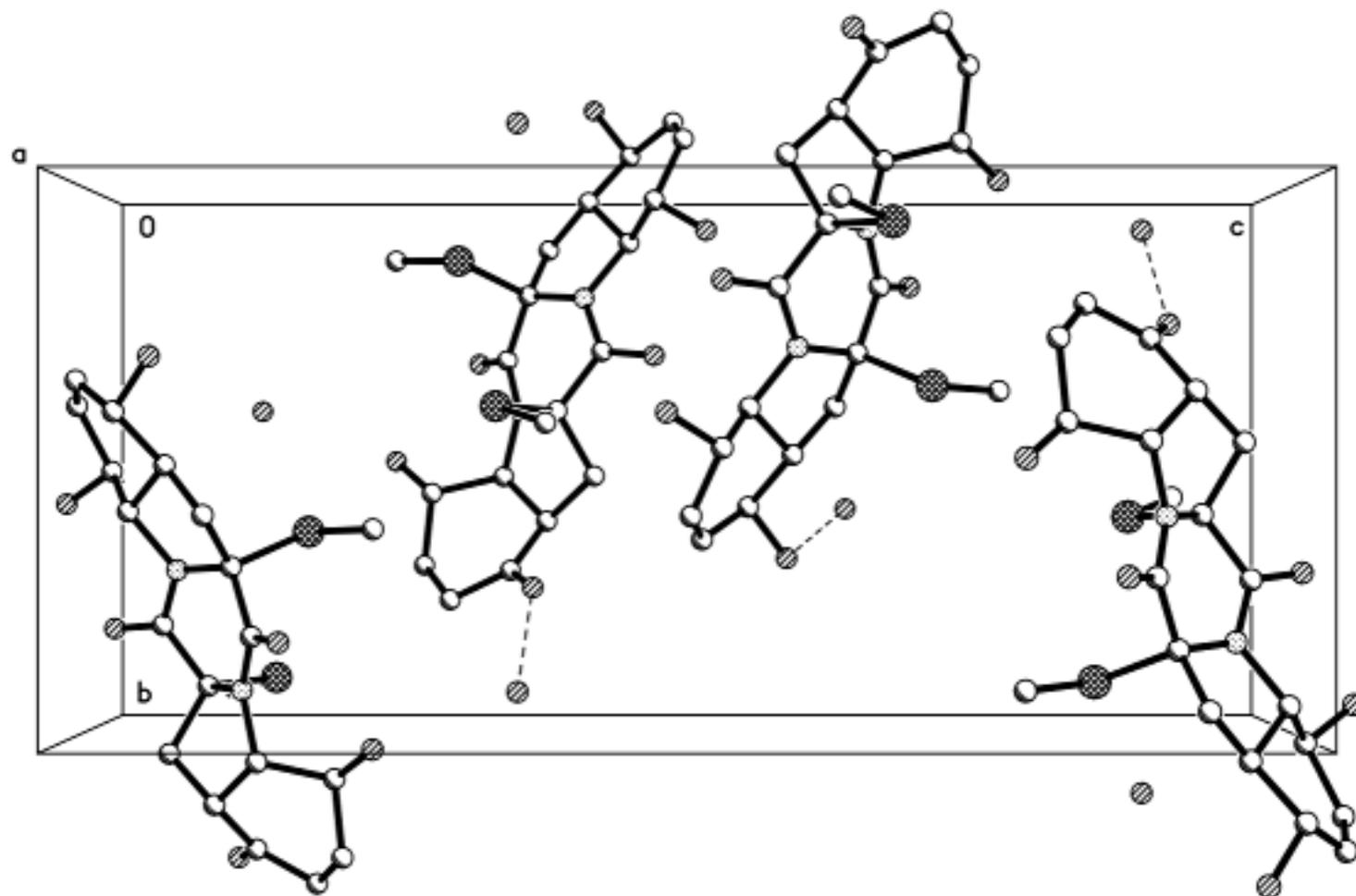


Figure S8. ^1H NMR (500 MHz, $\text{DMSO-}d_6$) spectrum of compound **2**.

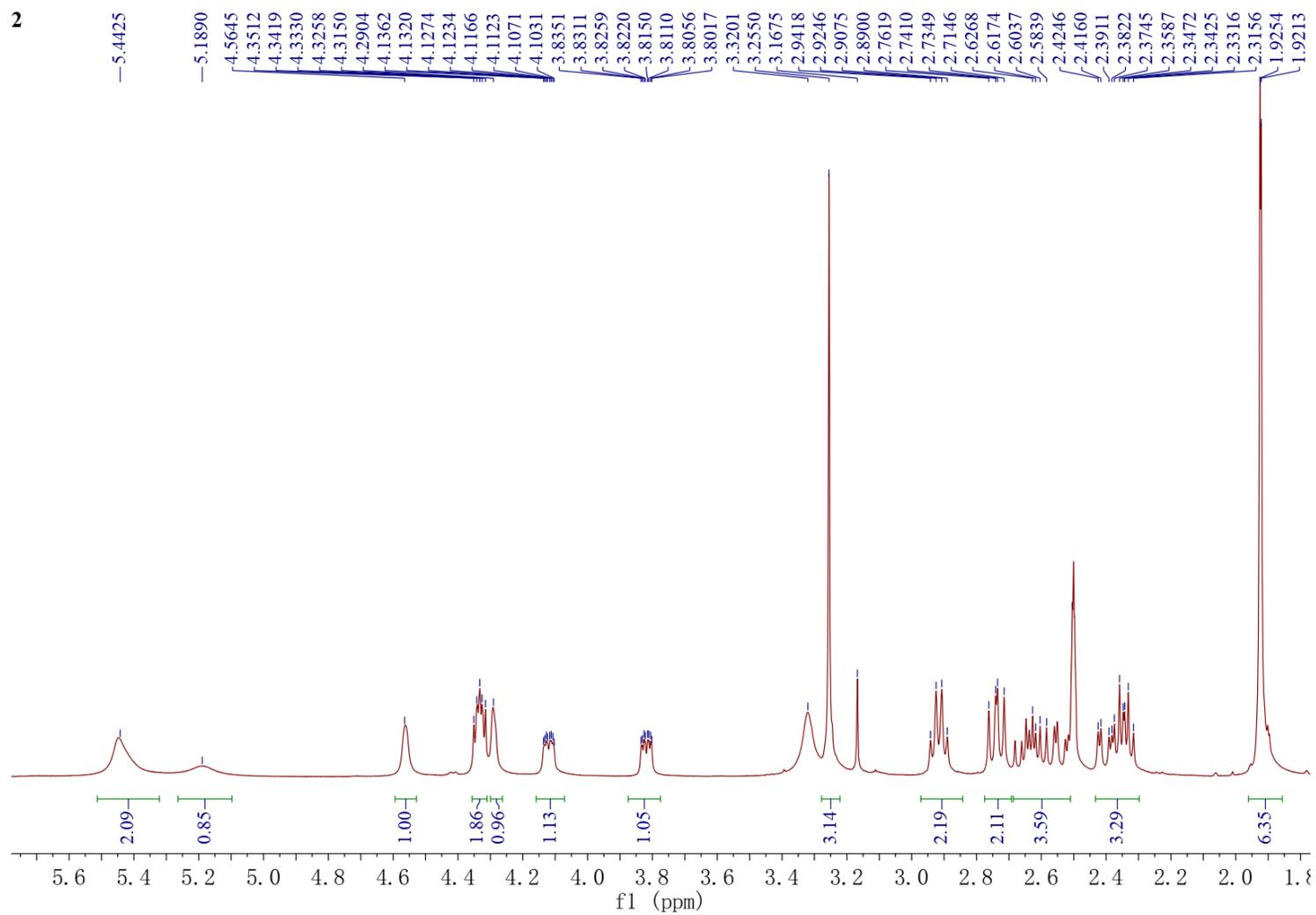


Figure S9. ^{13}C NMR and DEPT (125 MHz, $\text{DMSO-}d_6$) spectrum of compound **2**.

2

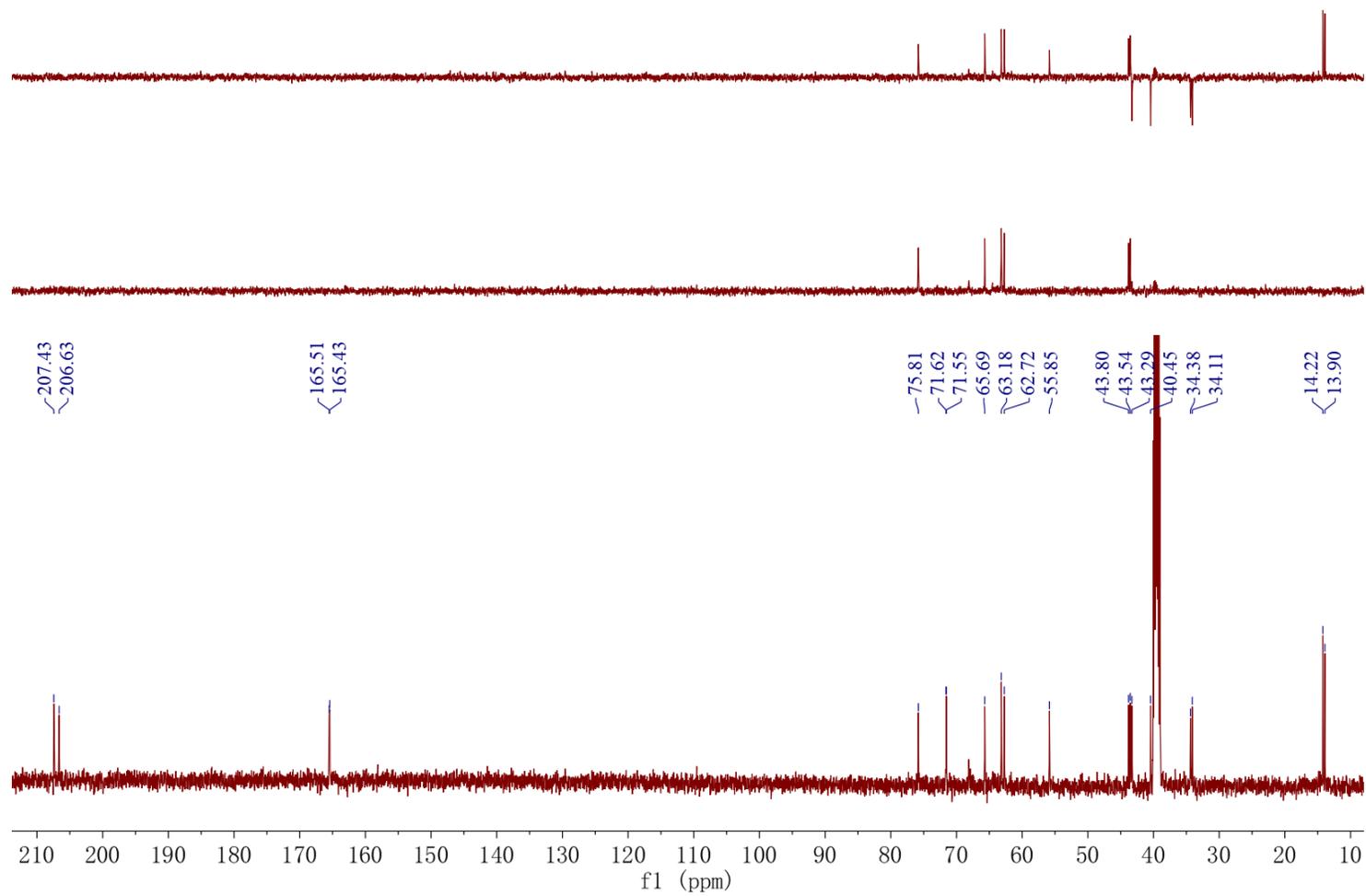


Figure S10. COSY spectrum of compound 2.

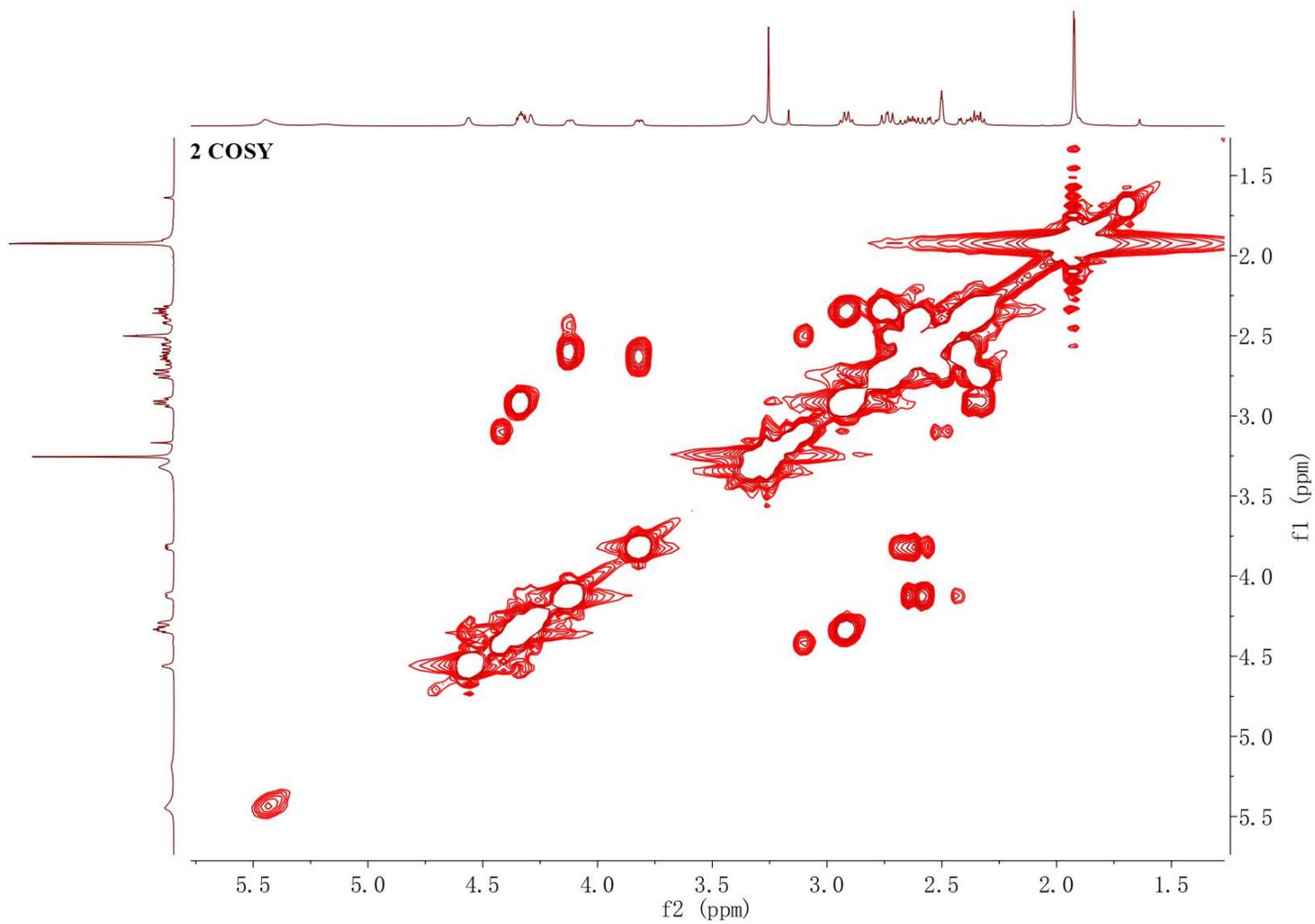


Figure S11. HSQC spectrum of compound 2.

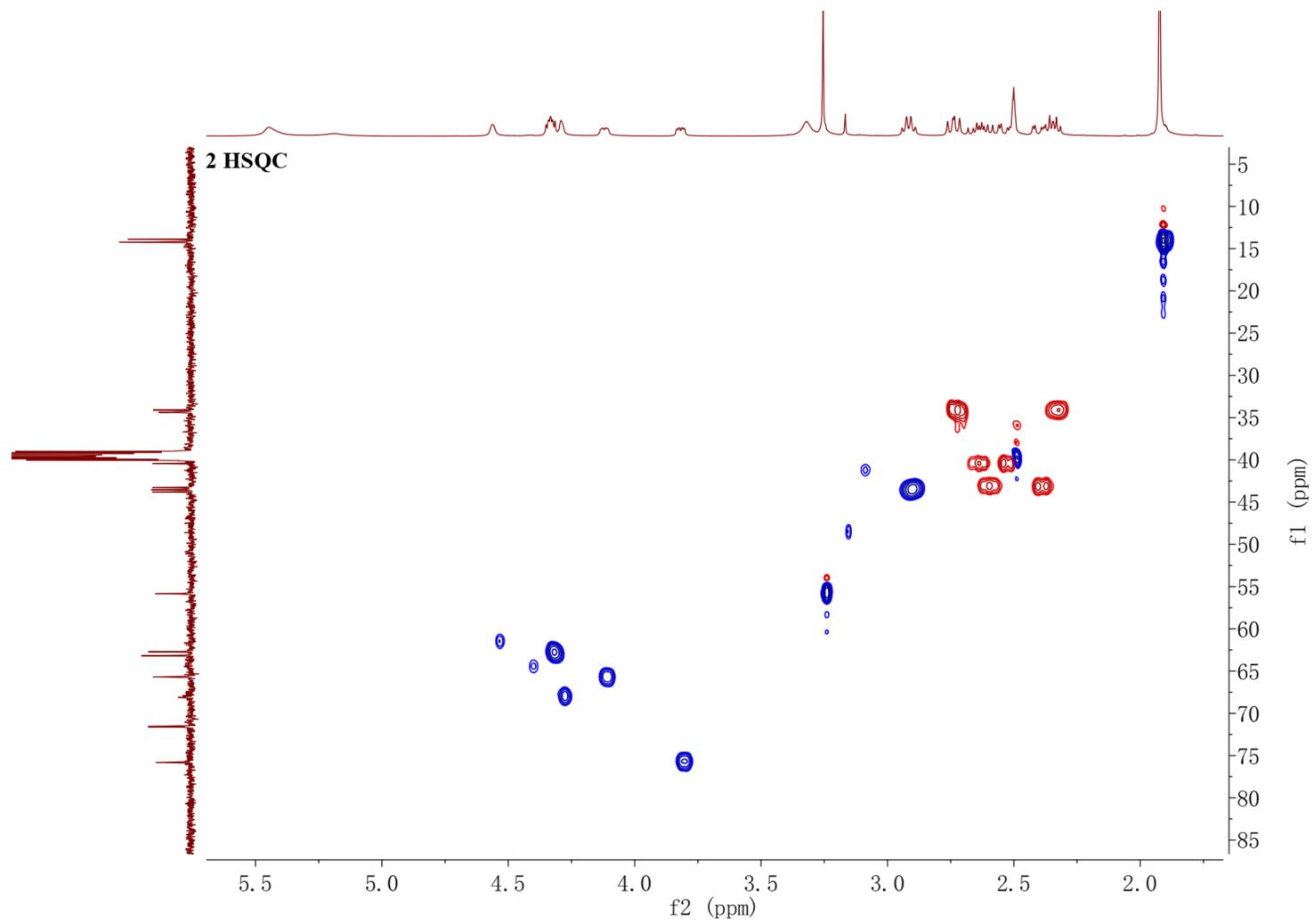


Figure S12. HMBC spectrum of compound 2.

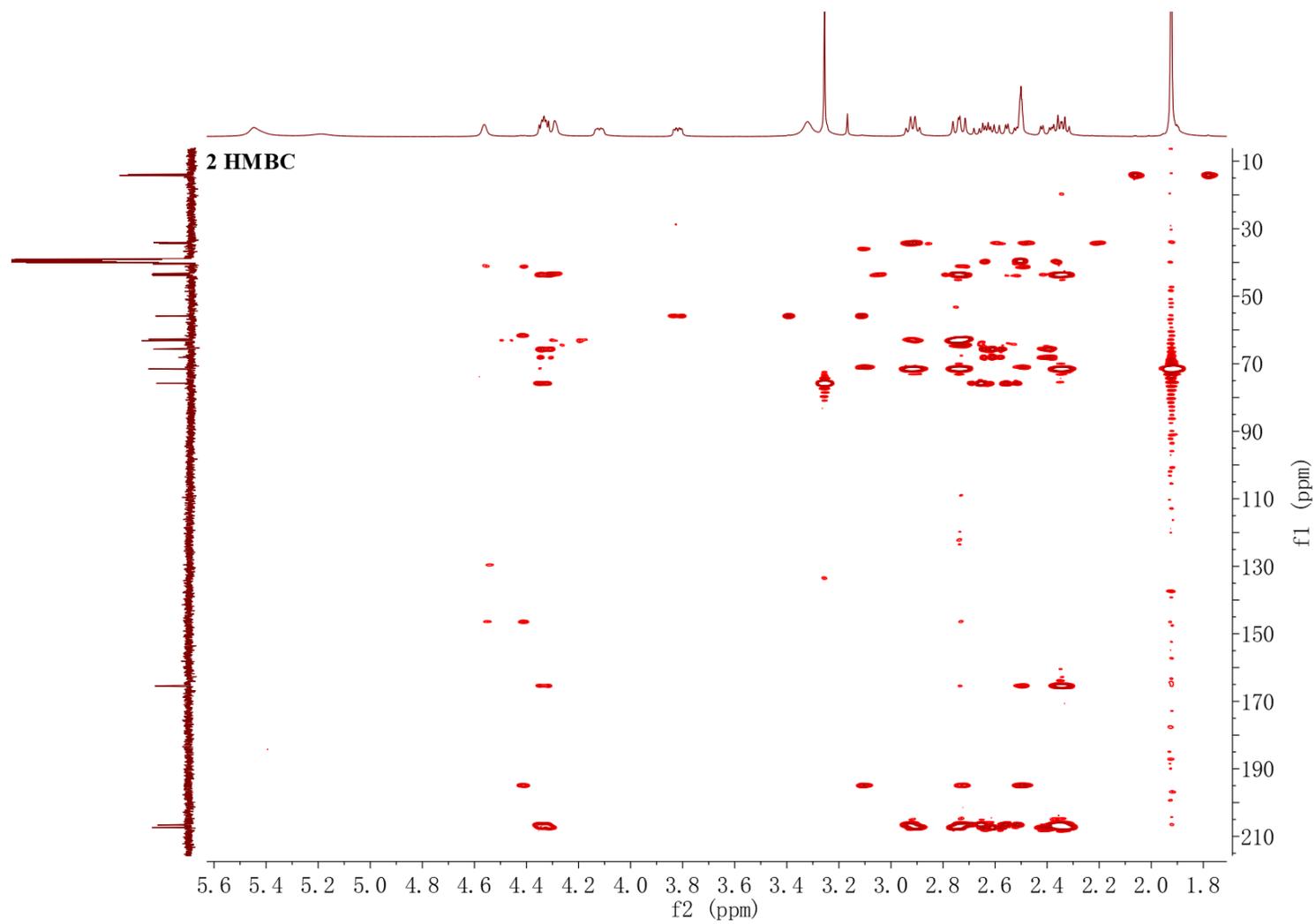


Figure S13. NOESY spectrum of compound 2.

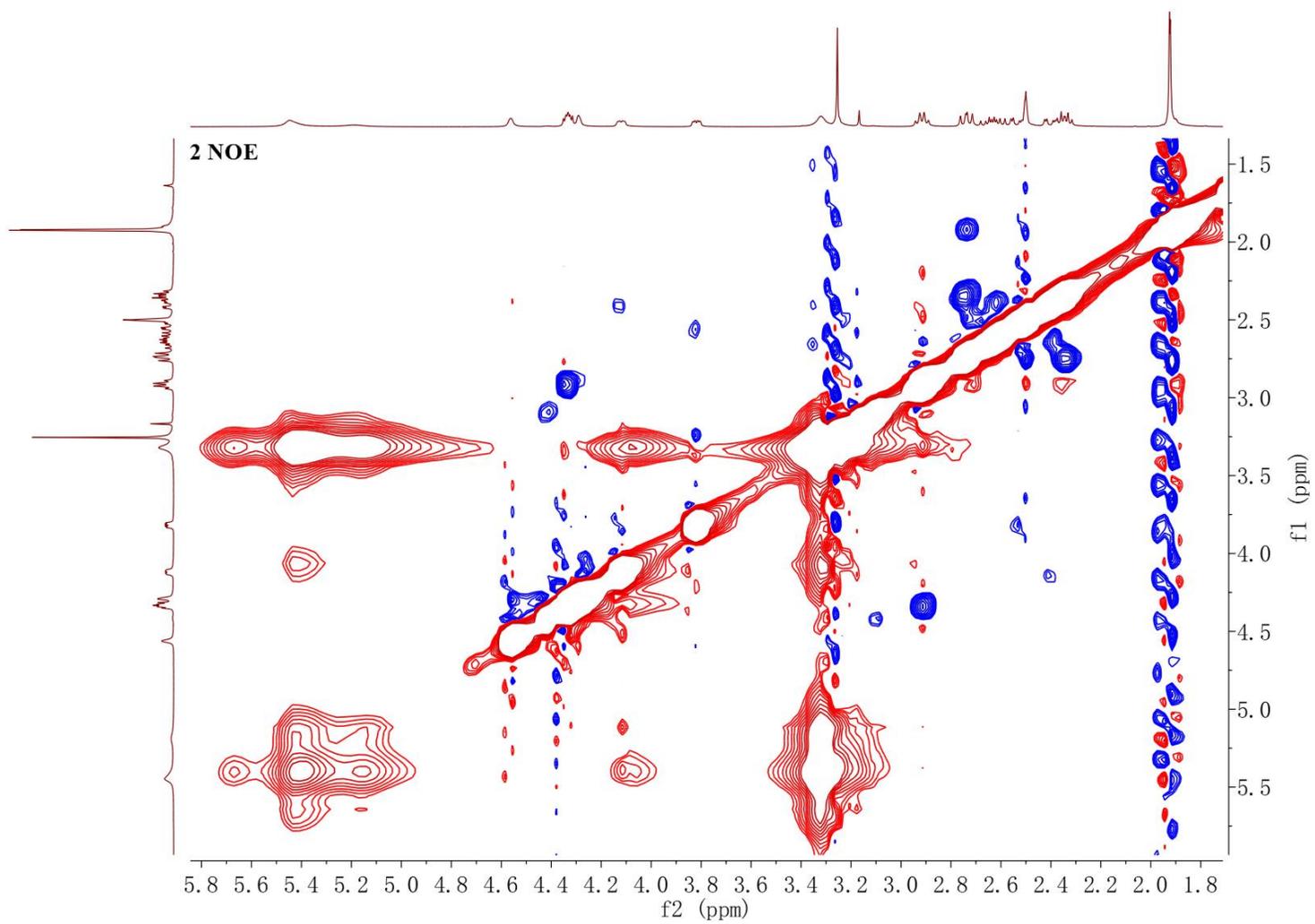


Figure S14. HR-ESI-MS of compound 2.

20190417-SD388-58_190404155858

4/17/2019 2:22:38 PM

SD388-58

20190417-SD388-58_190404155858 #54 RT: 0.43 AV: 1 NL: 9.94E6
T: FTMS + p ESI Full ms [100.00-1000.00]

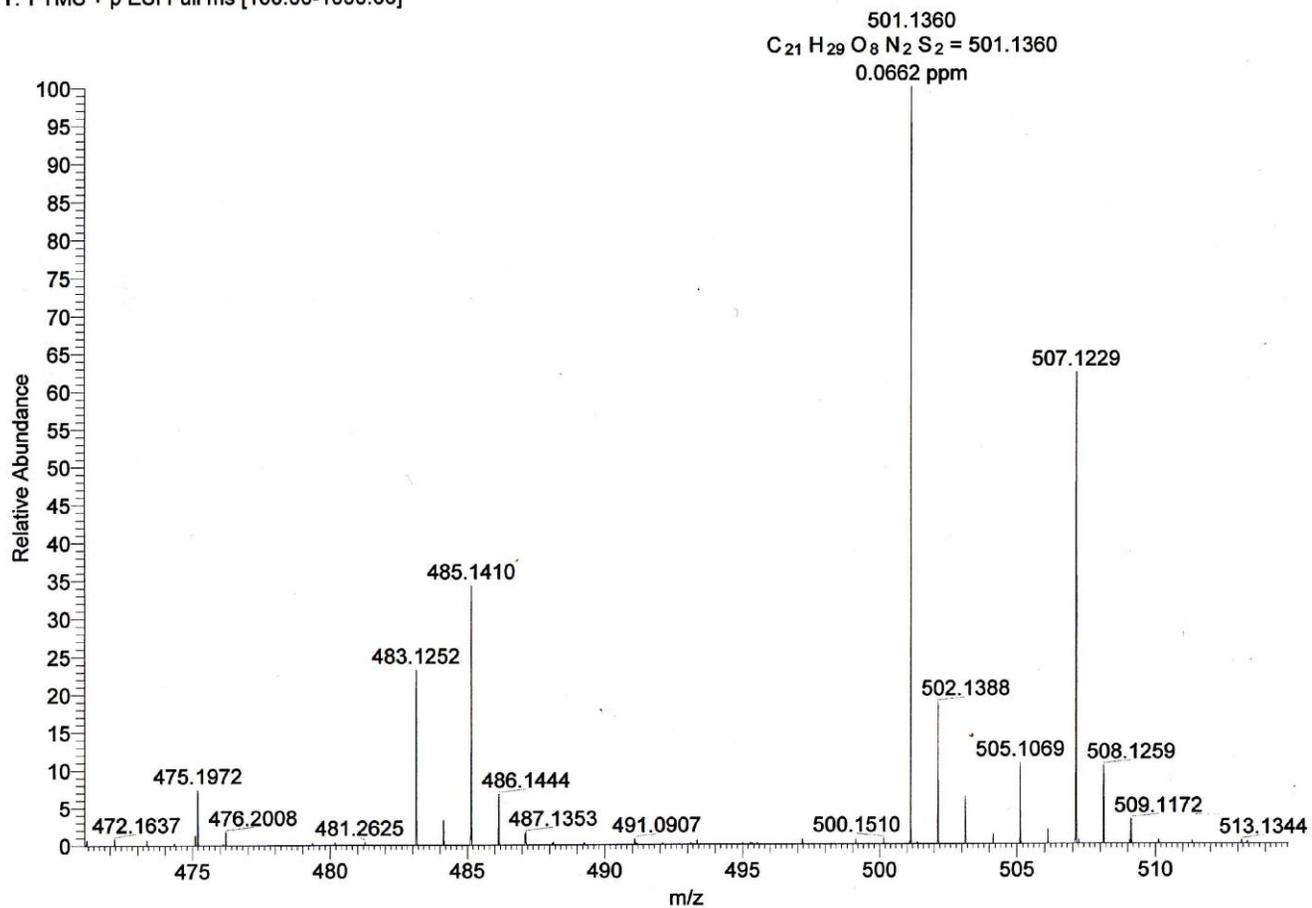


Figure S15. Crystal packing of compound 2 at 293(2) K;

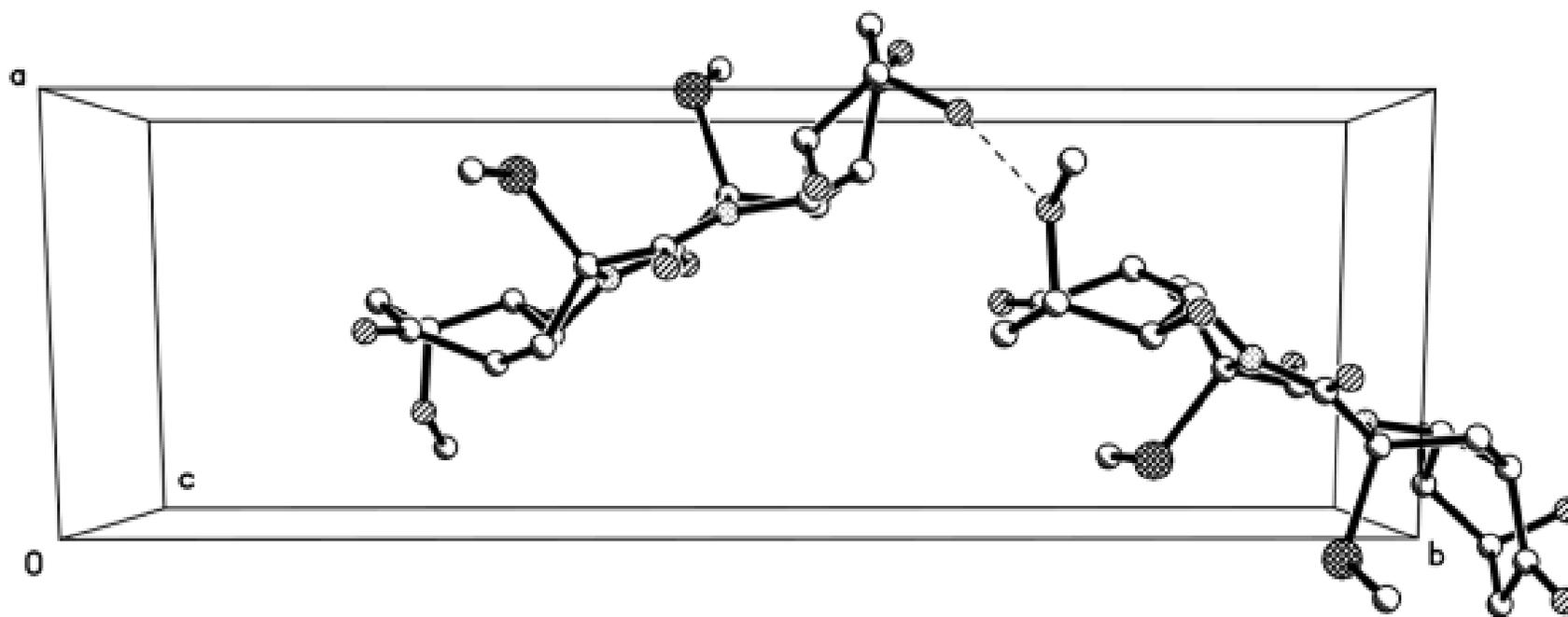


Figure S16. ^1H NMR (500 MHz, $\text{DMSO-}d_6$) spectrum of compound **3**.

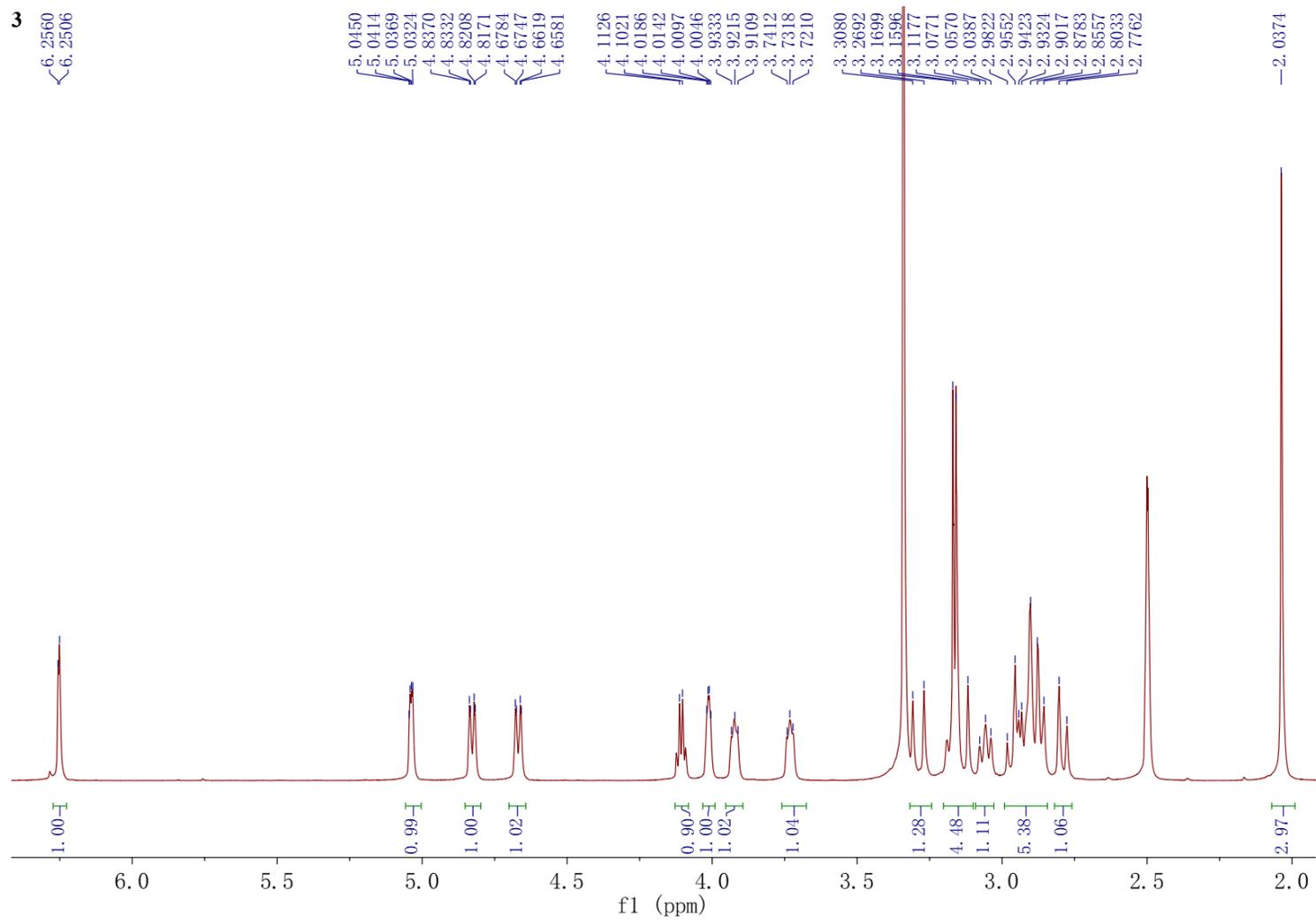


Figure S17. ^{13}C NMR and DEPT (125 MHz, $\text{DMSO}-d_6$) spectrum of compound **3**.

3

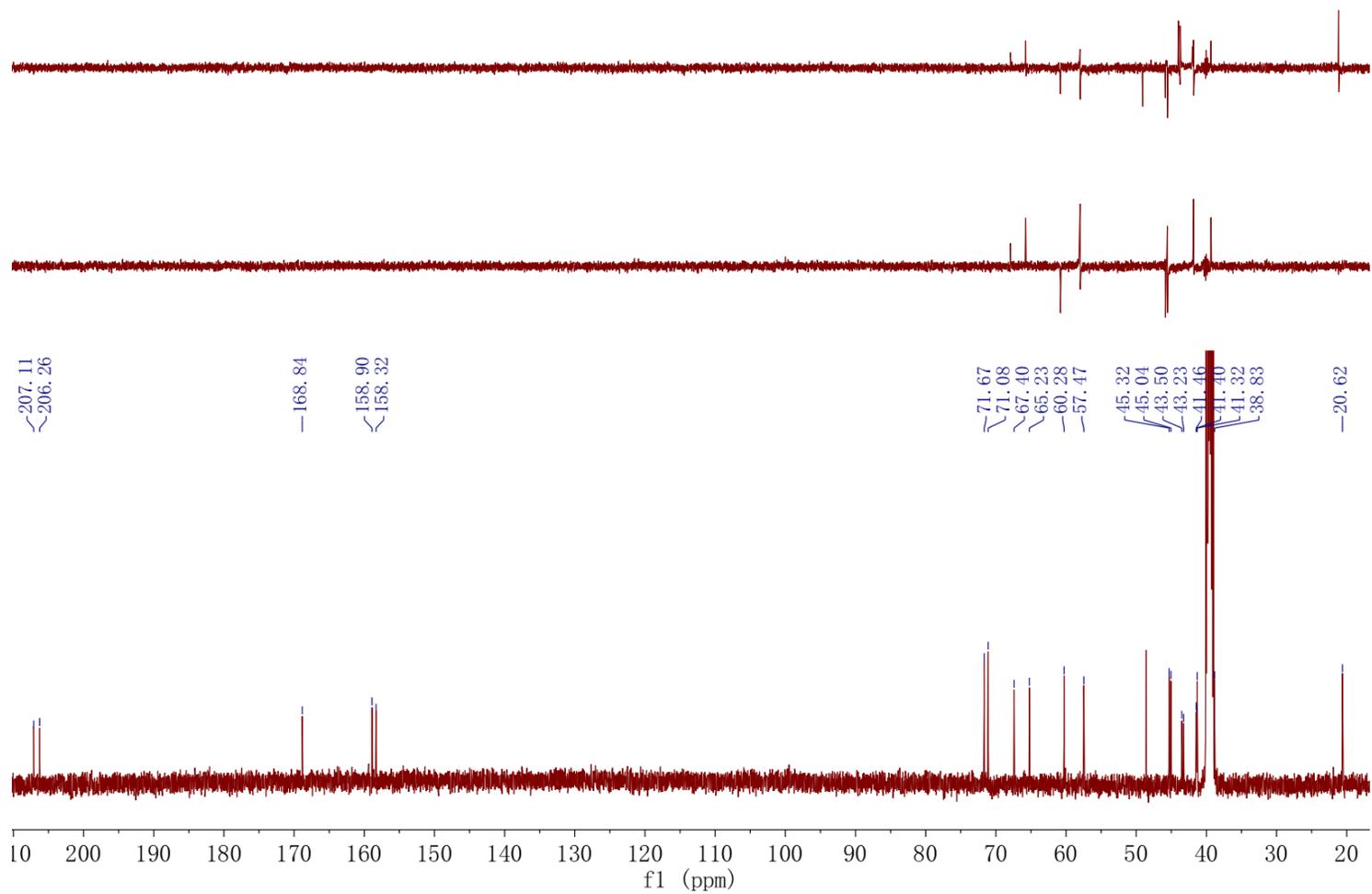


Figure S18. COSY spectrum of compound 3.

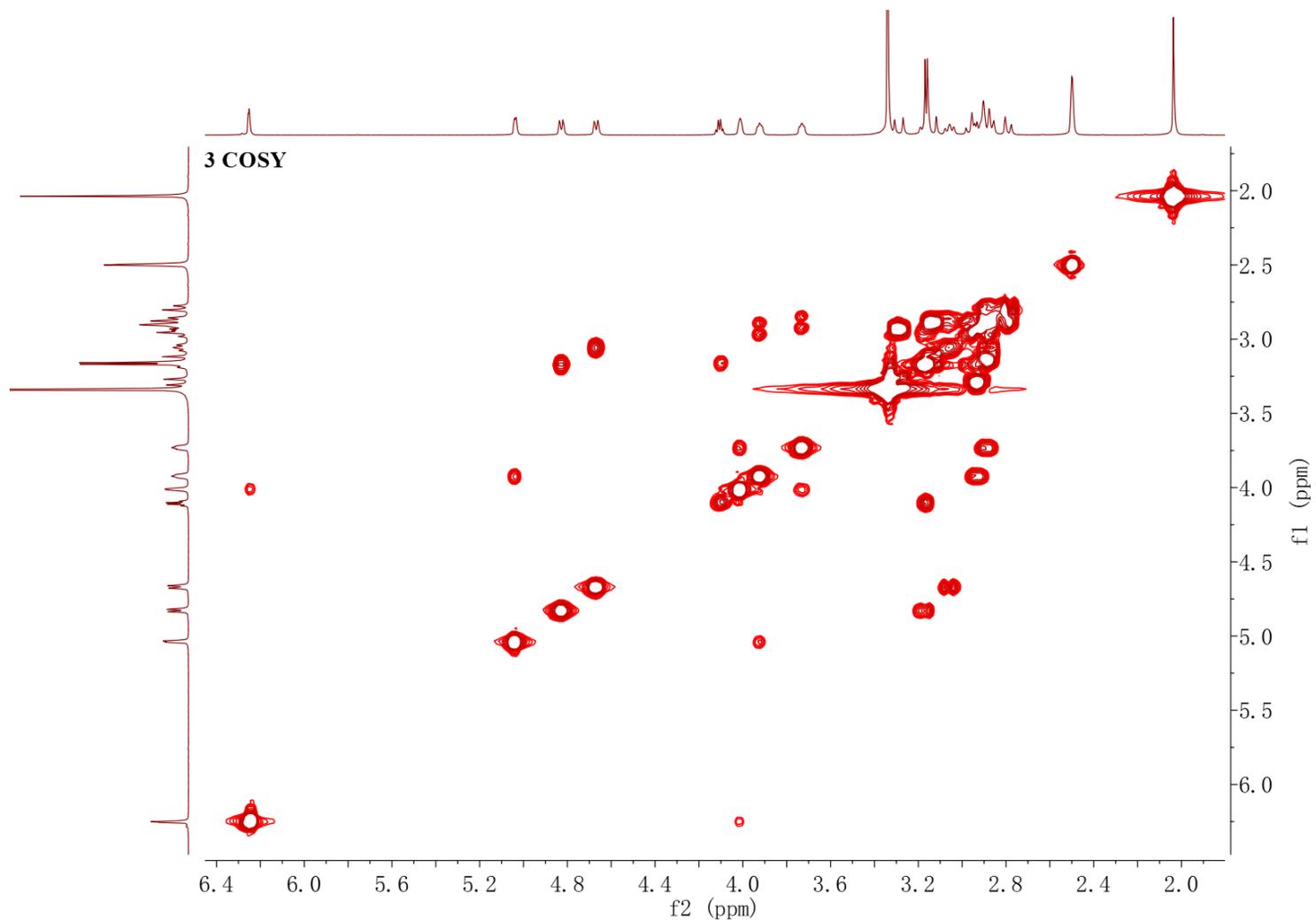


Figure S19. HMBC spectrum of compound 3.

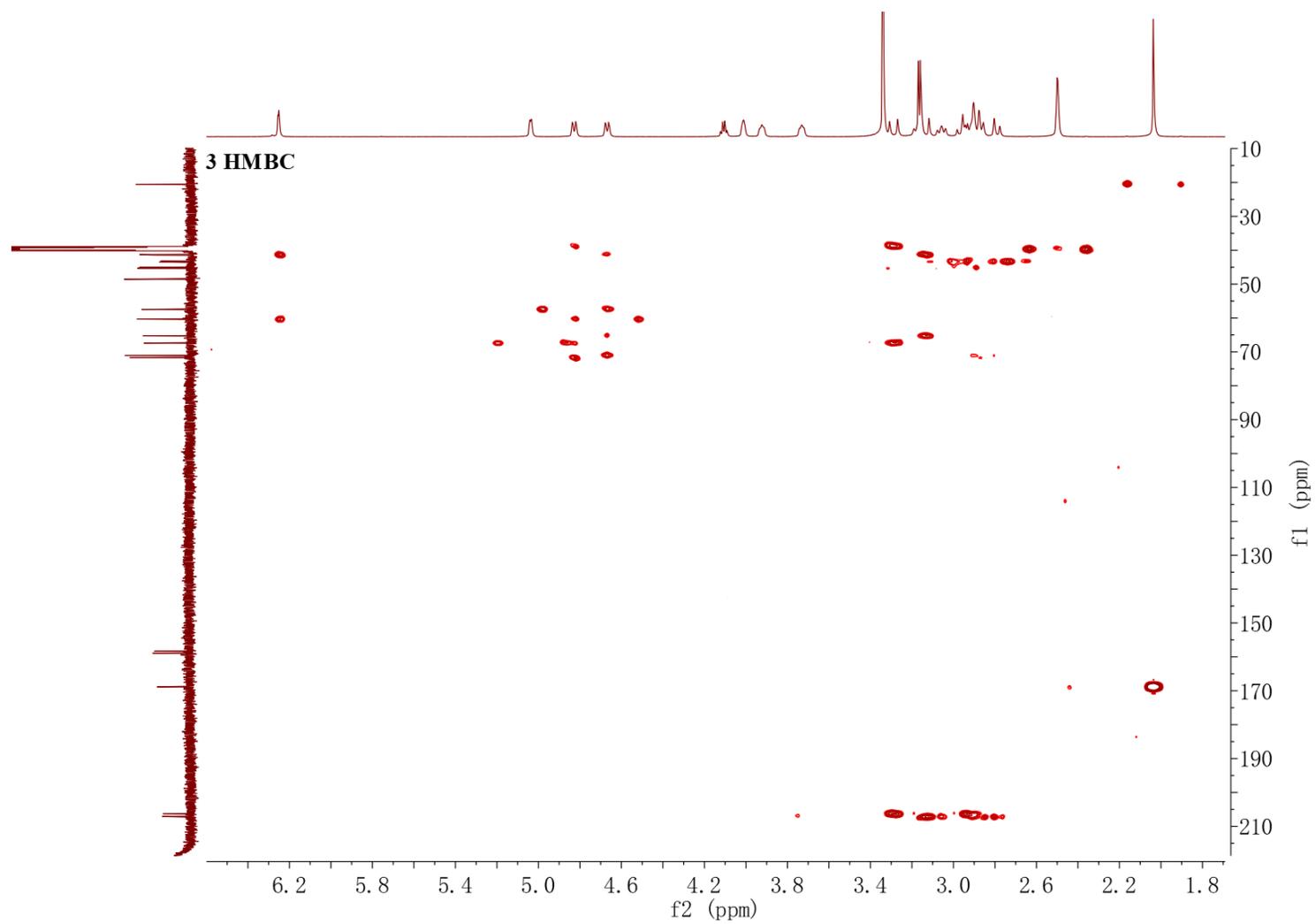


Figure S20. NOESY spectrum of compound 3.

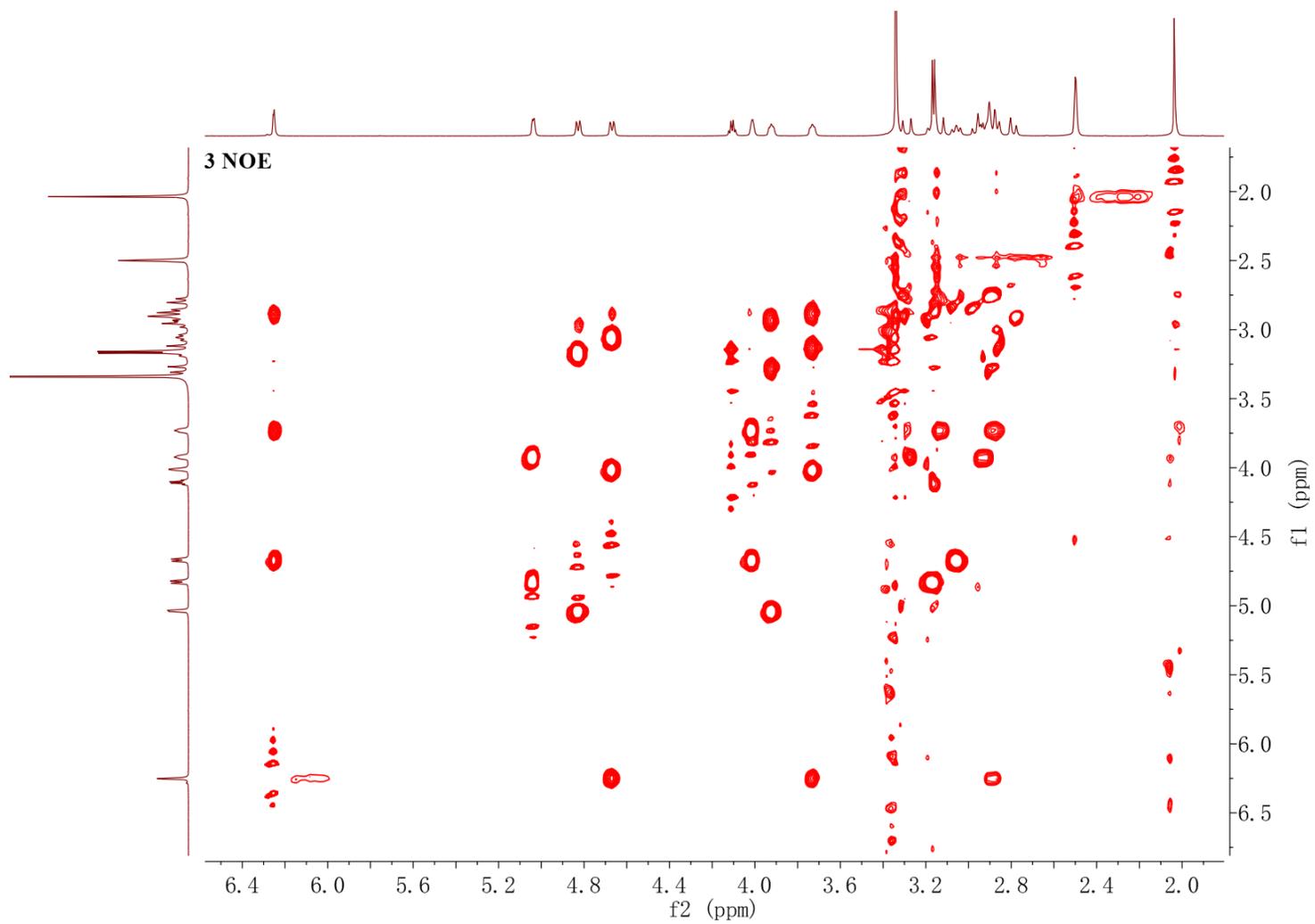


Figure S21. HR-ESI-MS of compound 3.

20180710-SD388-28_180710133327

7/10/2018 2:00:51 PM

SD388-28

20180710-SD388-28_180710133327 #52-54 RT: 0.42-0.44 AV: 3 NL: 6.65E6

T: FTMS + p ESI Full ms [100.00-1000.00]

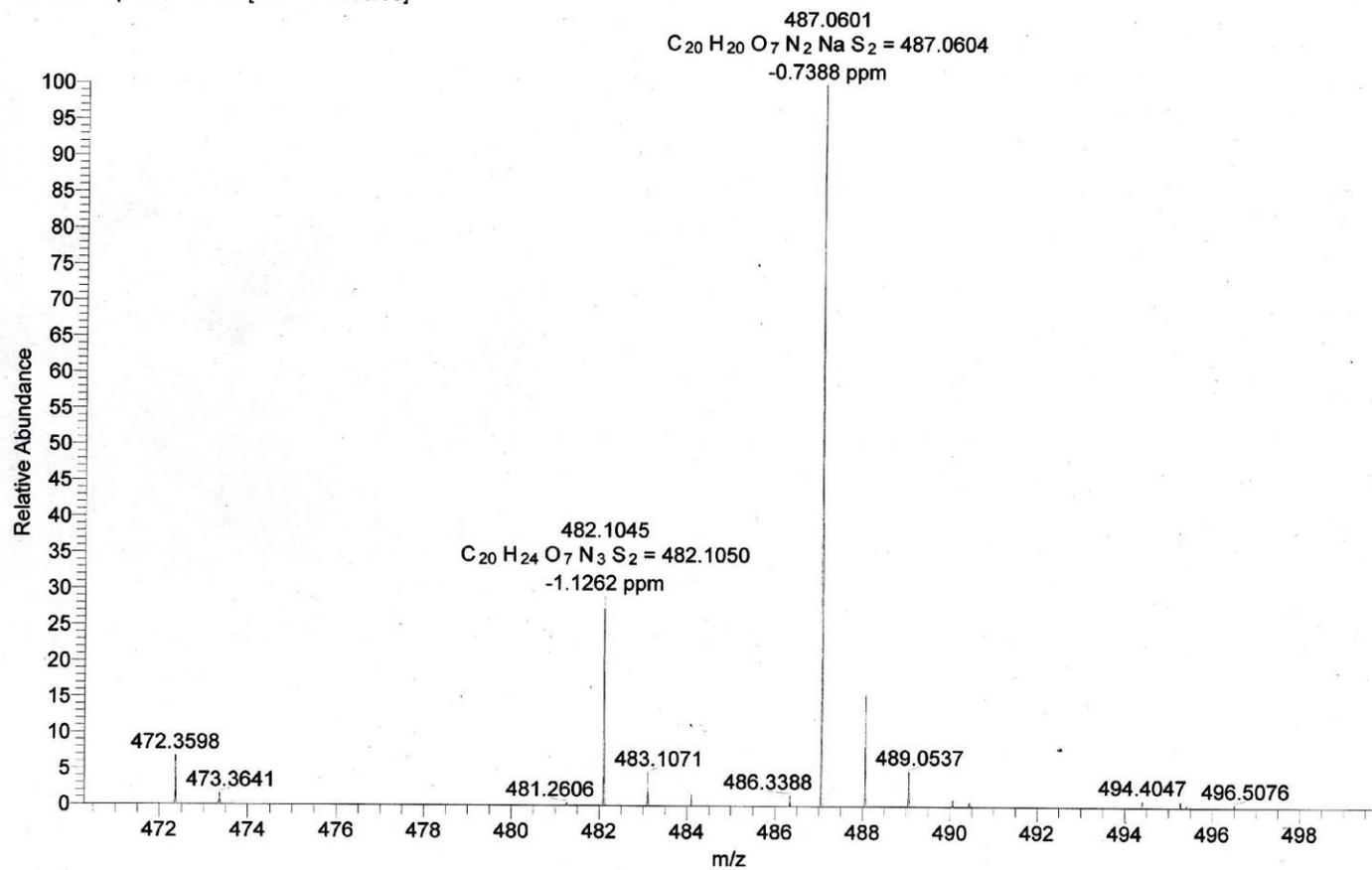


Figure S22. Crystal packing of compound 3 at 293(2) K;

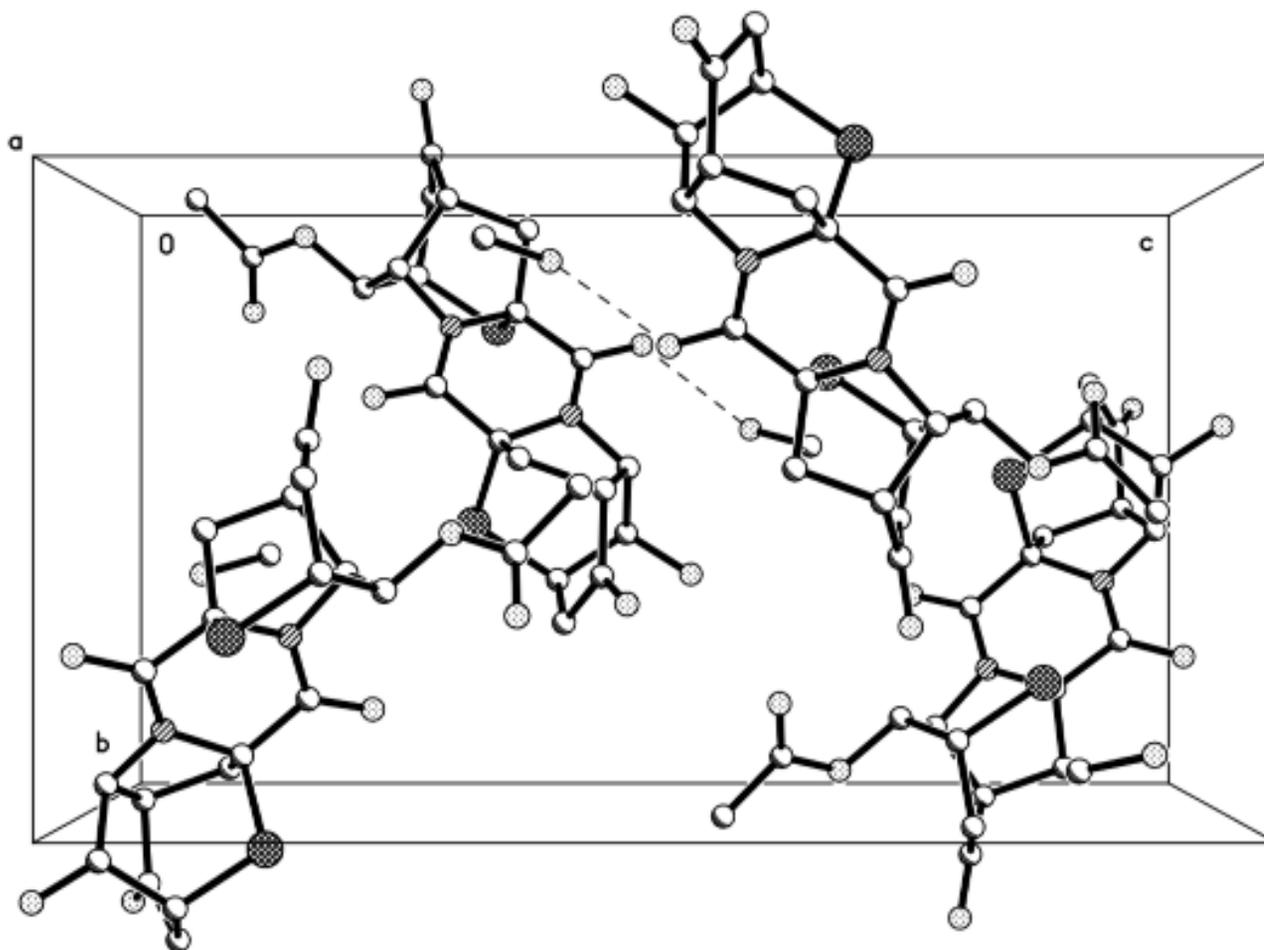


Figure S23. ¹H NMR (500 MHz, DMSO-*d*₆) spectrum of compound 4.

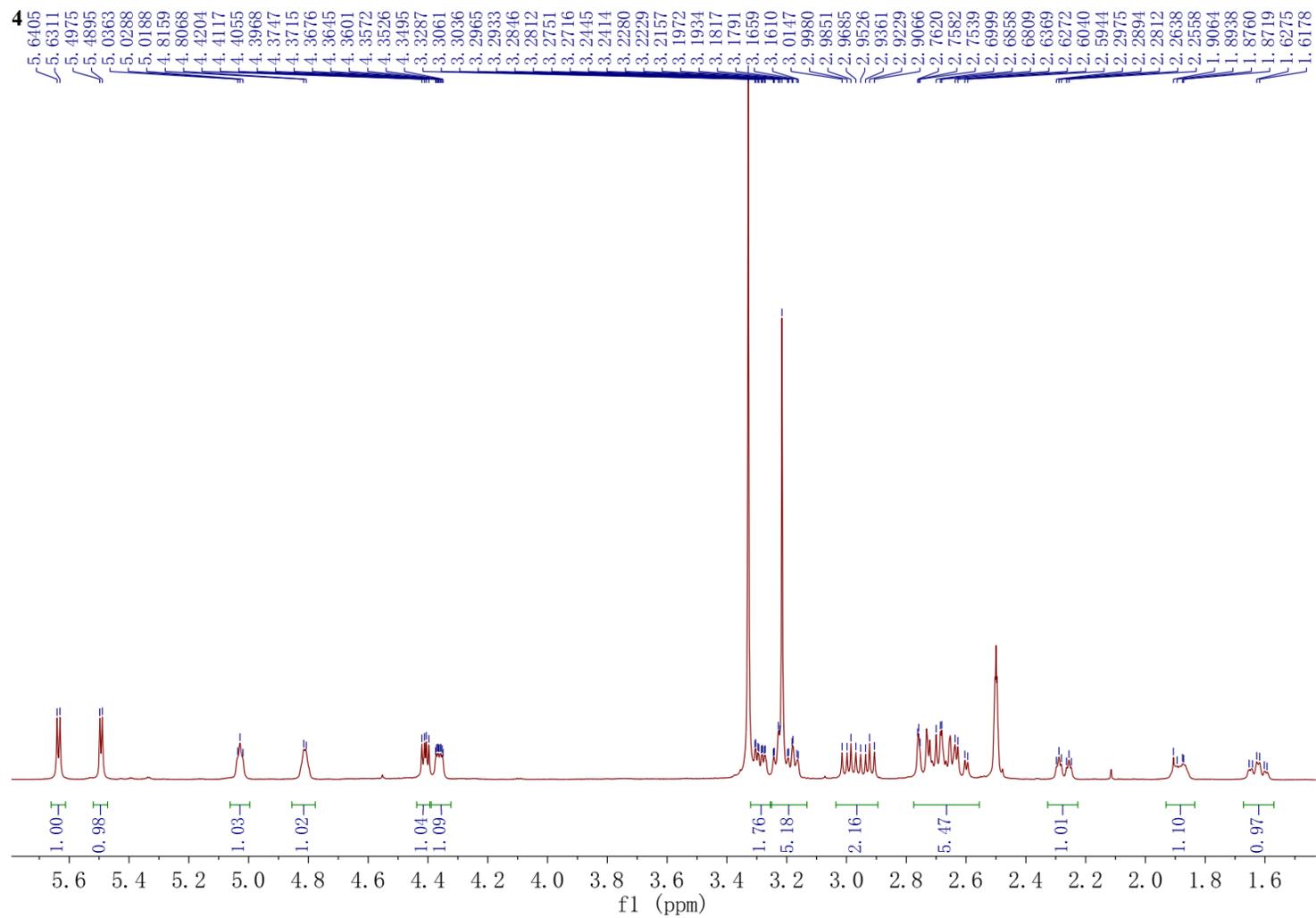


Figure S24. ^{13}C NMR and DEPT (125 MHz, $\text{DMSO}-d_6$) spectrum of compound 4.

4

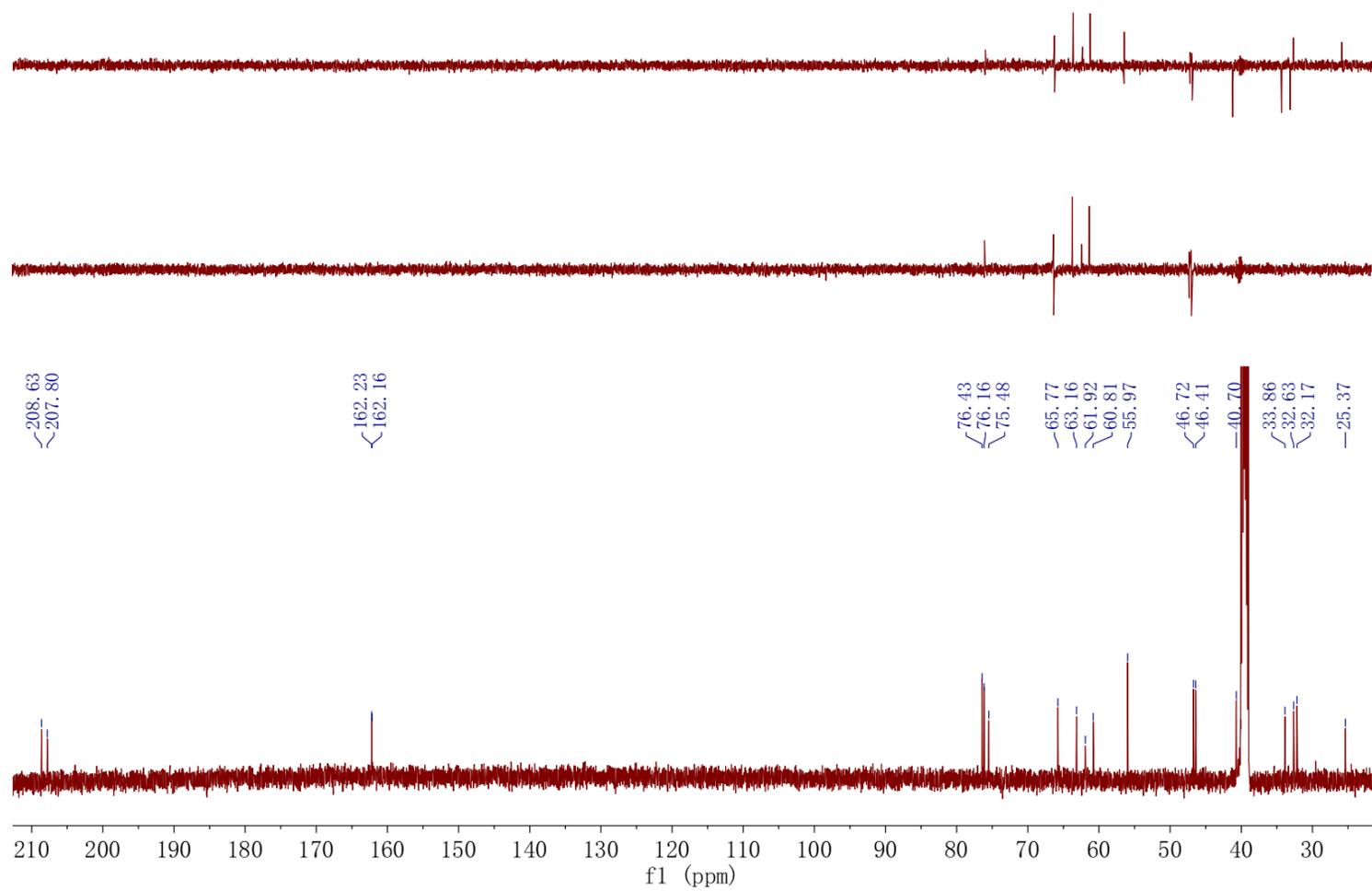


Figure S25. COSY spectrum of compound 4.

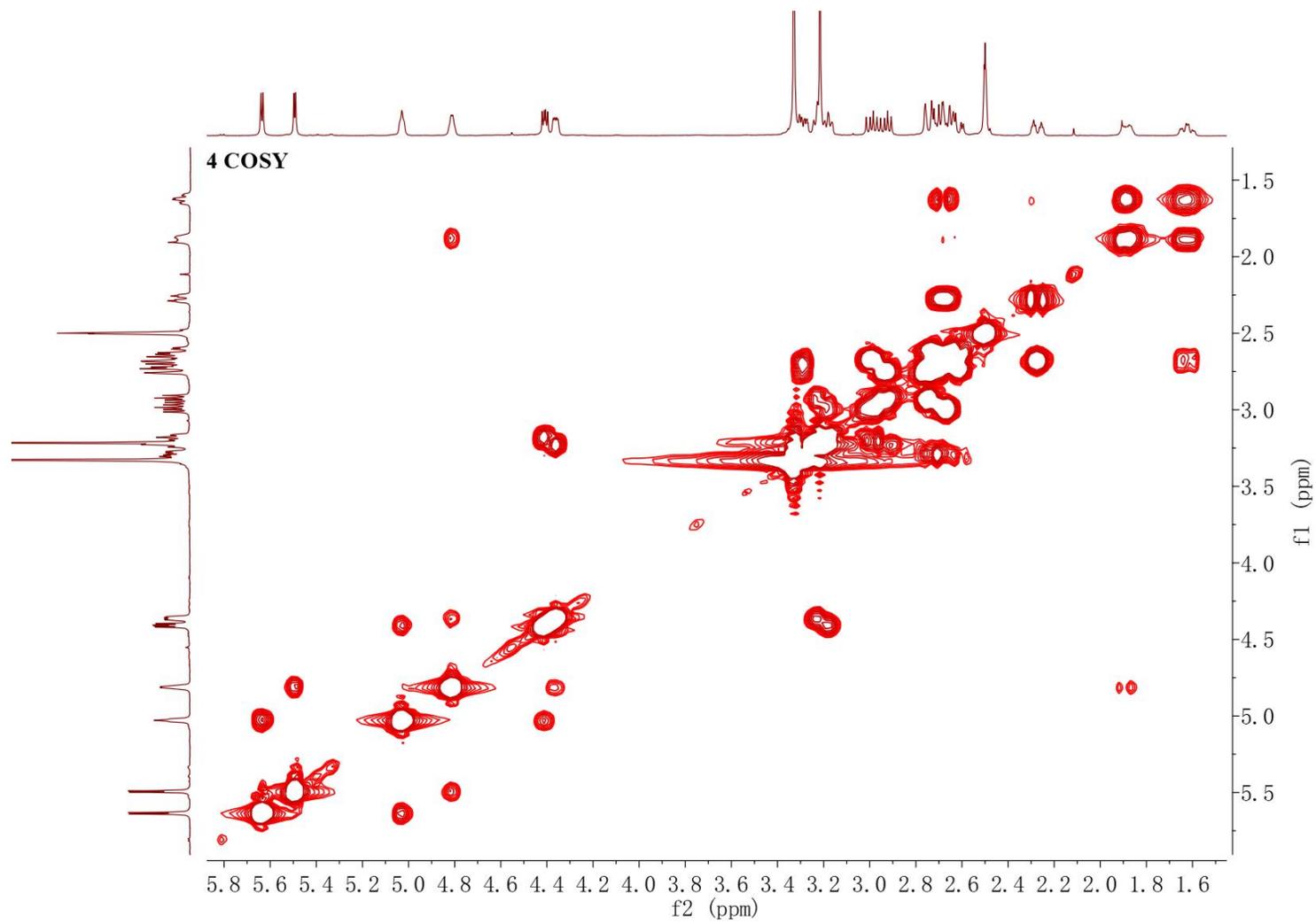


Figure S26. HSQC spectrum of compound 4.

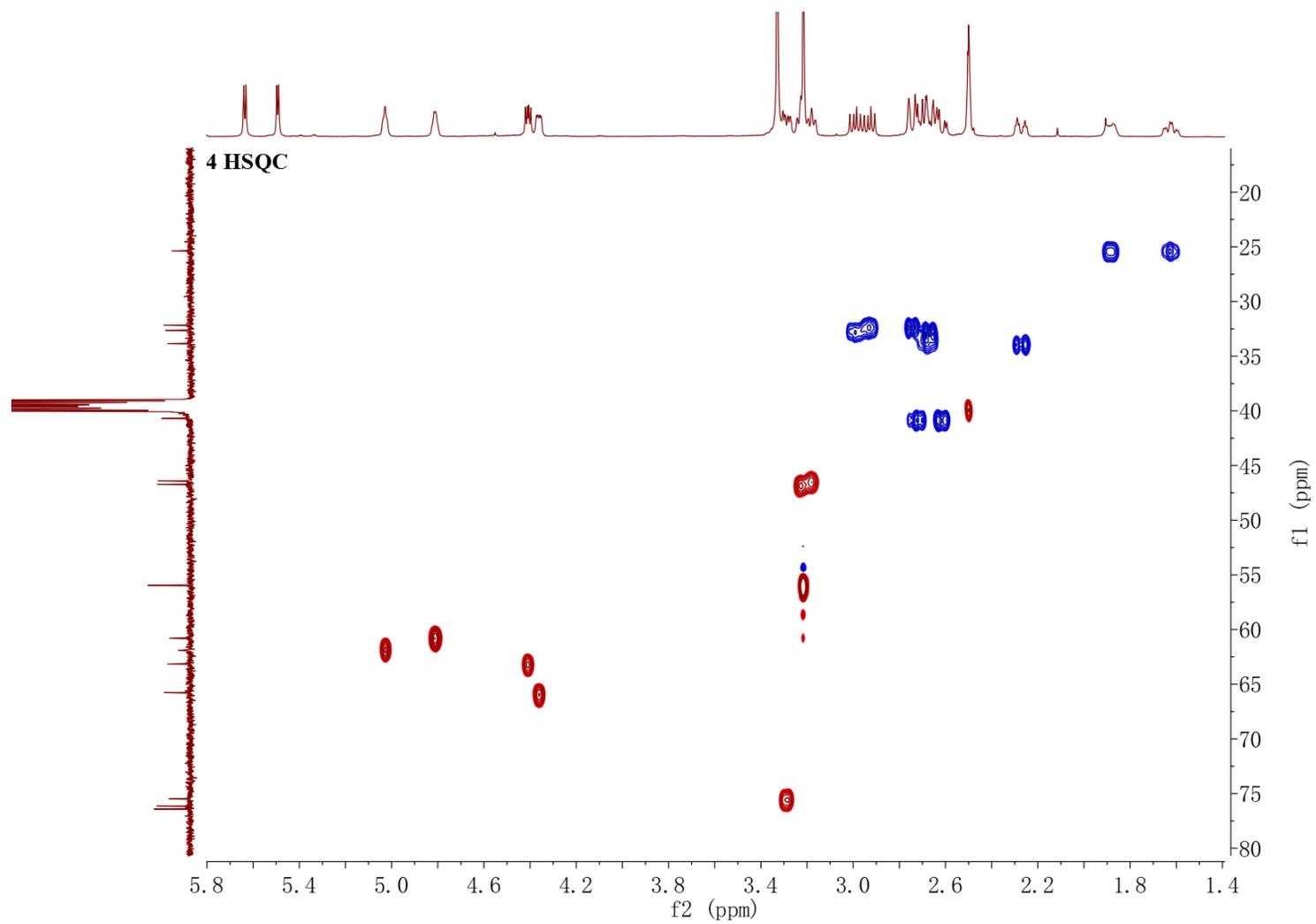


Figure S27. HMBC spectrum of compound 4.

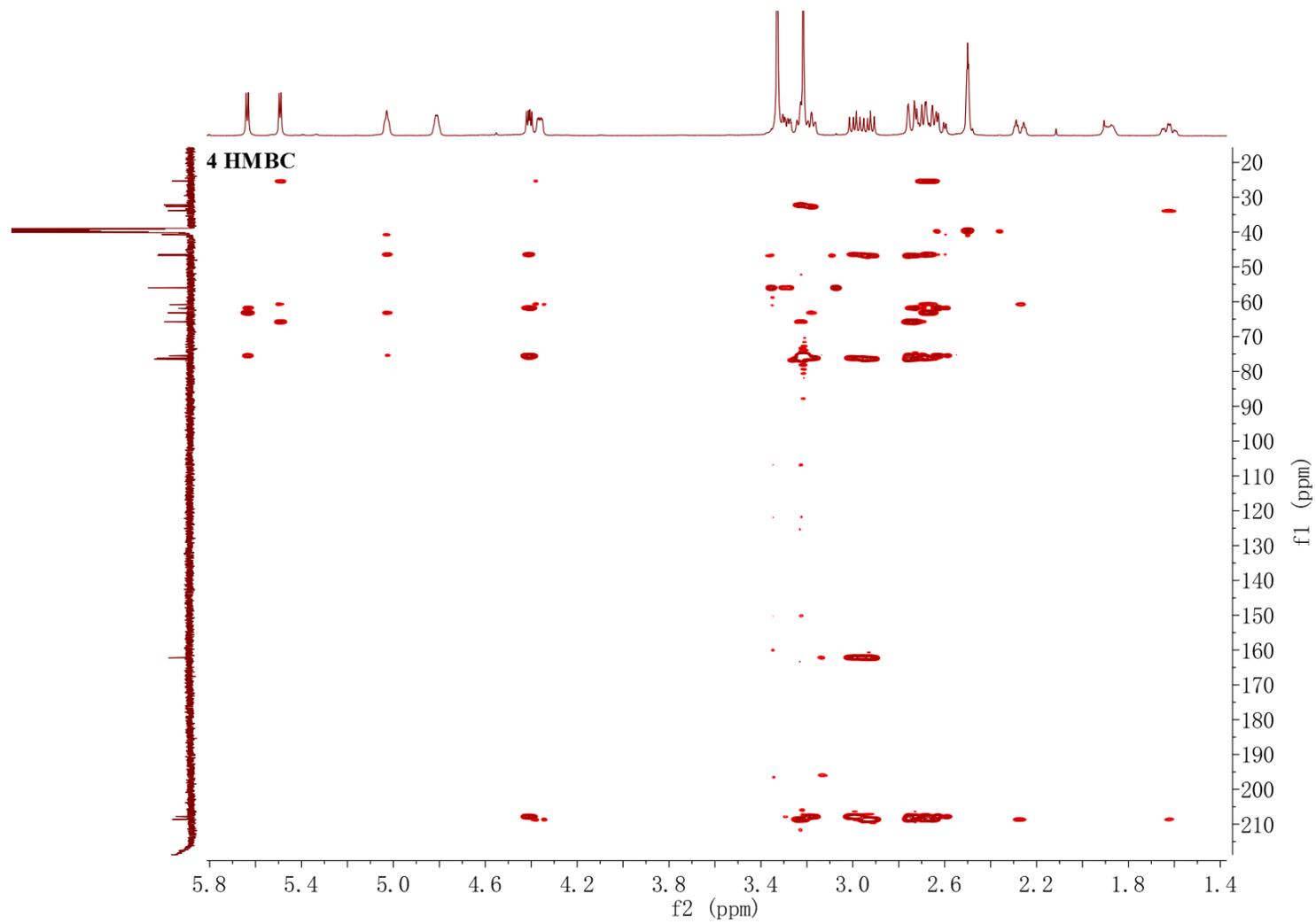


Figure S28. NOESY spectrum of compound 4.

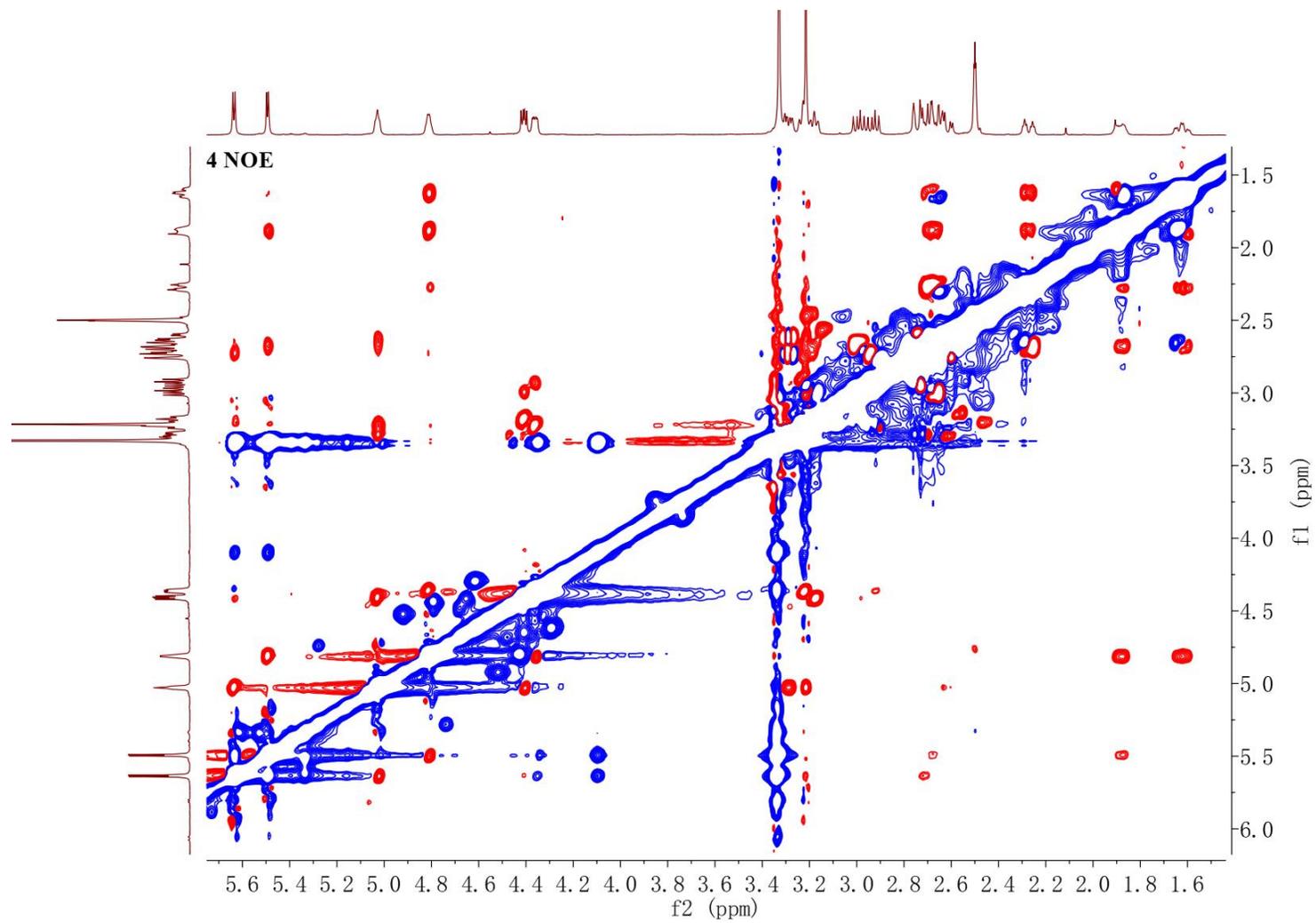


Figure S29. HR-ESI-MS of compound 4.

20180905-SD388-33_180904143152

9/5/2018 9:42:10 AM

SD388-33

20180905-SD388-33_180904143152 #80 RT: 0.65 AV: 1 NL: 5.90E5

T: FTMS + p ESI Full ms [100.00-1000.00]

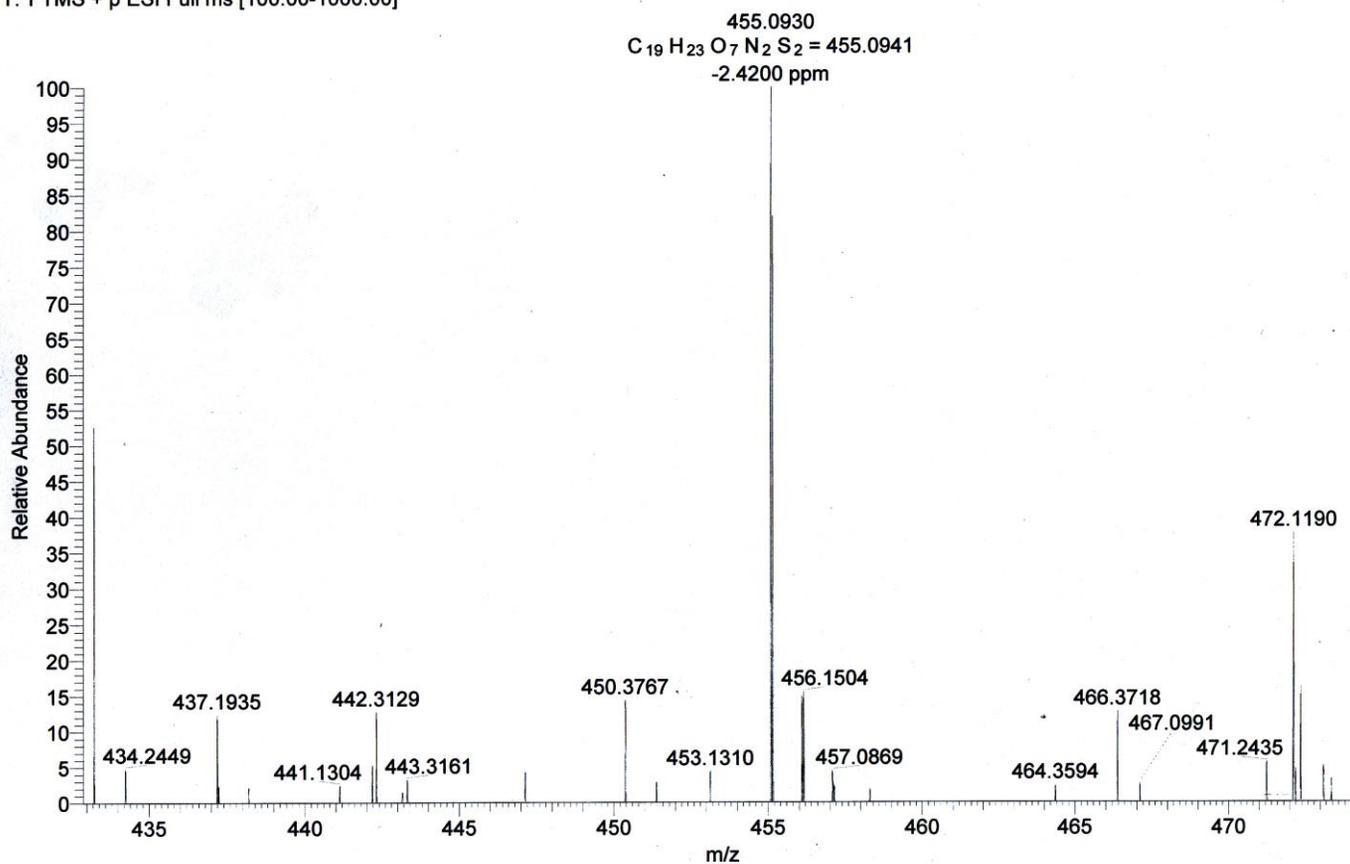


Figure S30. ECD spectrum of compound 4.

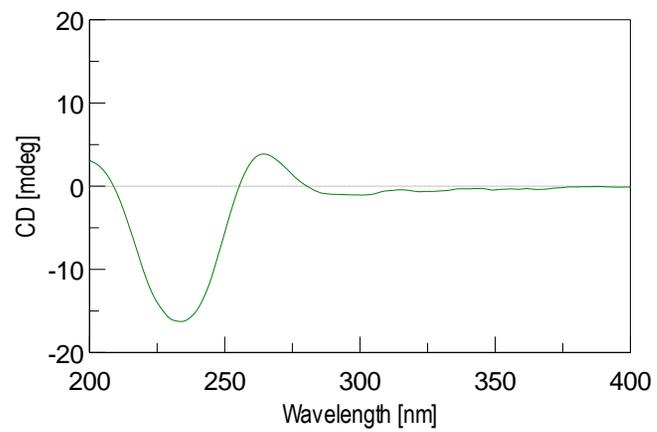


Figure S31. ^1H NMR (500 MHz, $\text{DMSO-}d_6$) spectrum of compound 5.

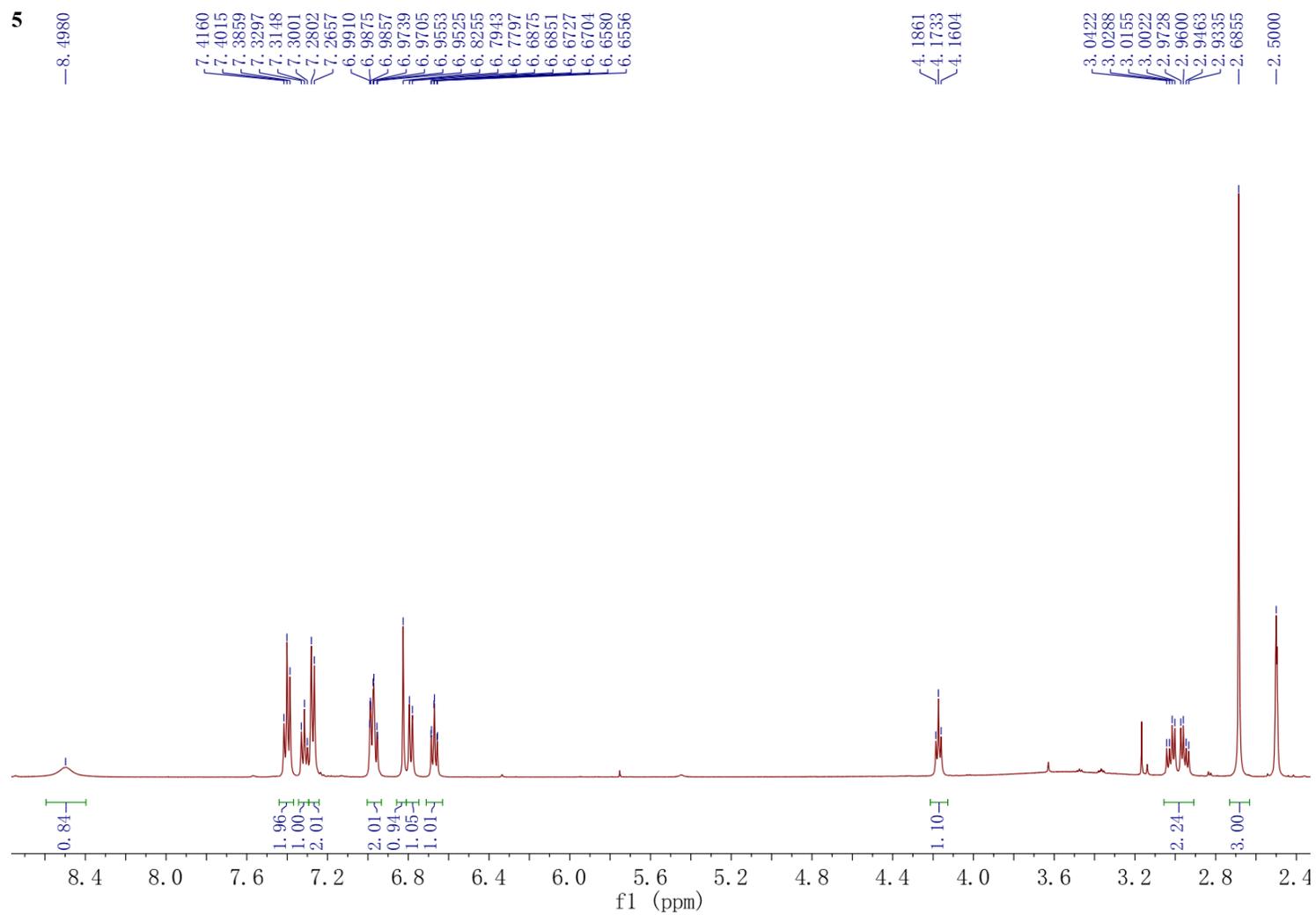


Figure S32. ^{13}C NMR and DEPT (125 MHz, $\text{DMSO}-d_6$) spectrum of compound 5.

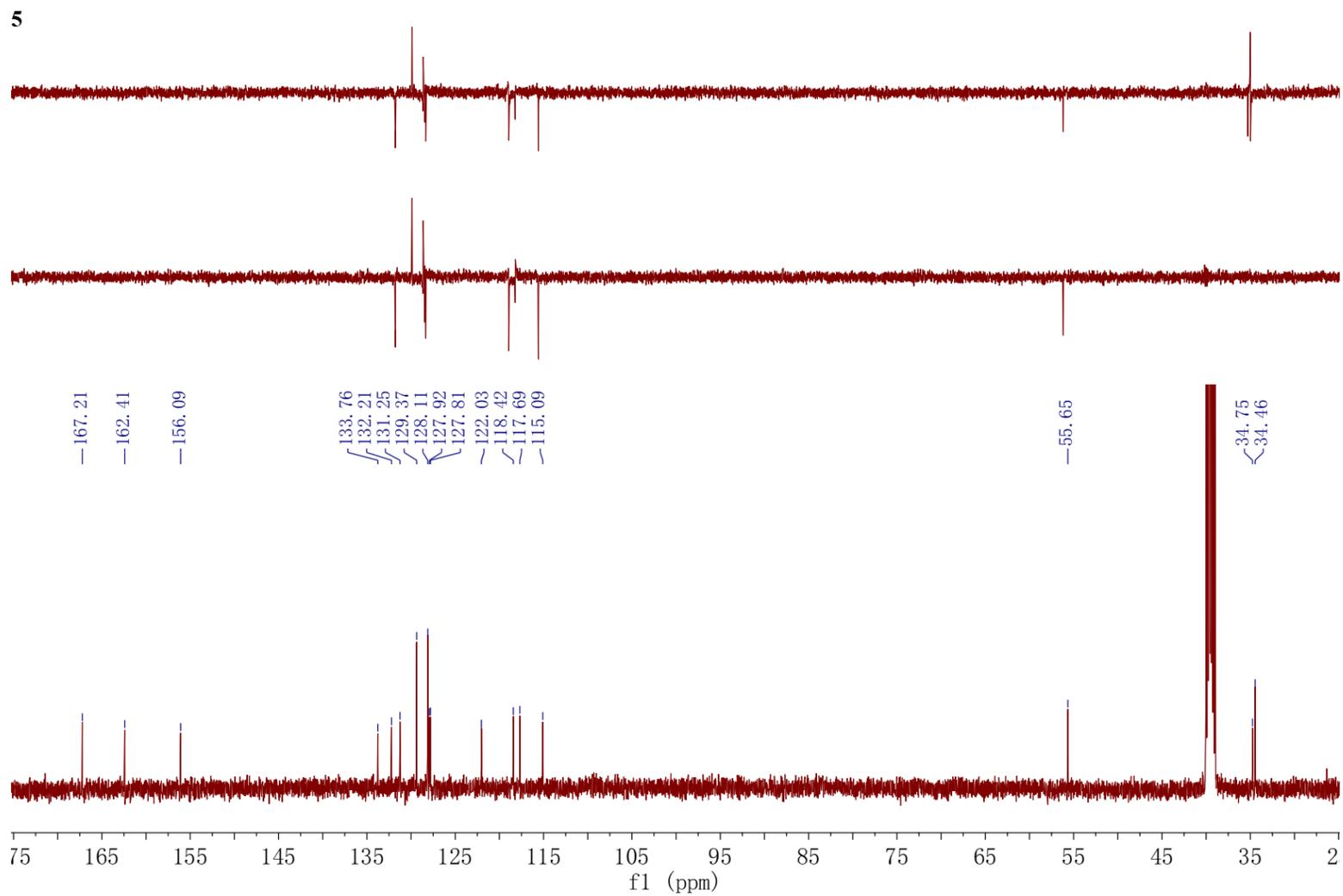


Figure S33. COSY spectrum of compound 5.

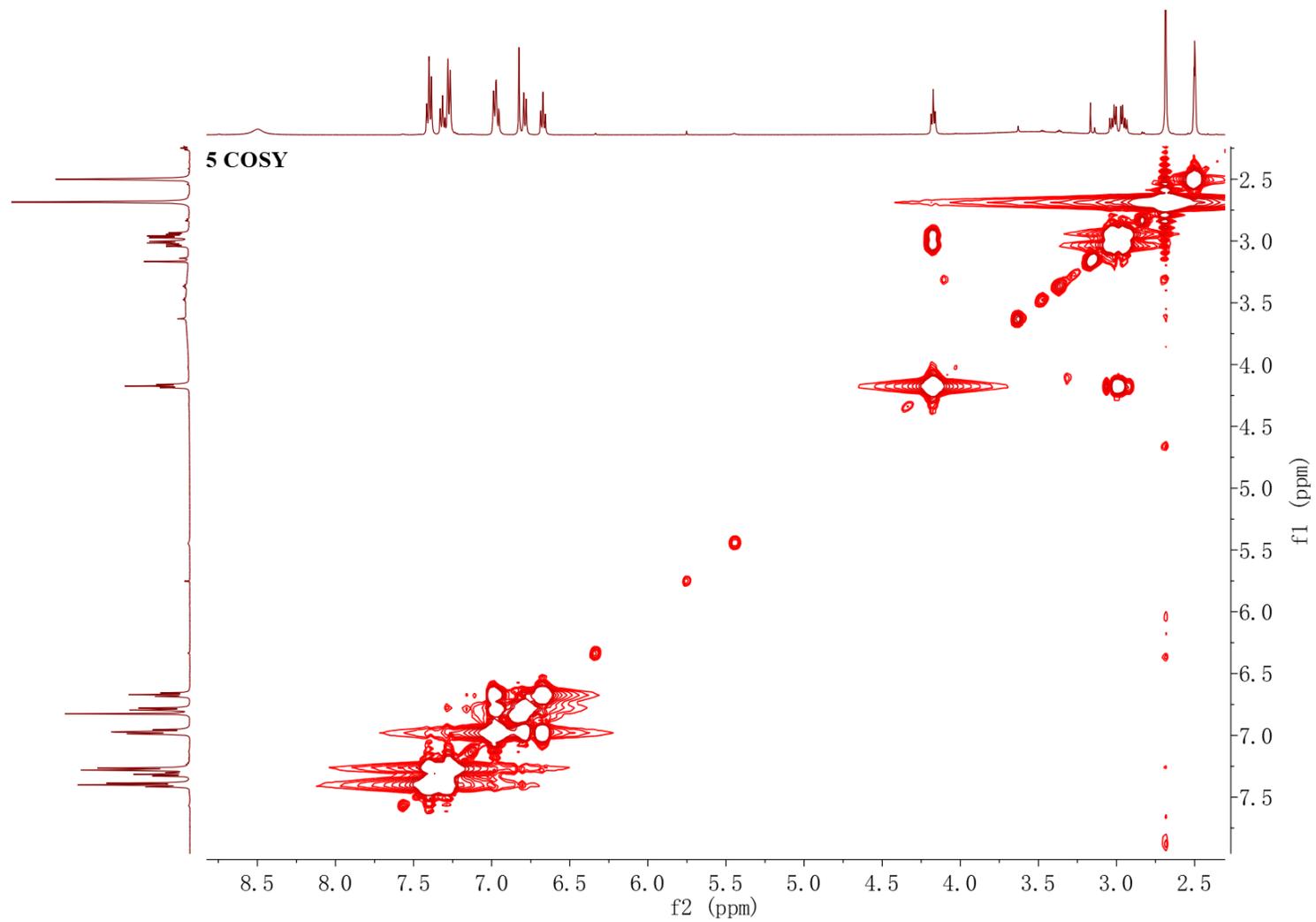


Figure S34. HMBC spectrum of compound 5.

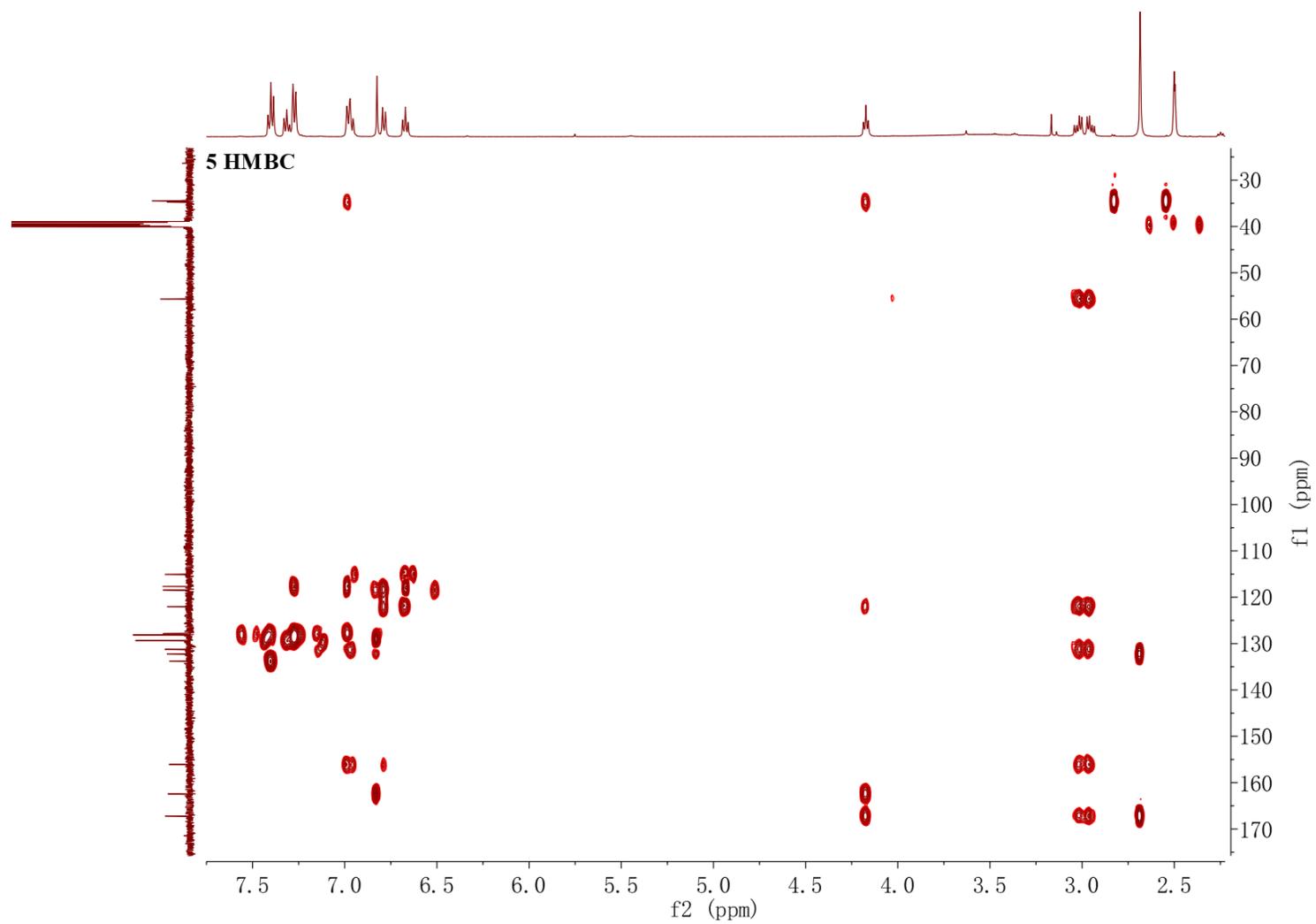


Figure S35. HR-ESI-MS of compound 5.

20181016-SD388-46_181011105915

10/16/2018 11:04:33 AM

SD388-46

20181016-SD388-46_181011105915 #39 RT: 0.31 AV: 1 NL: 4.54E6
T: FTMS + p ESI Full ms [170.00-2000.00]

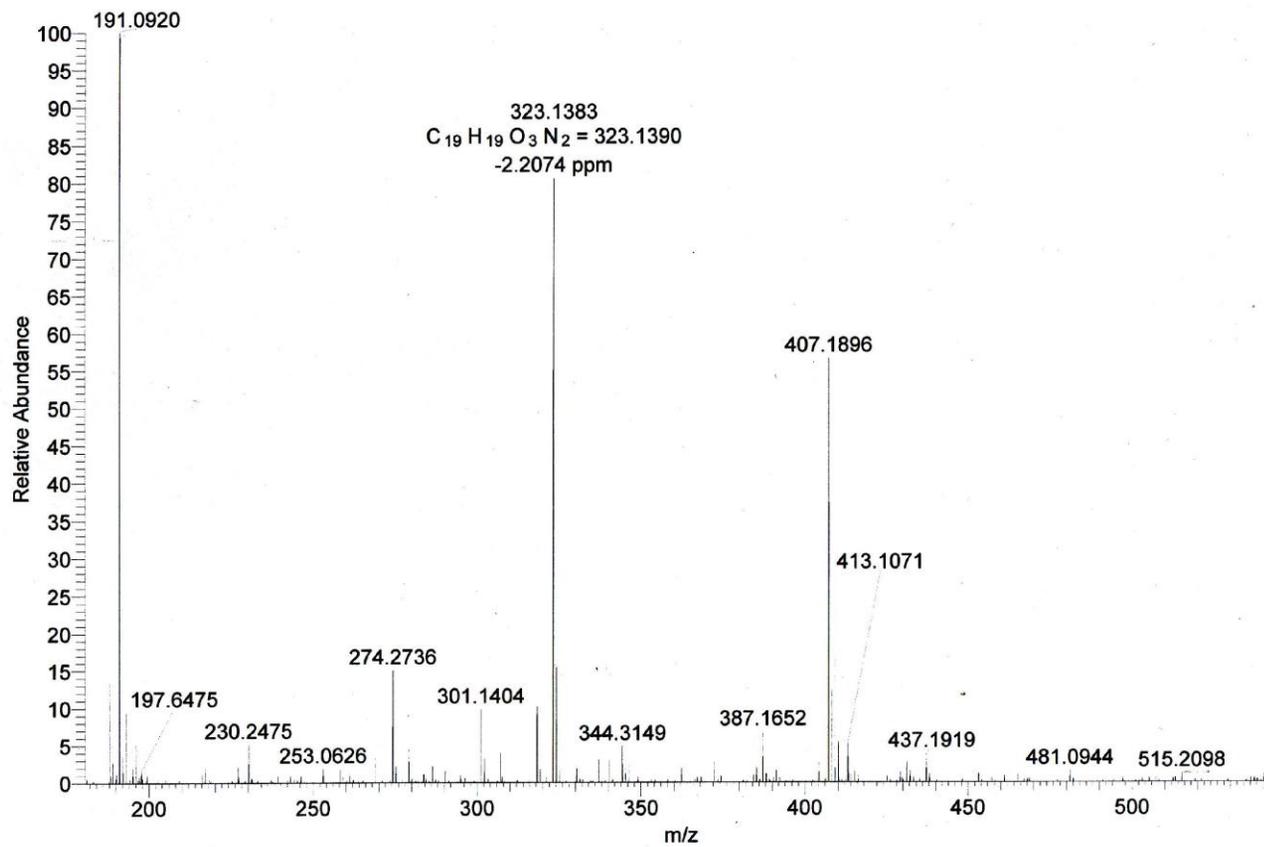


Figure S36. ECD spectrum of compound (\pm)-5.

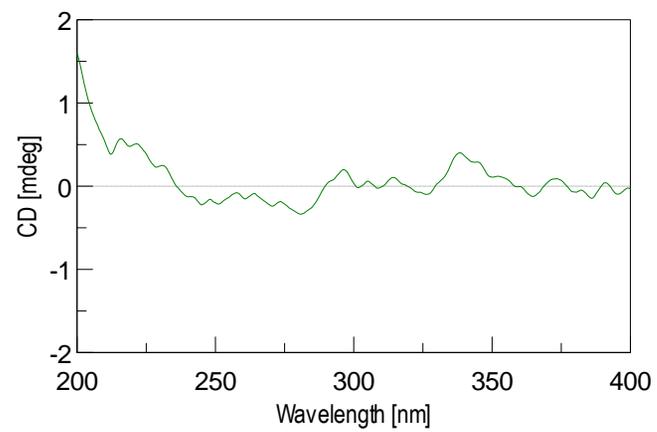


Figure S37. Chiral HPLC profile of compound (\pm)-5.

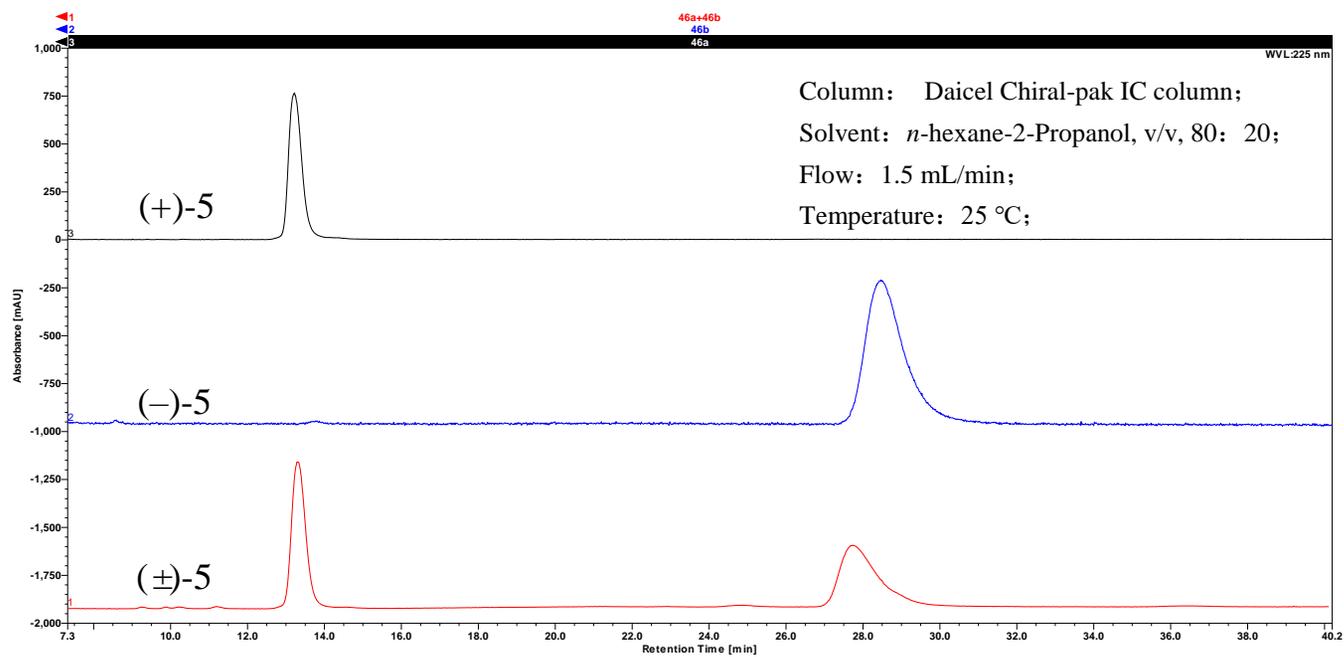


Figure S38. ECD spectrum of compound (+)-5.

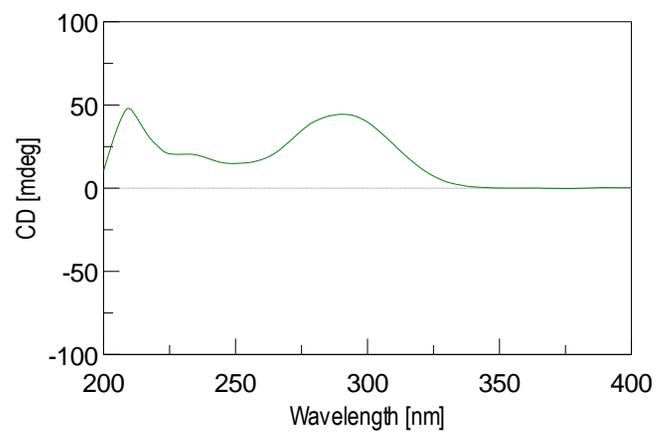


Figure S39. ECD spectrum of compound (-)-5.

