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Figure S1. HRESIMS spectrum of asperopiperazine A (**1**).

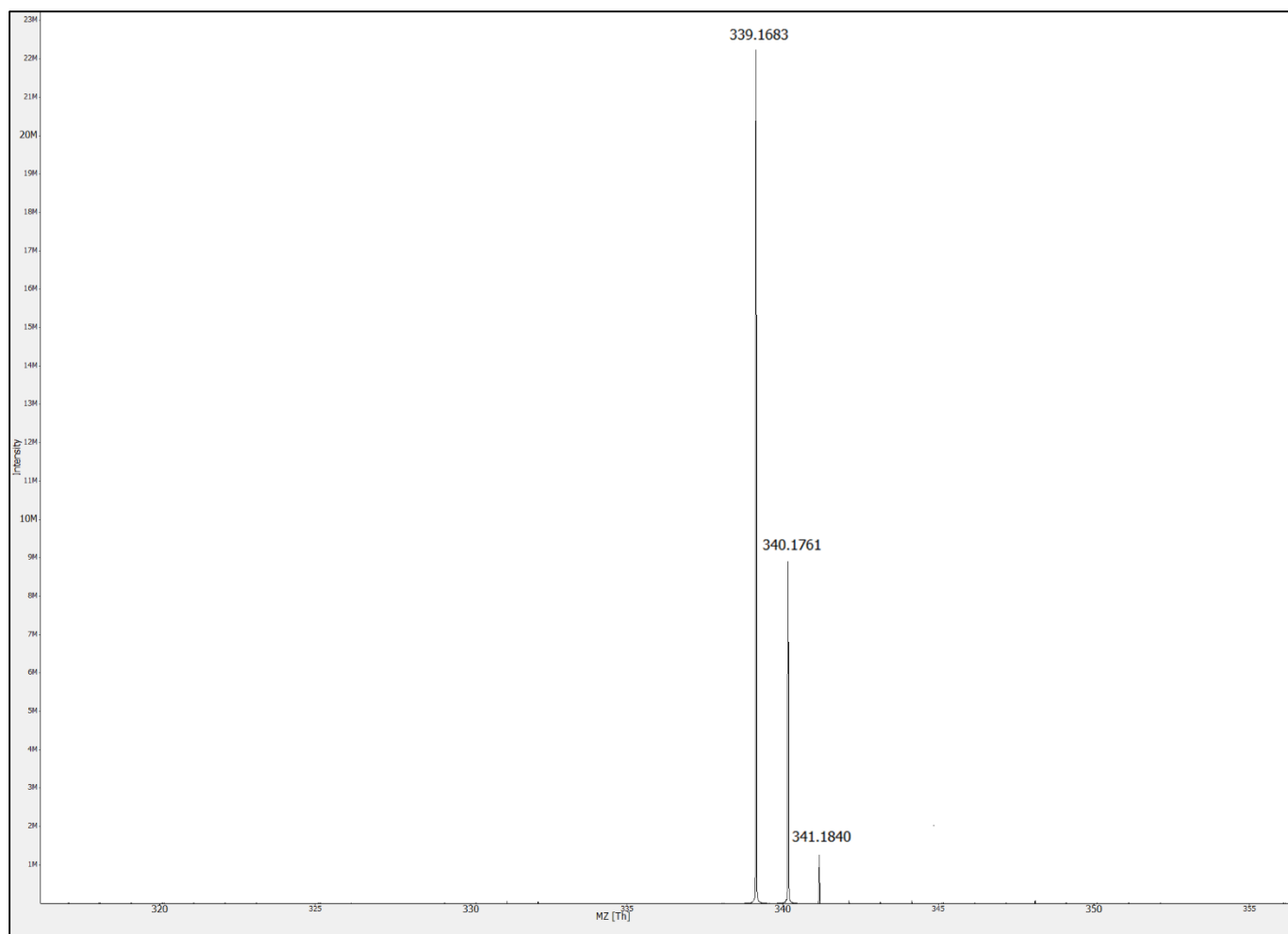


Figure S2. 600 MHz ^1H NMR spectrum of asperopiperazine A (**1**) (CD_3OD).

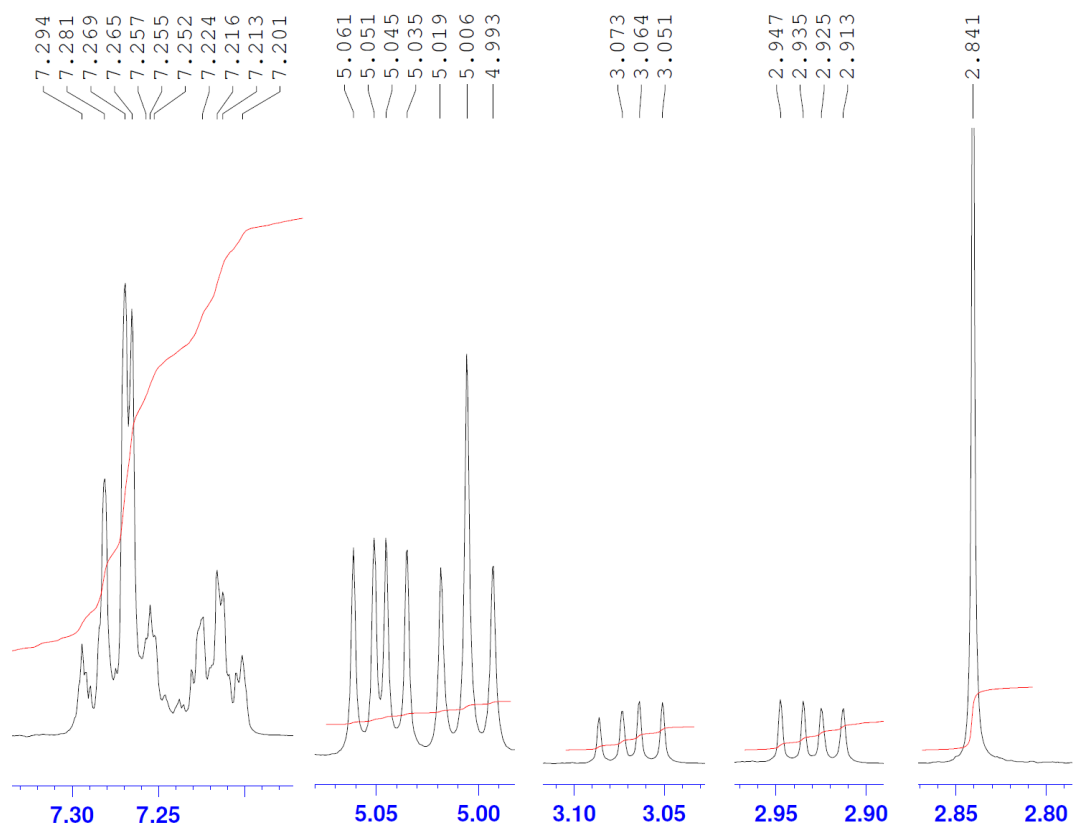
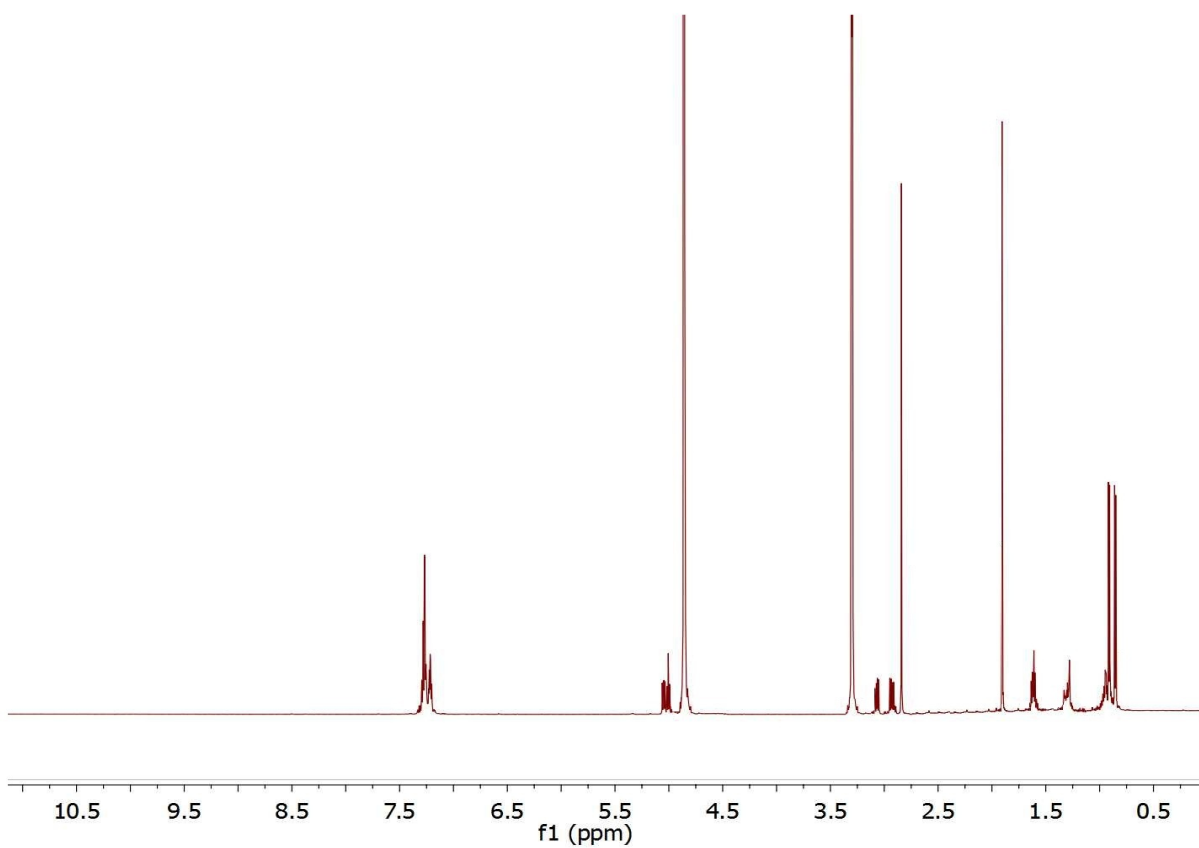


Figure S2. 600 MHz ^1H NMR spectrum of asperopiperazine A (**1**) (CD_3OD) (Cont.).

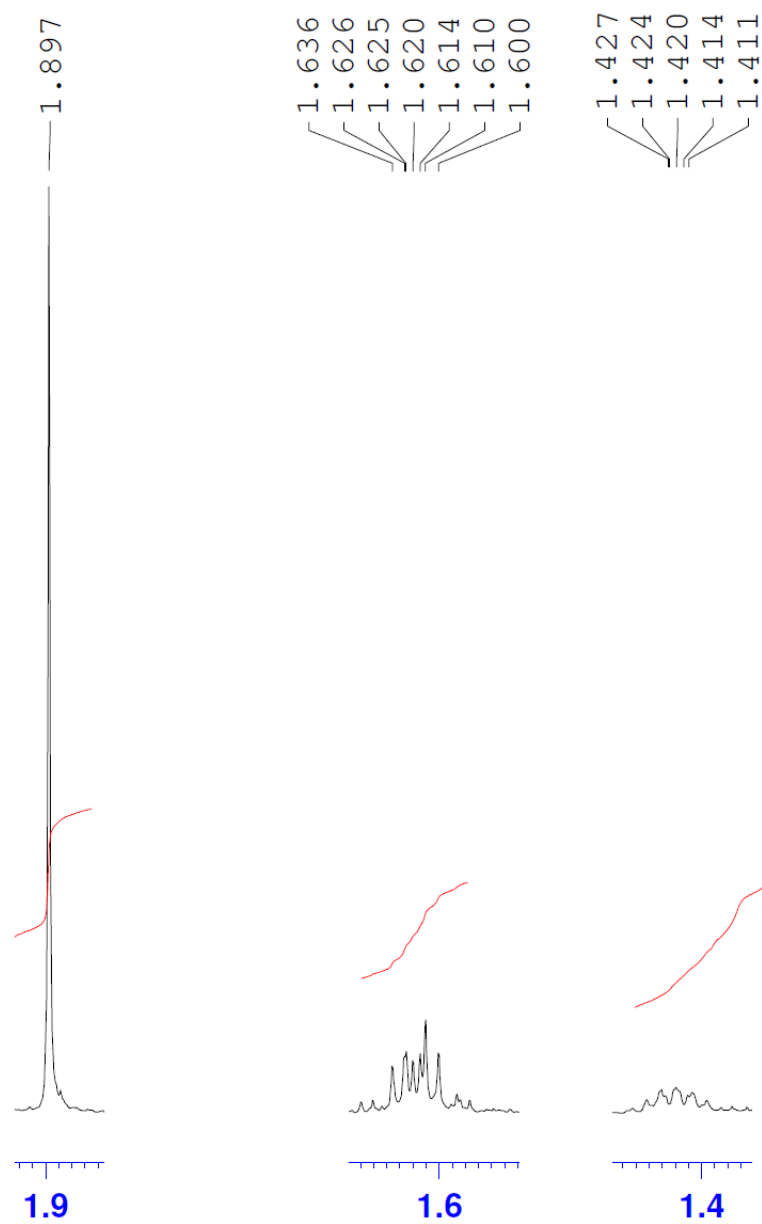


Figure S3. 150 MHz ^{13}C NMR spectrum of asperopiperazine A (**1**) (CD_3OD).

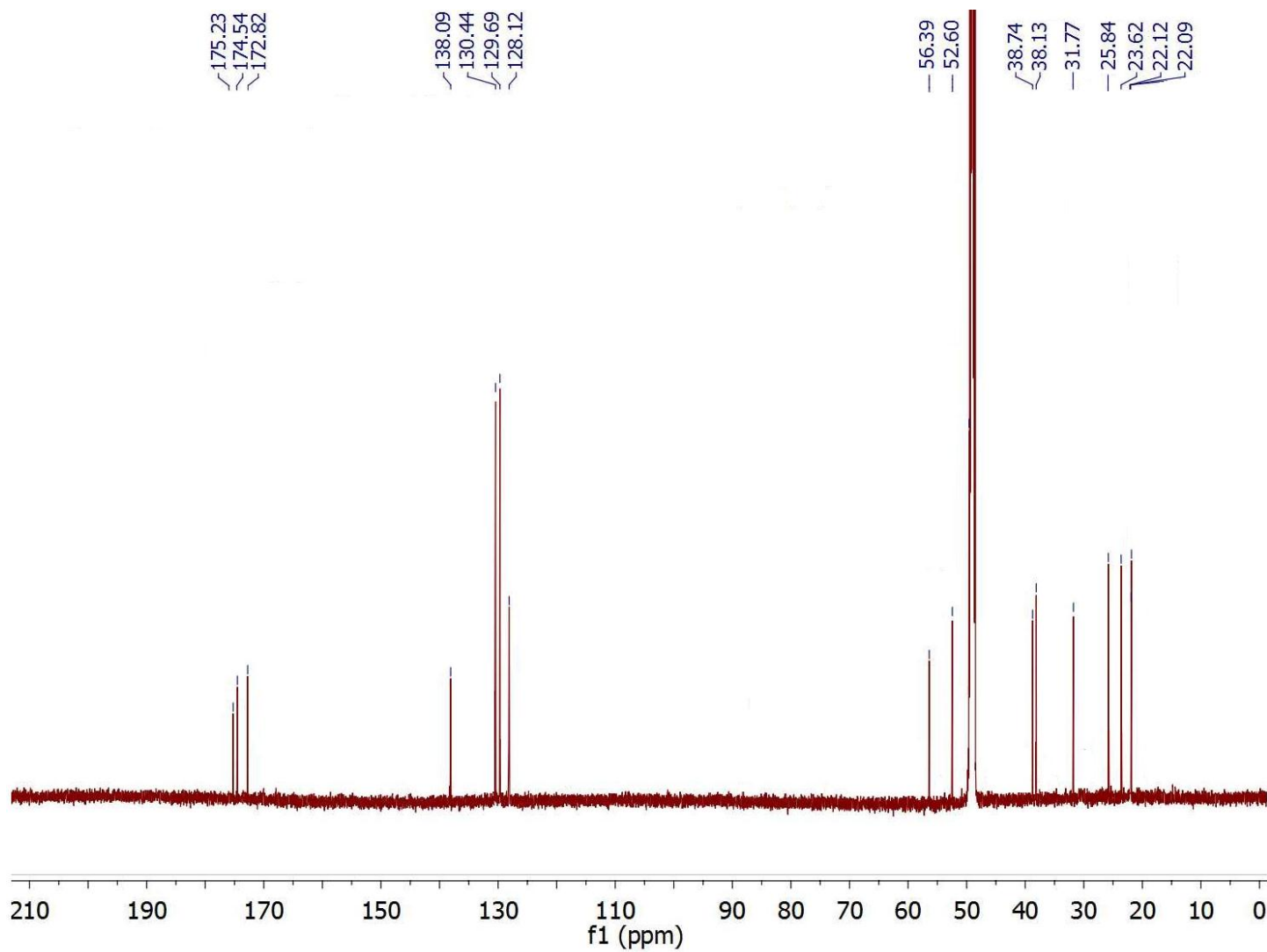


Figure S4. DEPT spectrum of asperopiperazine A (**1**) (CD₃OD).

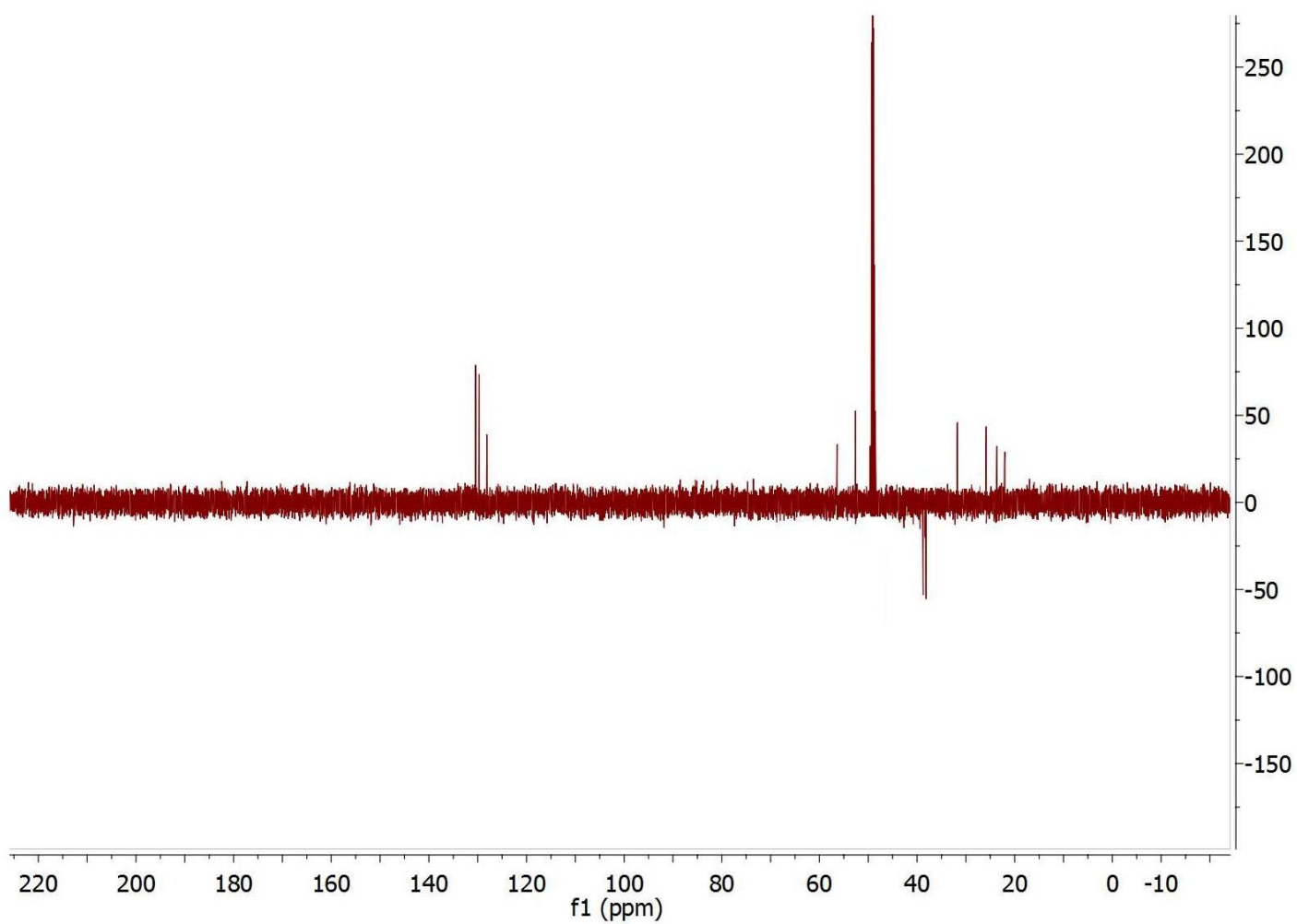


Figure S5. COSY spectrum of asperopiperazine A (**1**) (CD₃OD).

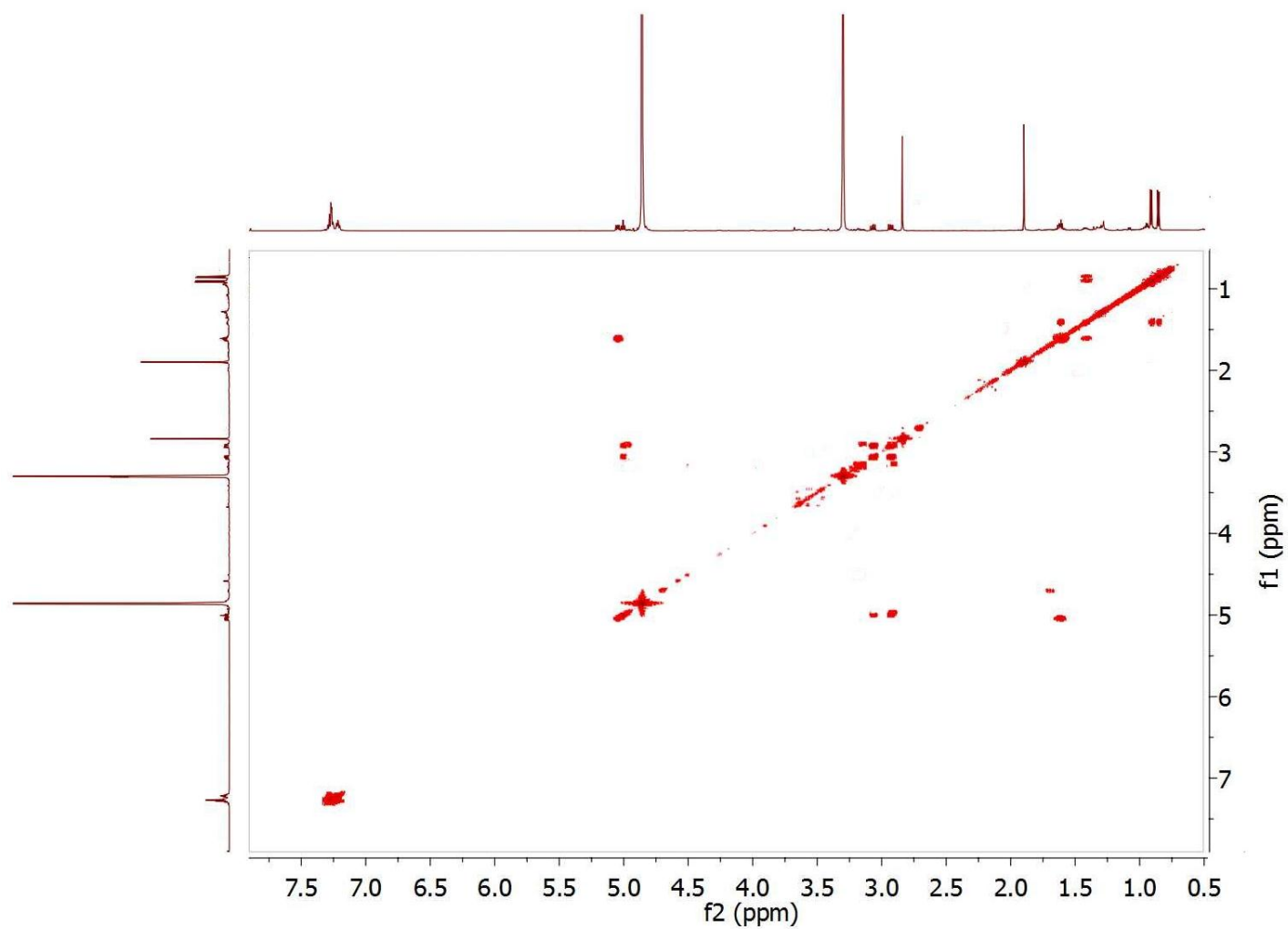


Figure S6. HSQC spectrum of asperopiperazine A (**1**) (CD₃OD).

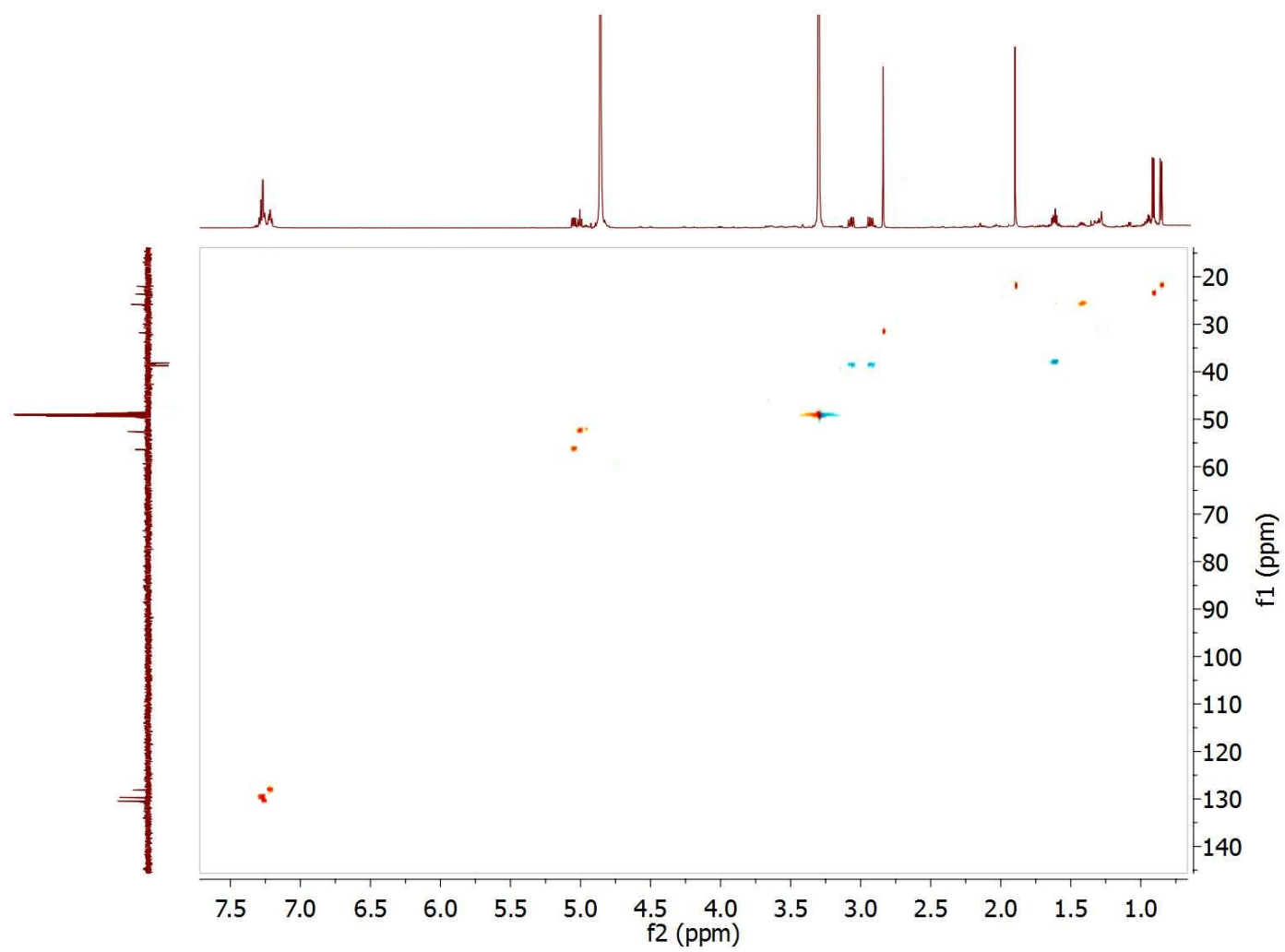


Figure S7. HMBC spectrum of asperopiperazine A (**1**) (CD₃OD).

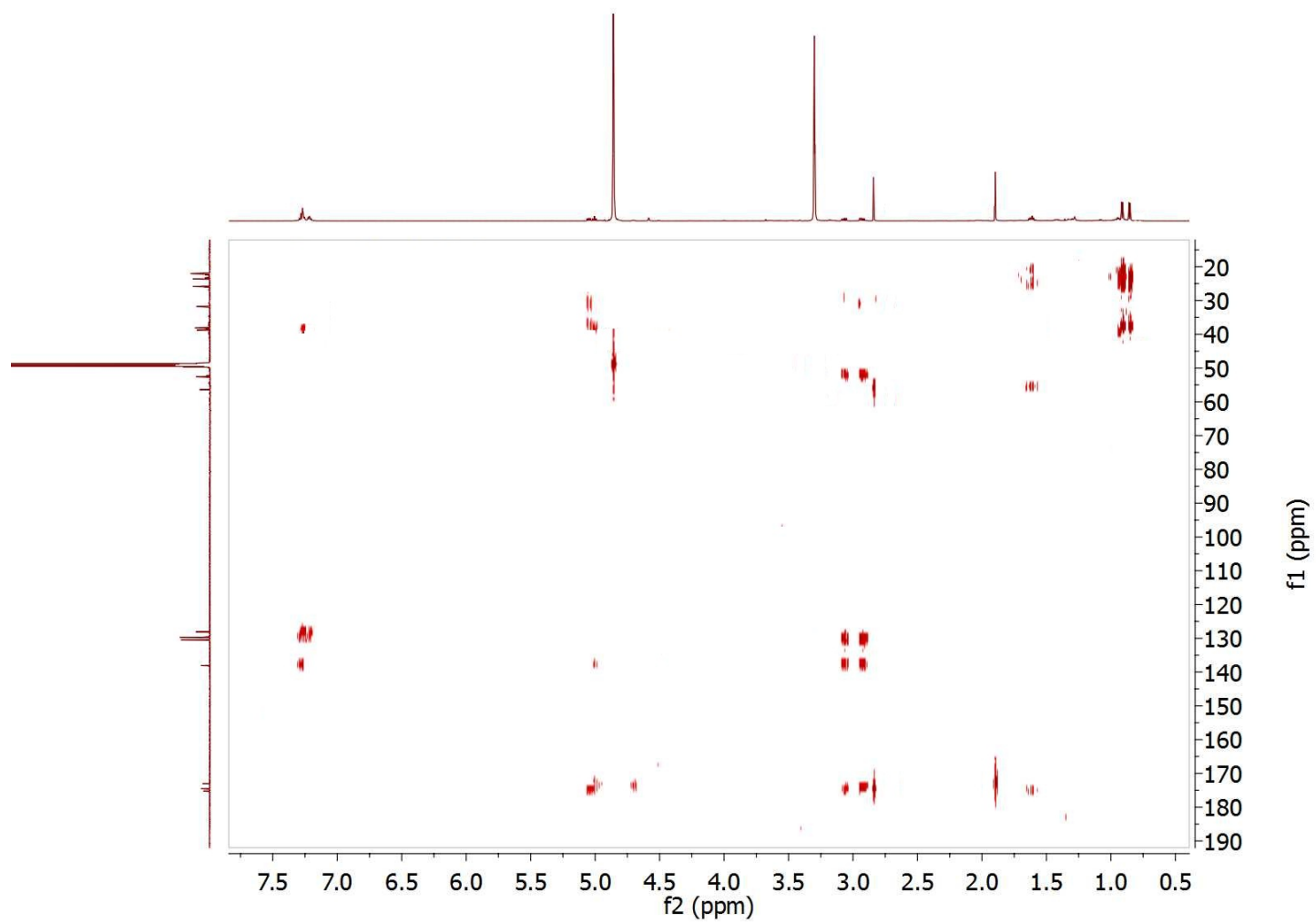


Figure S8. HRESIMS spectrum of asperopiperazine B (2).

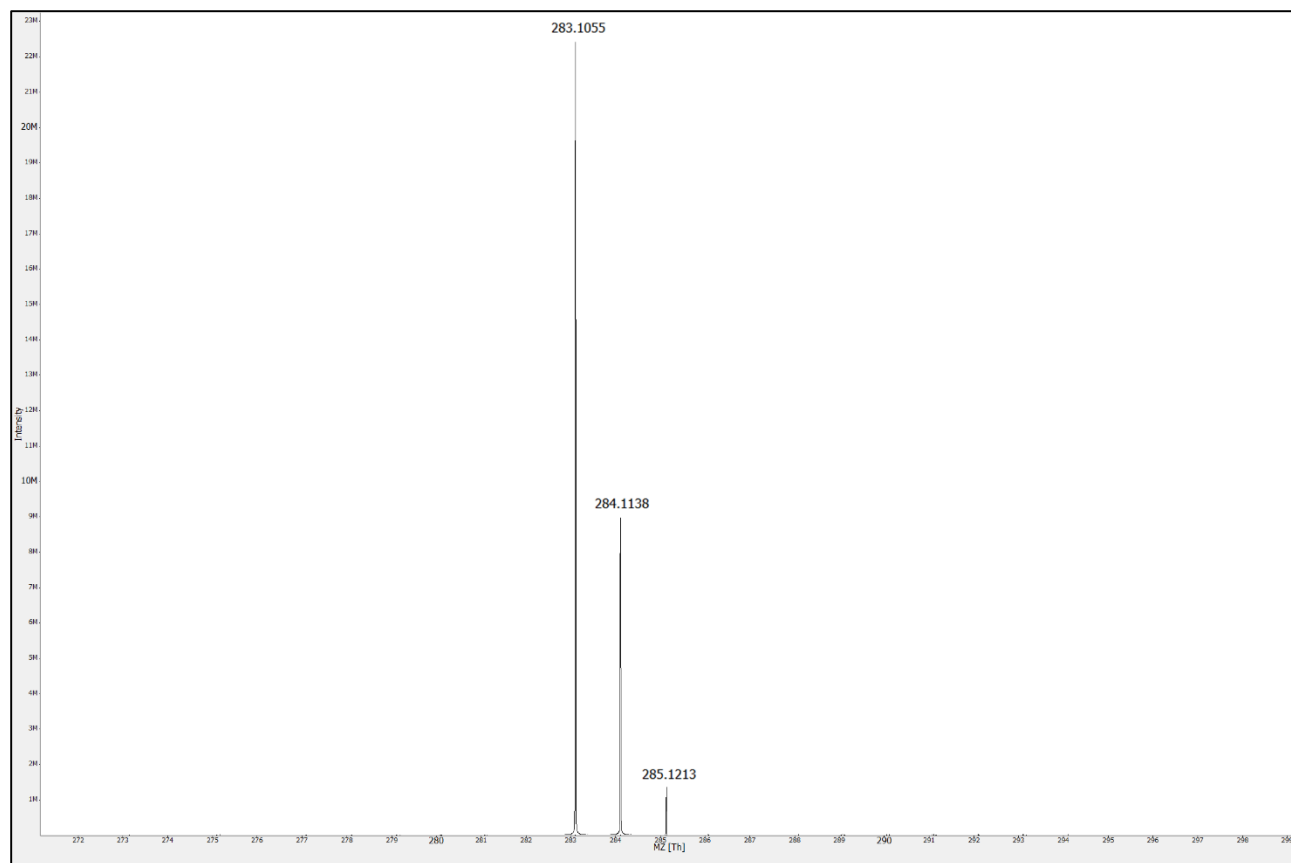


Figure S9. 850 MHz ^1H NMR spectrum of asperopiperazine B (**2**) (CDCl_3).

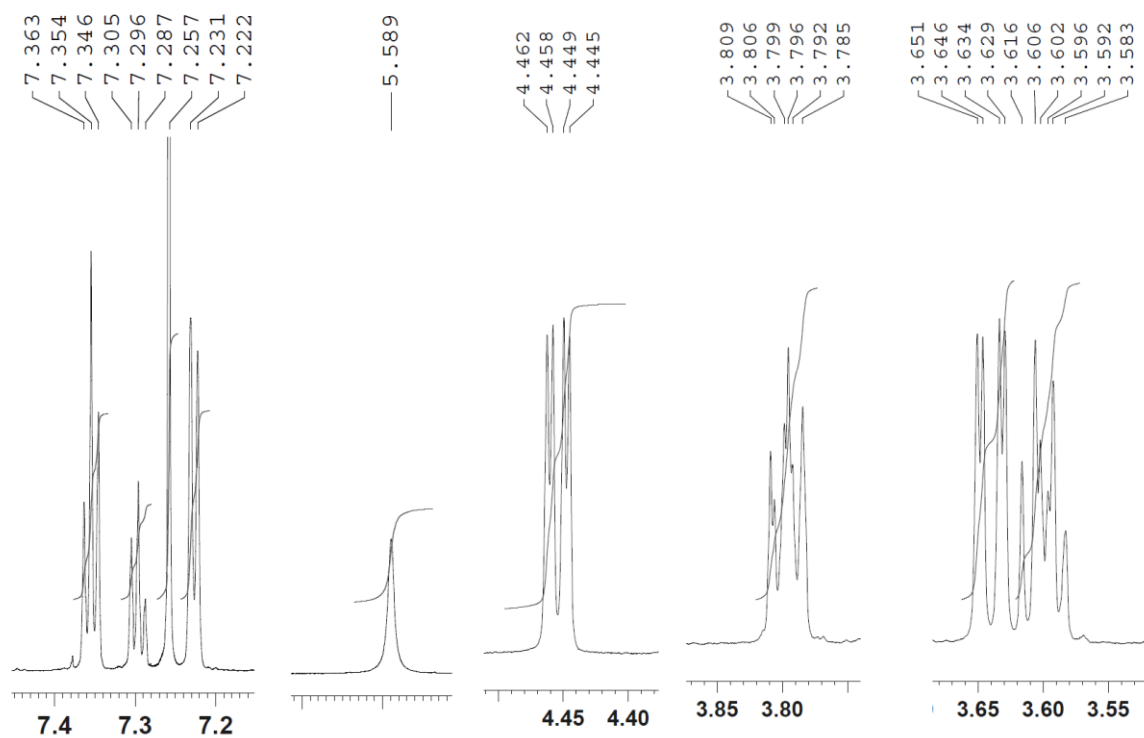
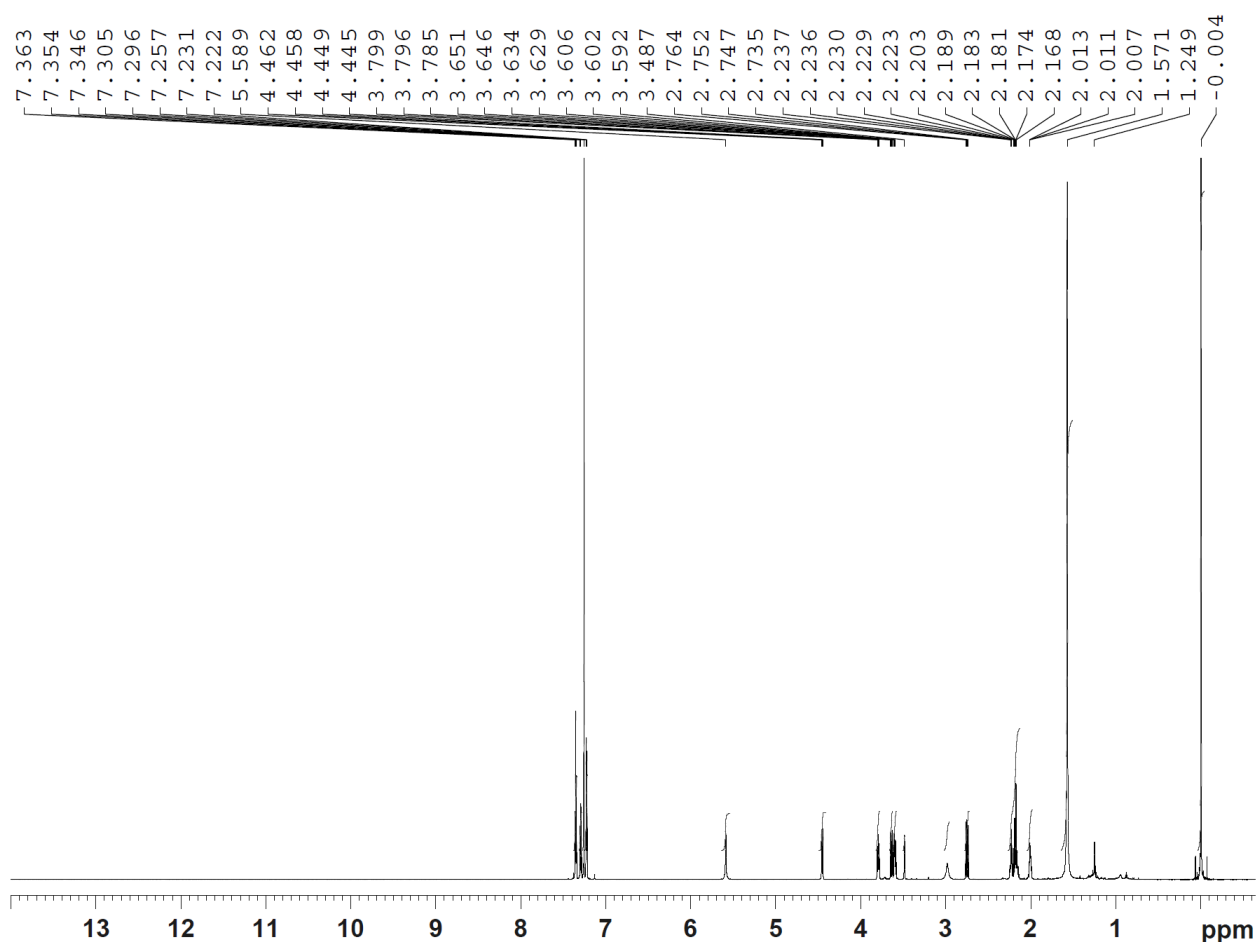


Figure S9. 850 MHz ^1H NMR spectrum of asperopiperazine B (**2**) (CDCl_3) (Cont.).

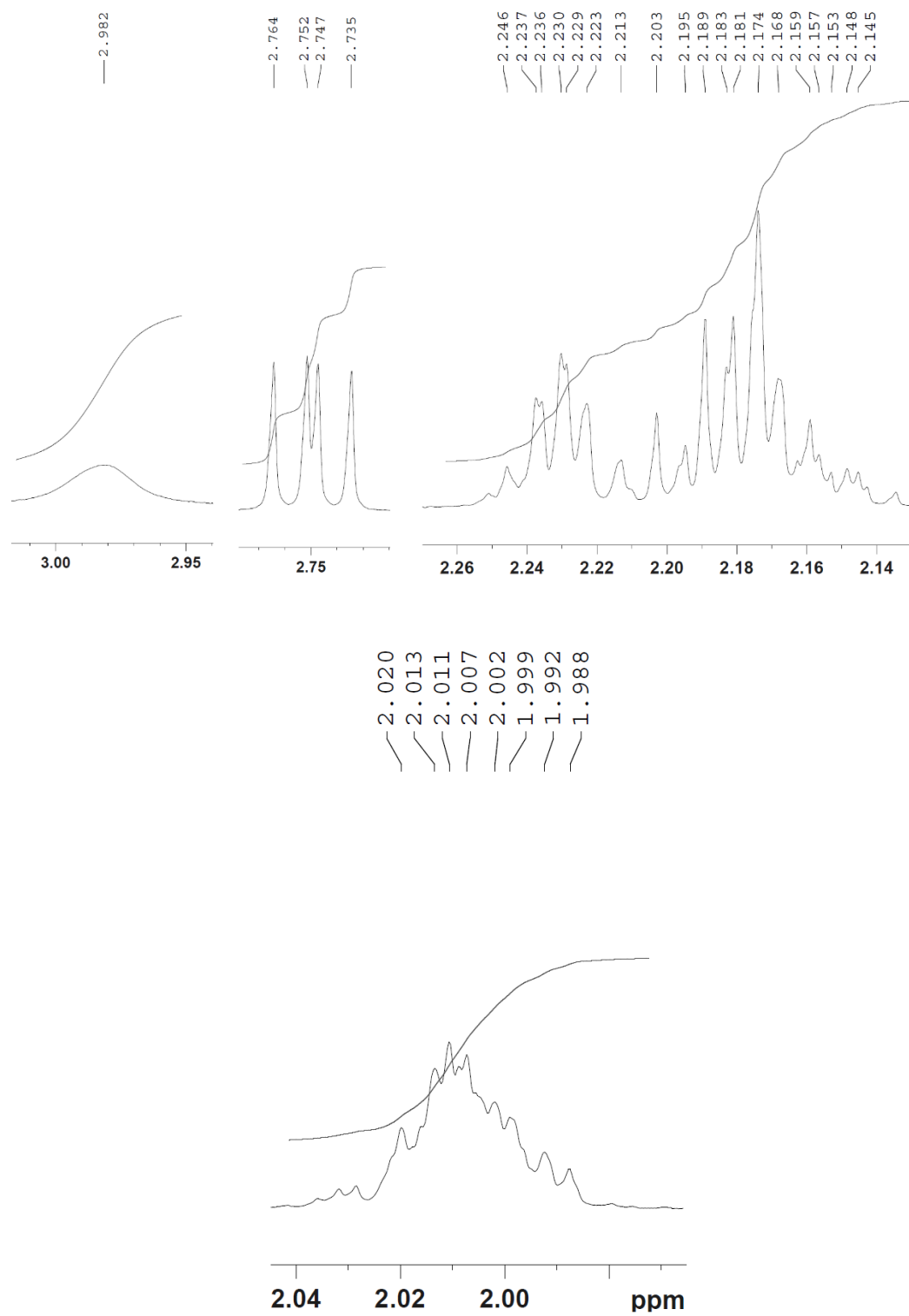


Figure S10. 213 MHz ^{13}C NMR spectrum of asperopiperazine B (**2**) (CDCl_3).

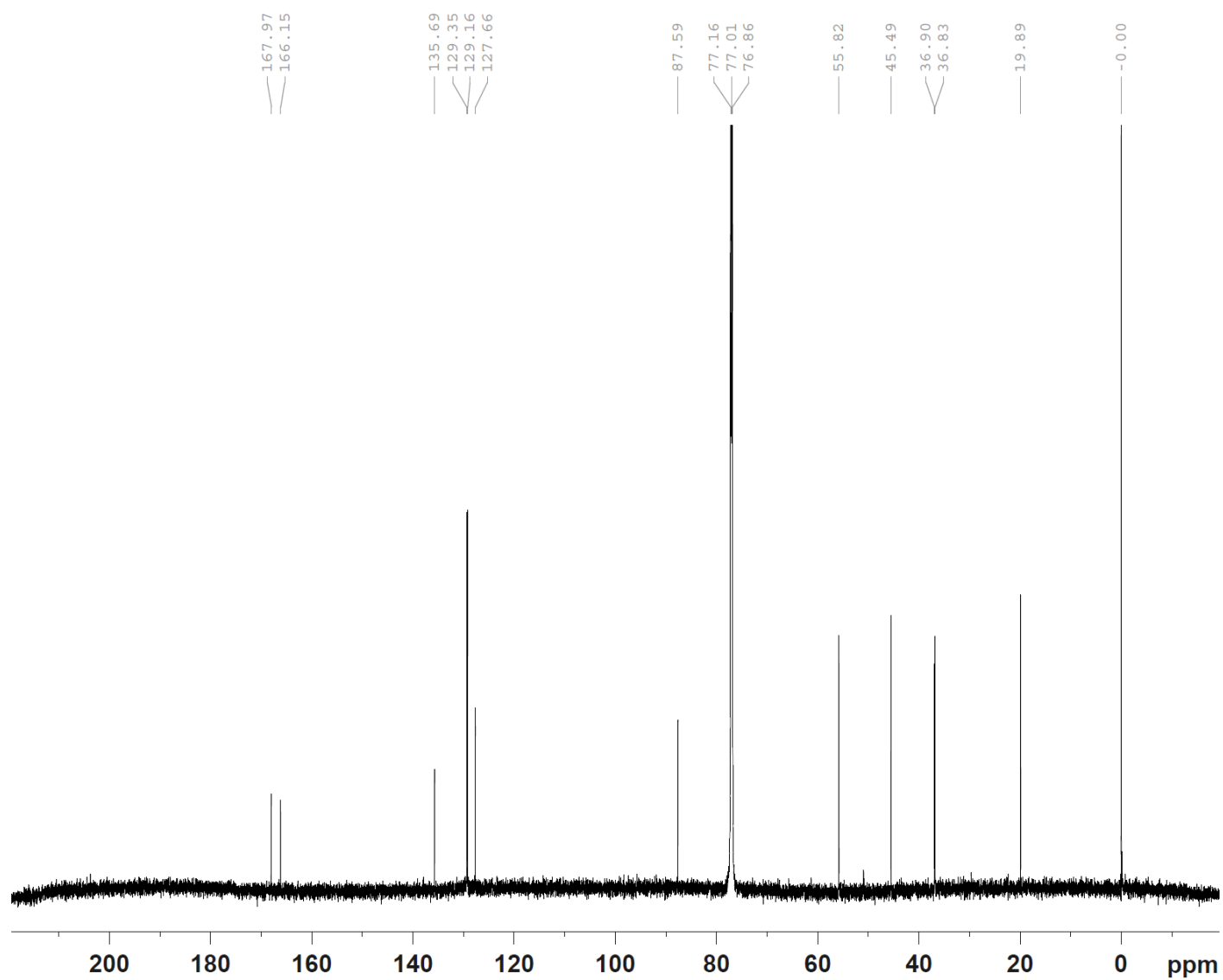


Figure S11. COSY spectrum of asperopiperazine B (**2**) (CDCl_3).

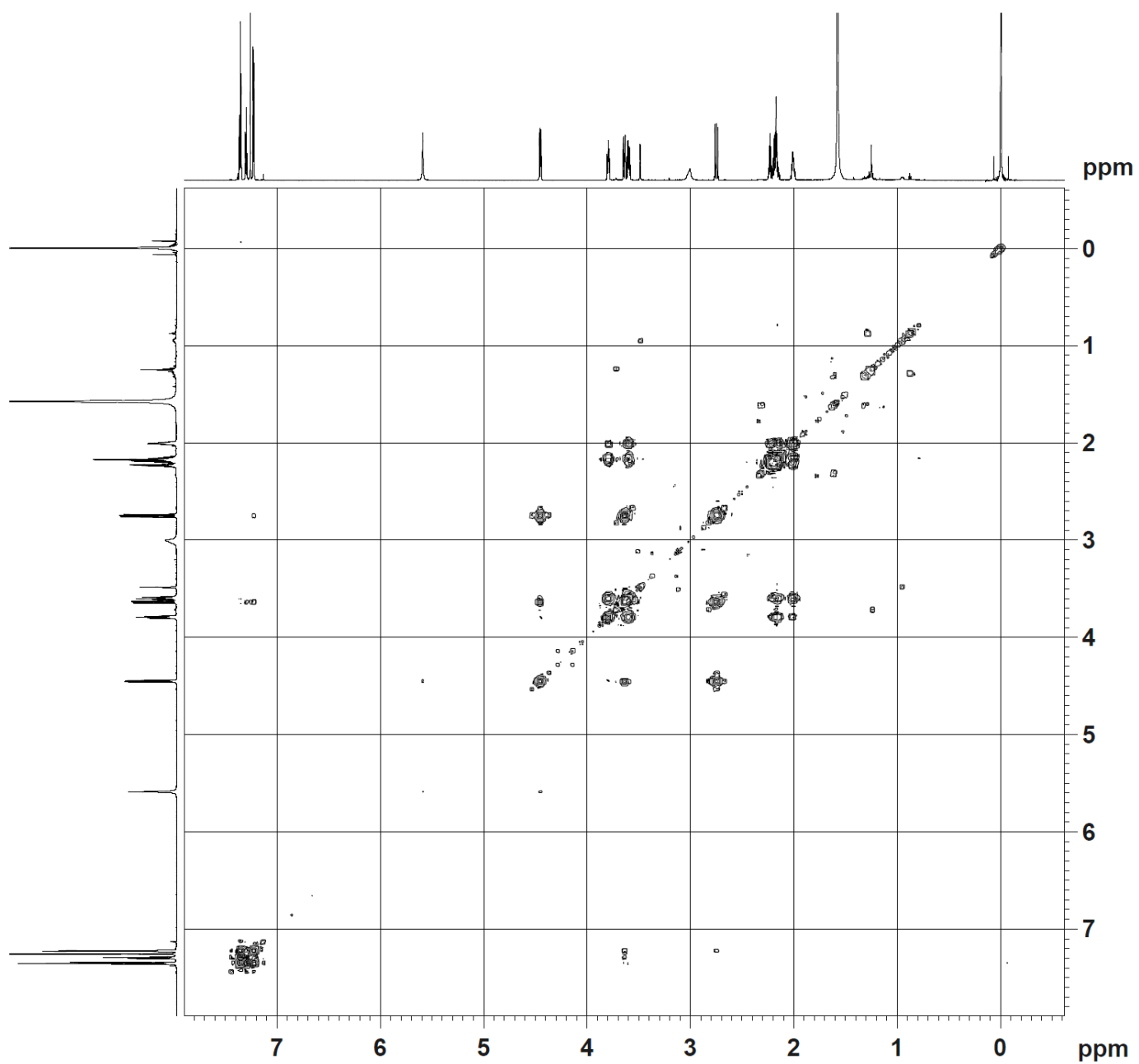


Figure S12. HSQC spectrum of asperopiperazine B (2) (CDCl_3).

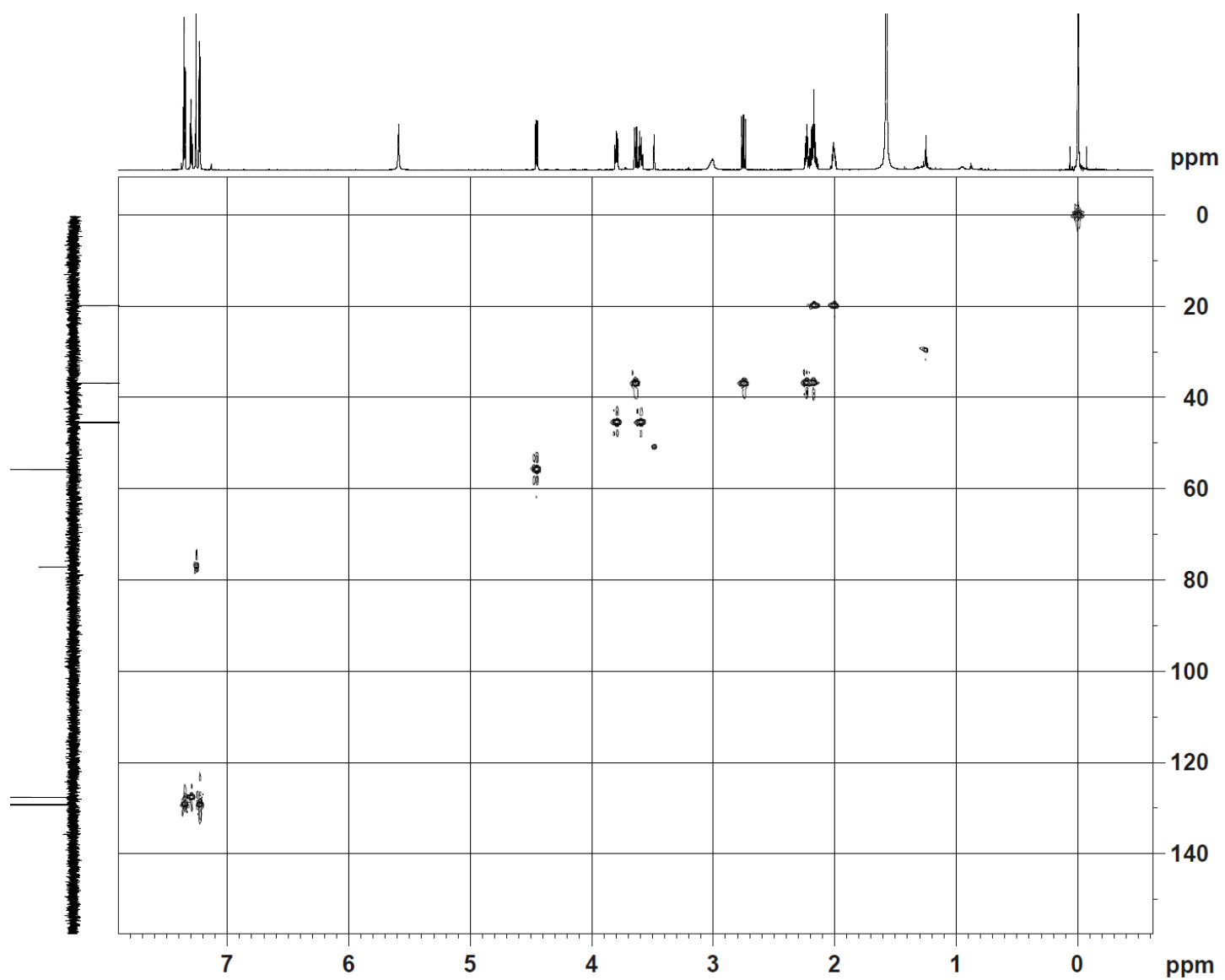


Figure S13. HMBC spectrum of asperopiperazine B (**2**) (CDCl_3).

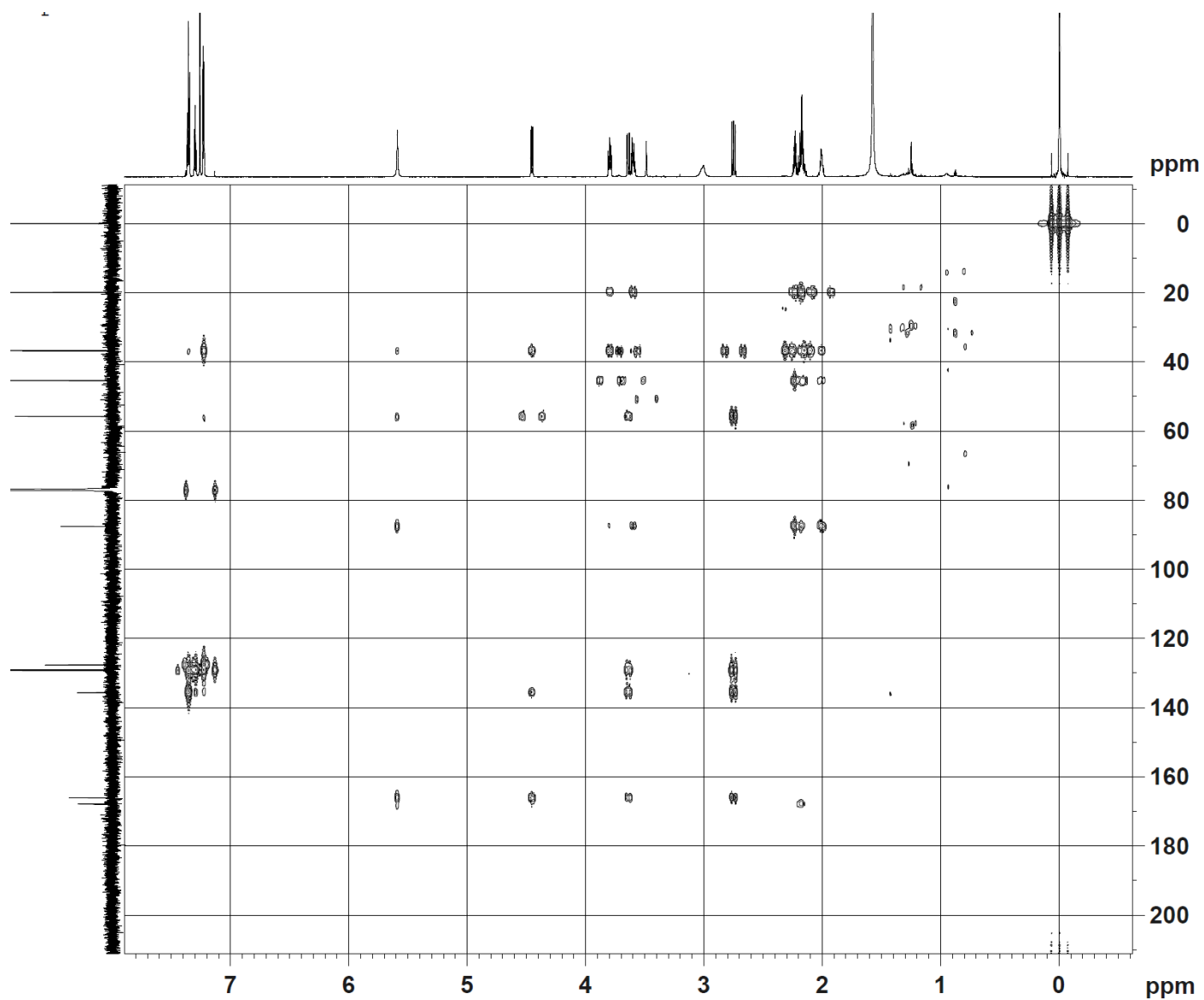


Figure S14. NOESY spectrum of asperopiperazine B (**2**) (CDCl_3).

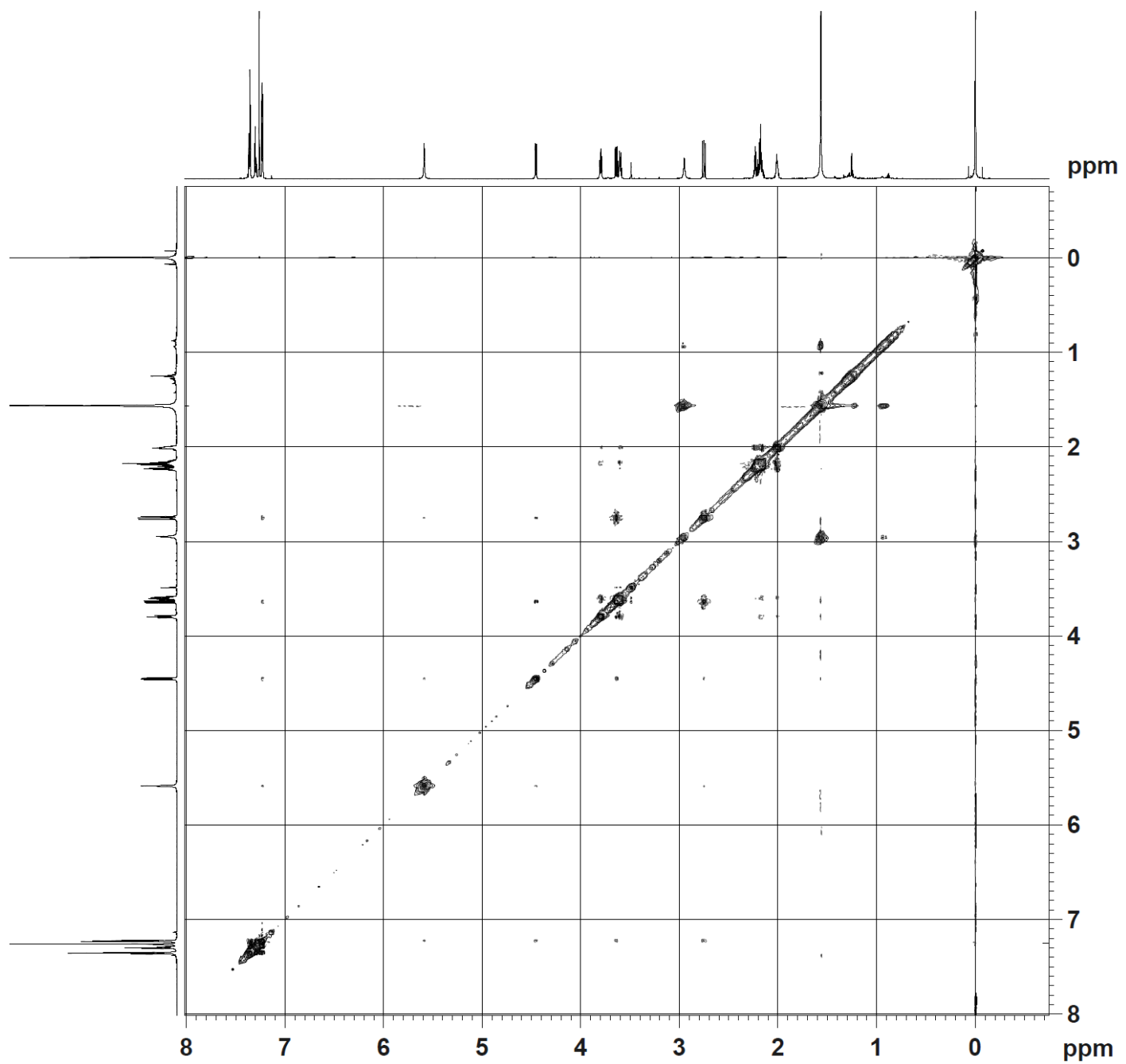


Figure 15. HRESIMS spectrum of (+)-citreoisocoumarin (**3**).

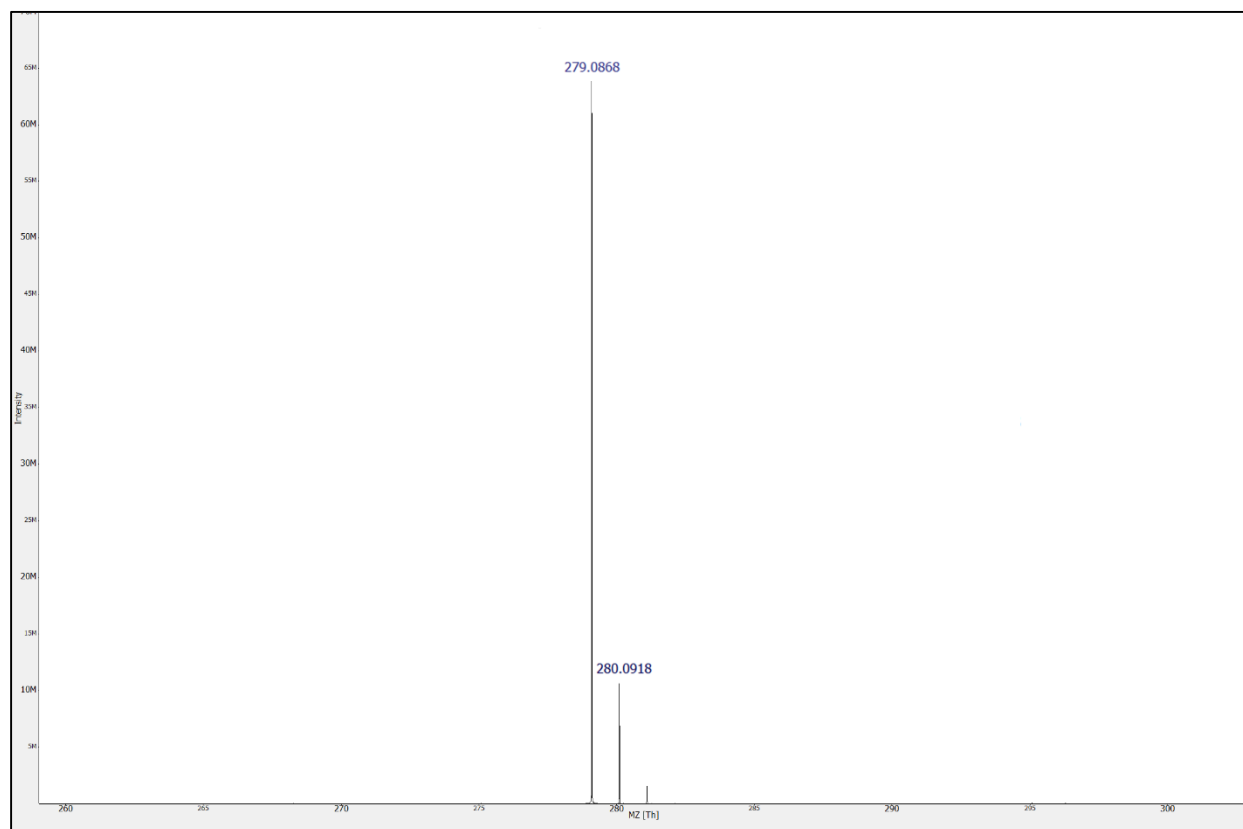


Figure S16. 600 MHz ^1H NMR spectrum of (+)-citreoisocoumarin (**3**) (CDCl_3).

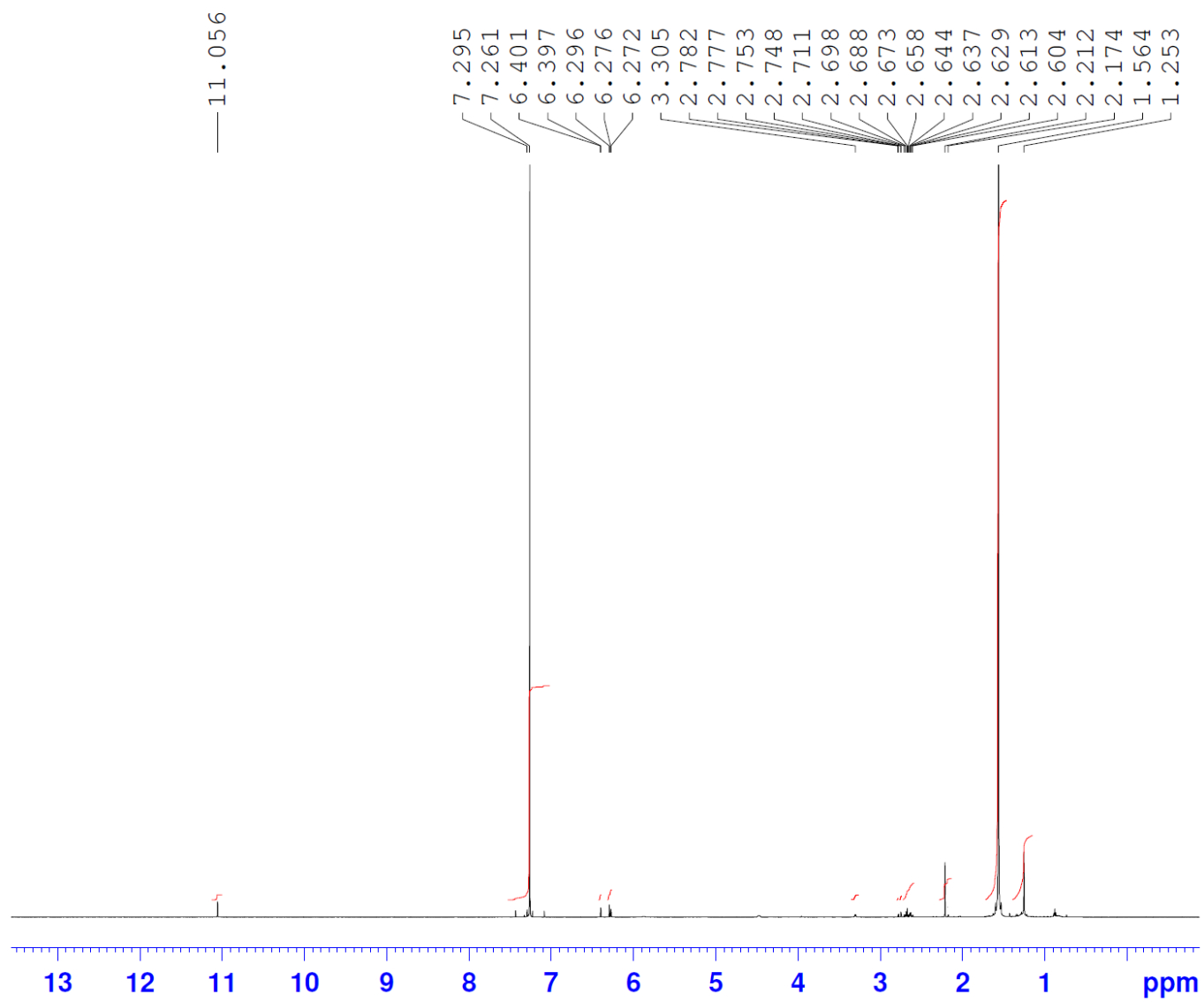


Figure S16. 600 MHz ^1H NMR spectrum of (+)-citreisocoumarin (**3**) (CDCl_3) (Cont.).

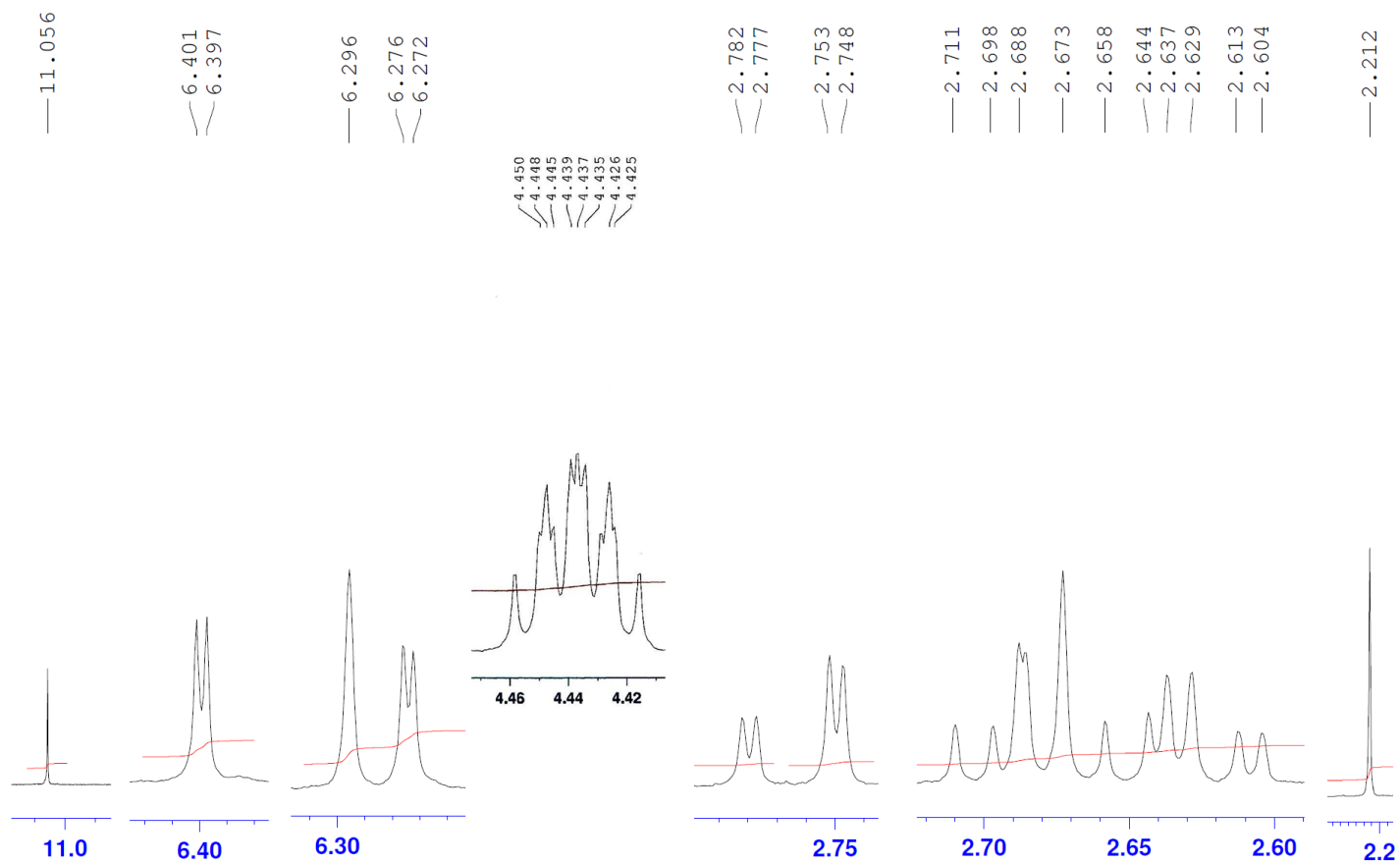


Figure S17. 150 MHz ^{13}C NMR spectrum of (+)-citreoisocoumarin (**3**) (CDCl_3).

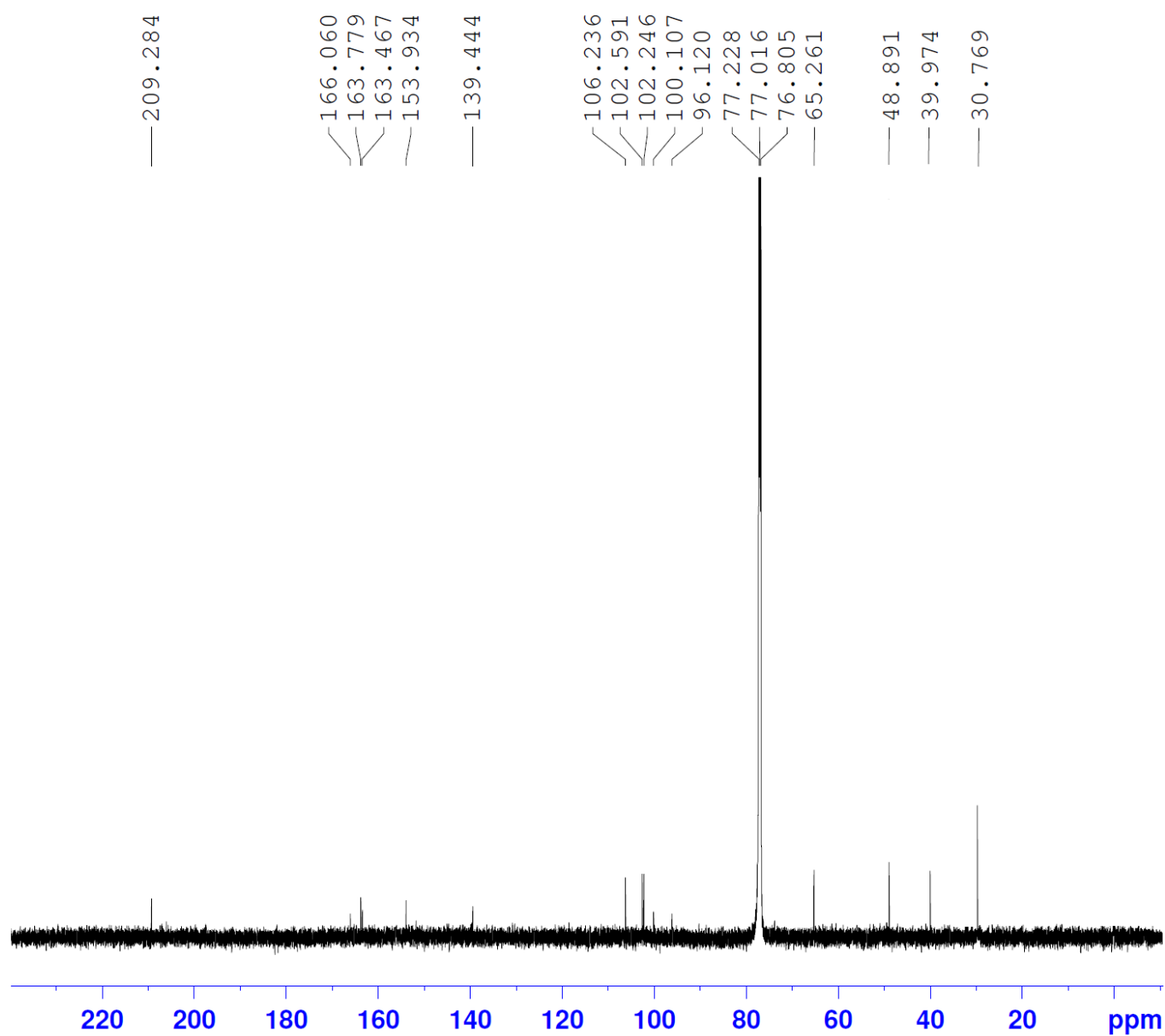


Figure S18. COSY spectrum of (+)-citreoisocoumarin (**3**) (CDCl₃).

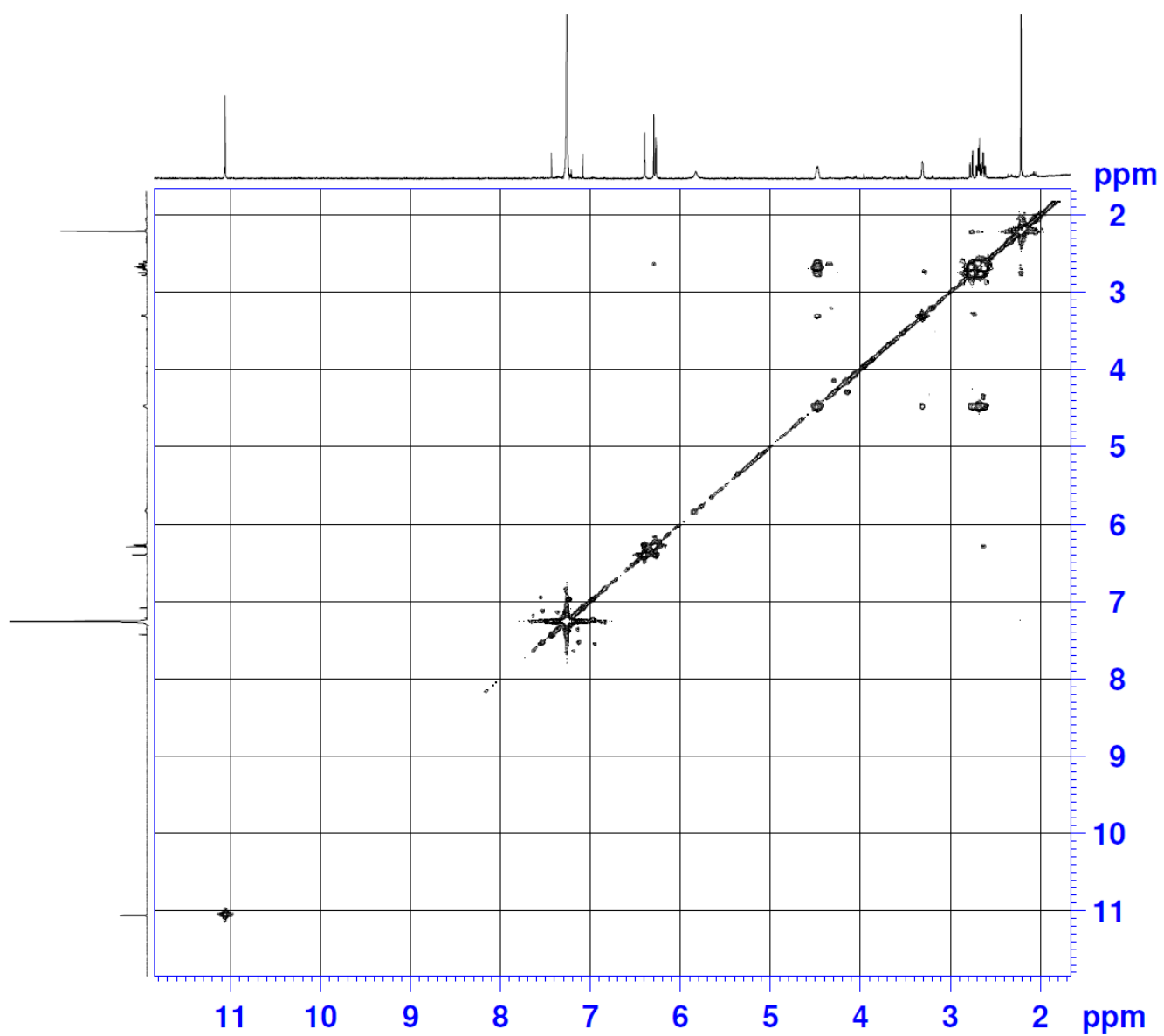


Figure S19. HSQC spectrum of (+)-citreoisocoumarin (**3**) (CDCl₃).

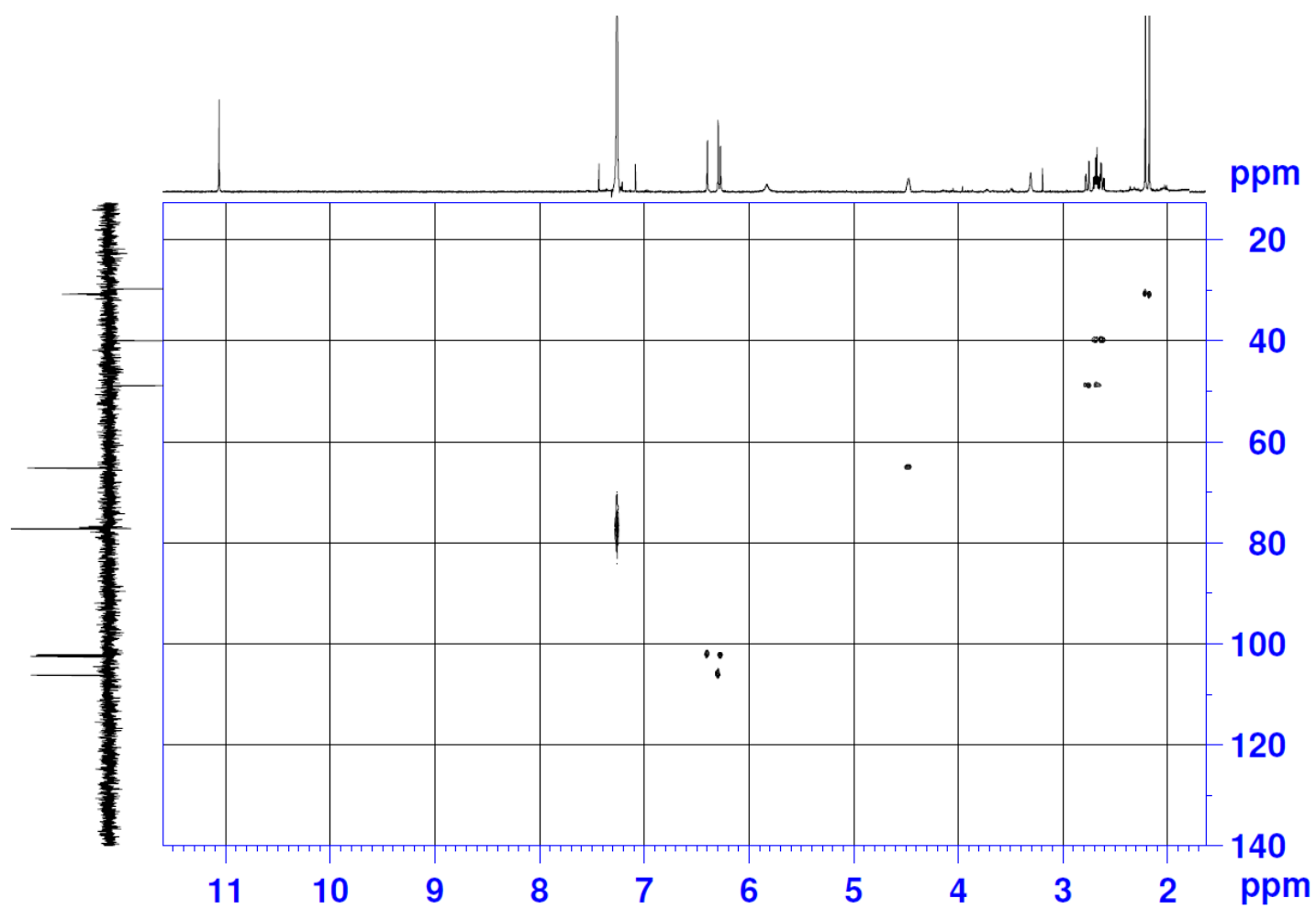


Figure S20. HMBC spectrum of (+)-citreisocoumarin (**3**) (CDCl₃).

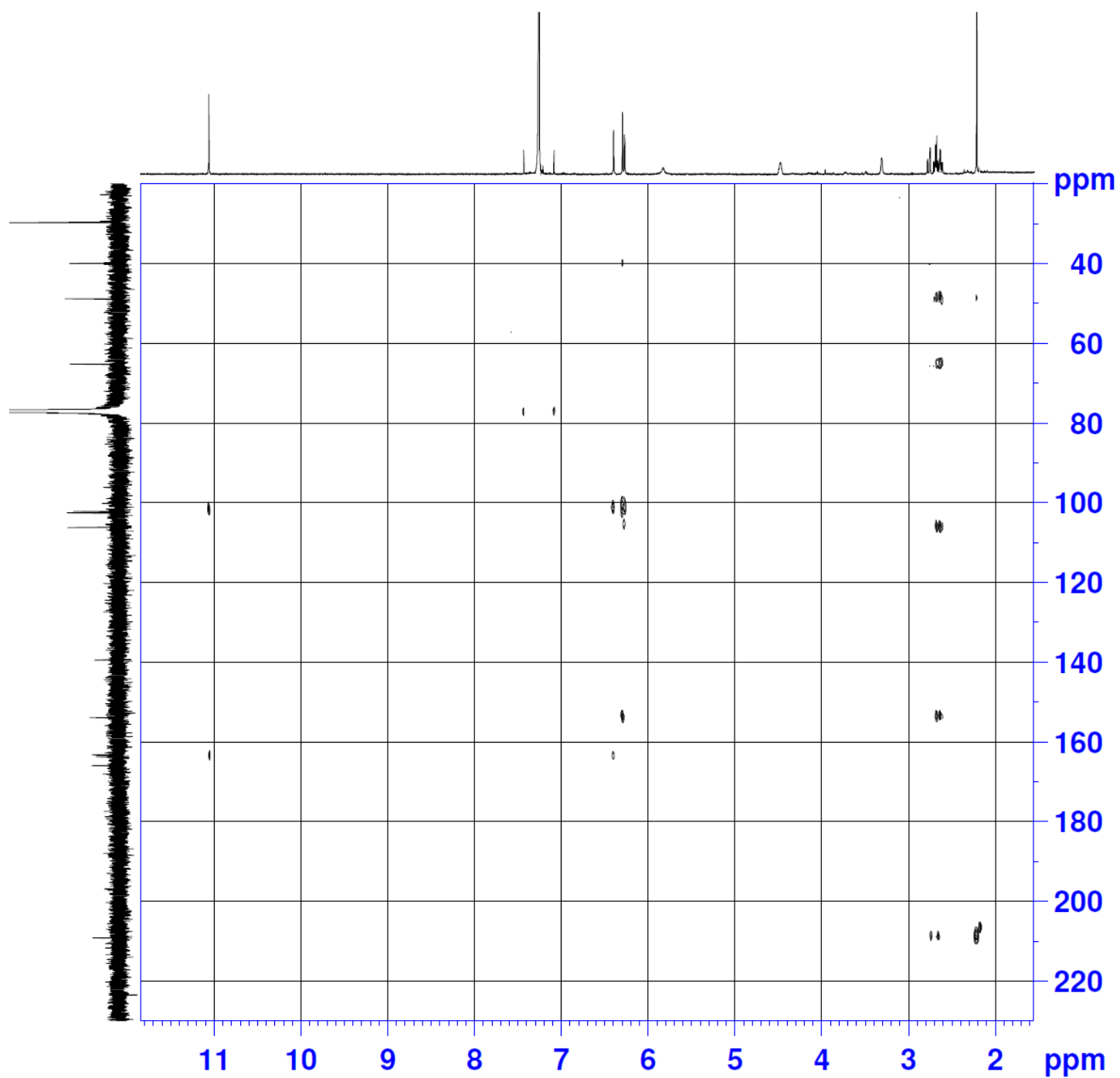


Figure 21. HRESIMS spectrum of (–)-6,8-di-*O*-methylocitreoisocoumarin (**4**).

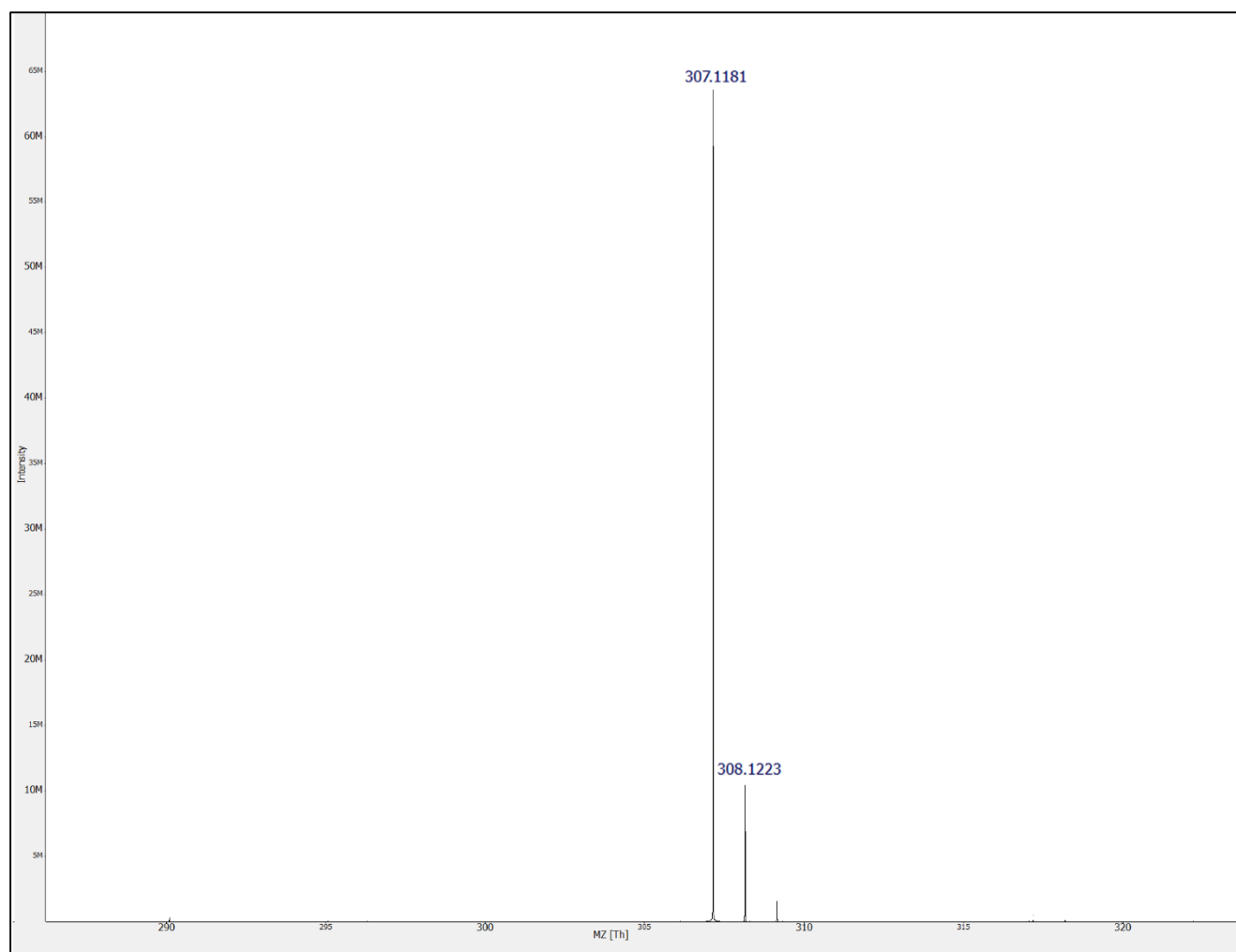


Figure S22. 600 MHz ^1H NMR spectrum of (-)-6,8-di-*O*-methylnitroisocoumarin (**4**) (CDCl_3).

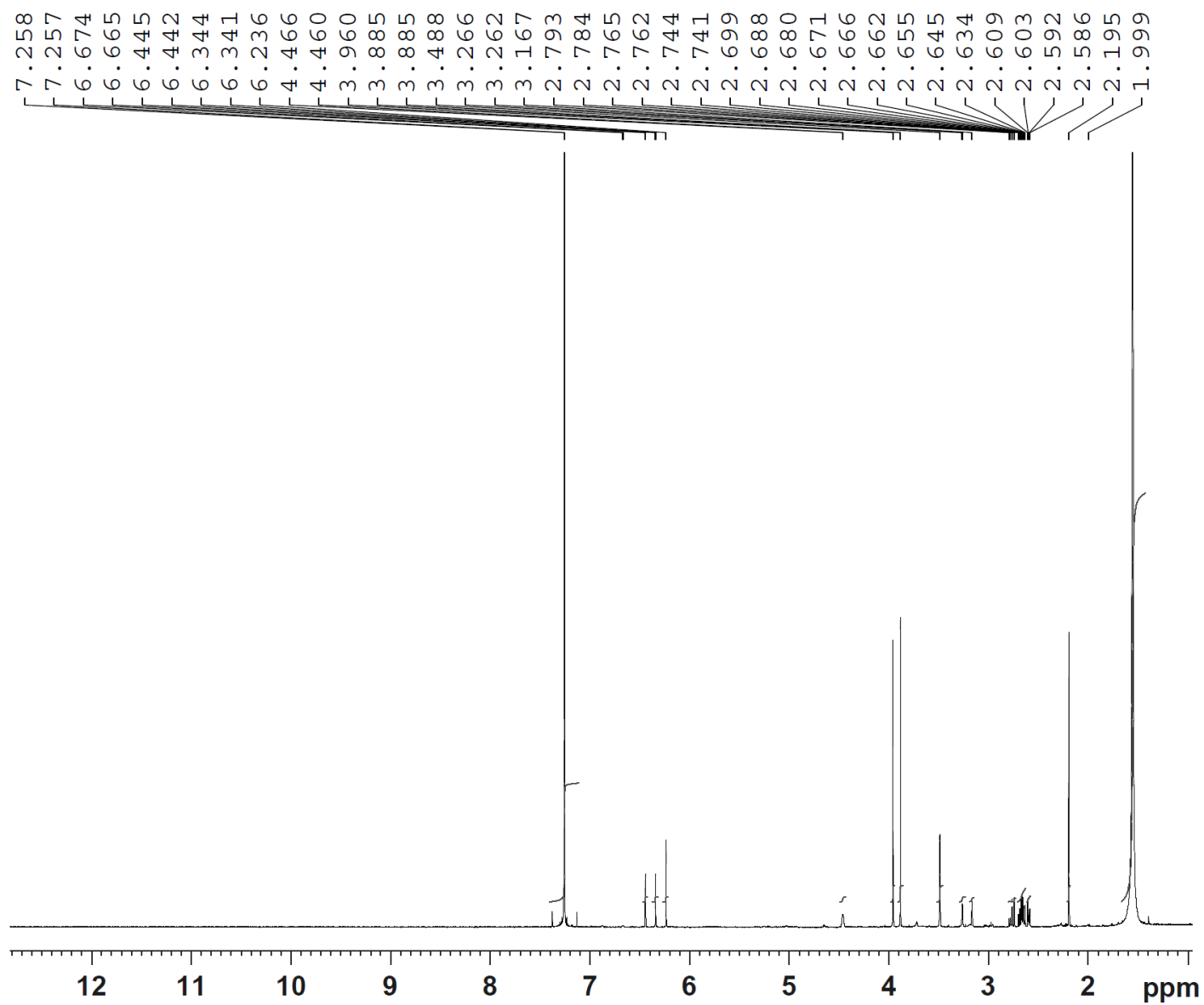


Figure S22. 600 MHz ^1H NMR spectrum of (-)-6,8-di-*O*-methylnitroisocoumarin (**4**) (CDCl_3) (Cont.).

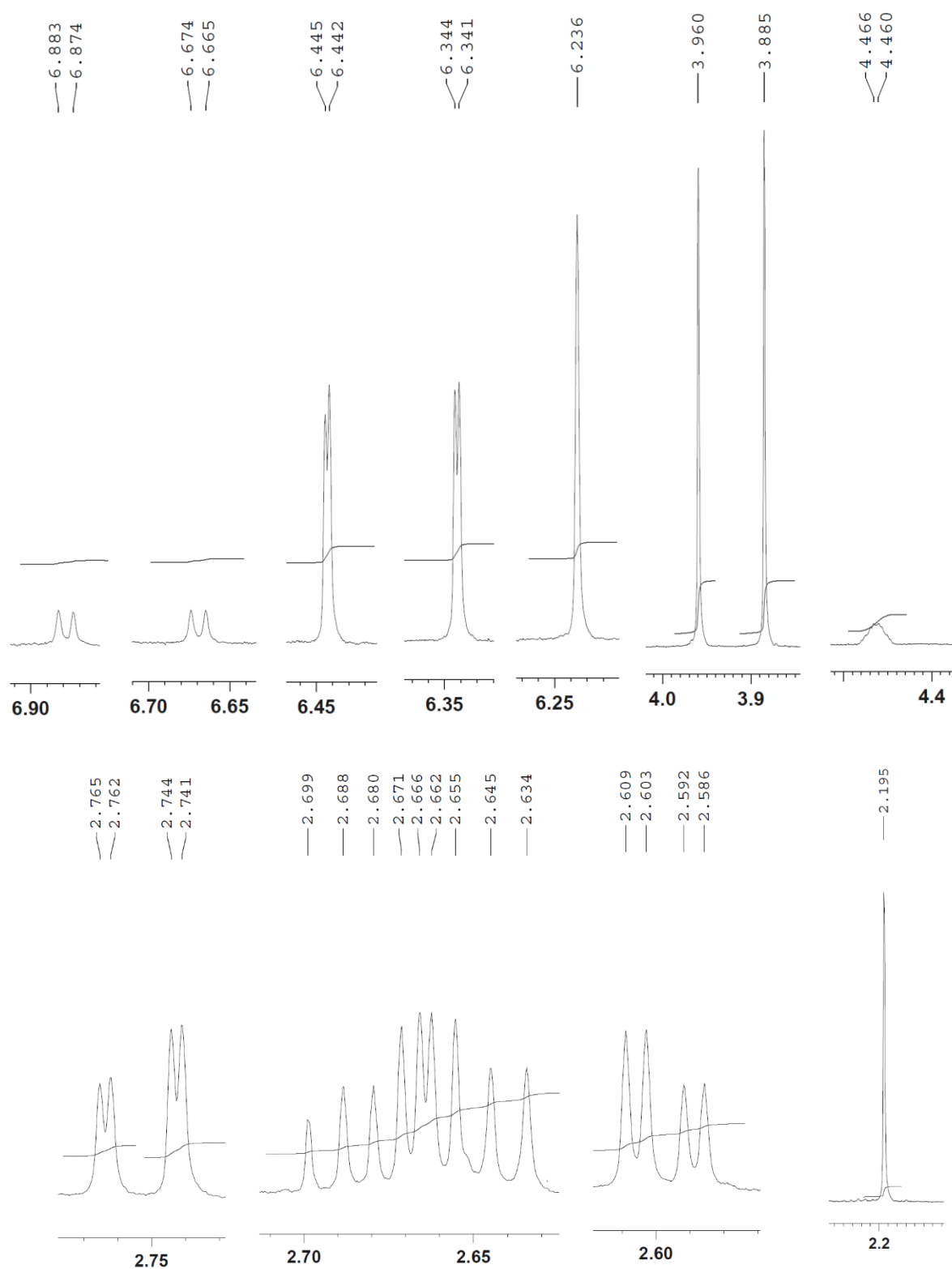


Figure S23. 150 MHz ^{13}C NMR spectrum of (–)-6,8-di-*O*-methylnitreoisocoumarin (**4**) (CDCl_3).

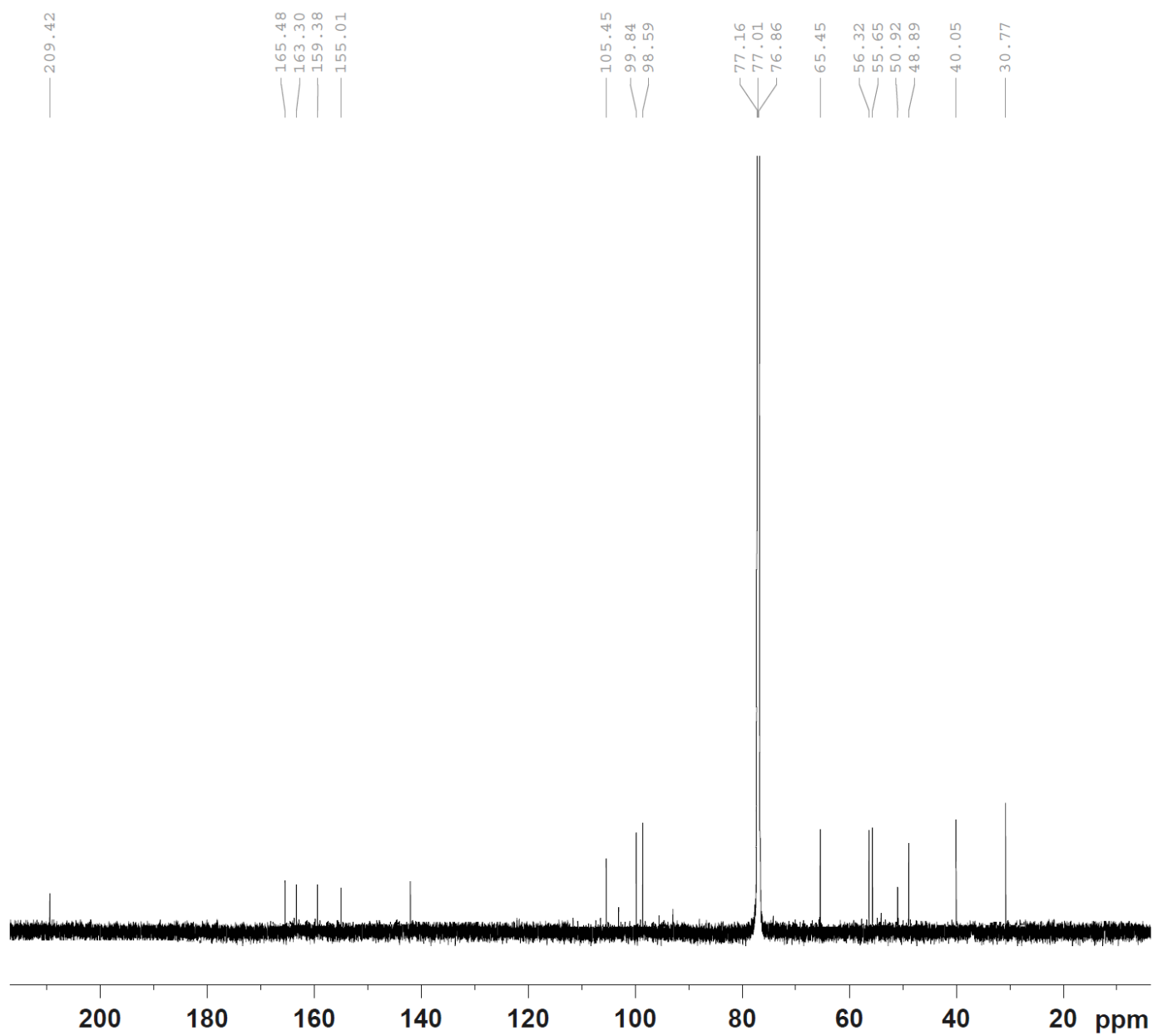


Figure S24. COSY spectrum of (-)-6,8-di-*O*-methylecitreoisocoumarin (**4**) (CDCl₃).

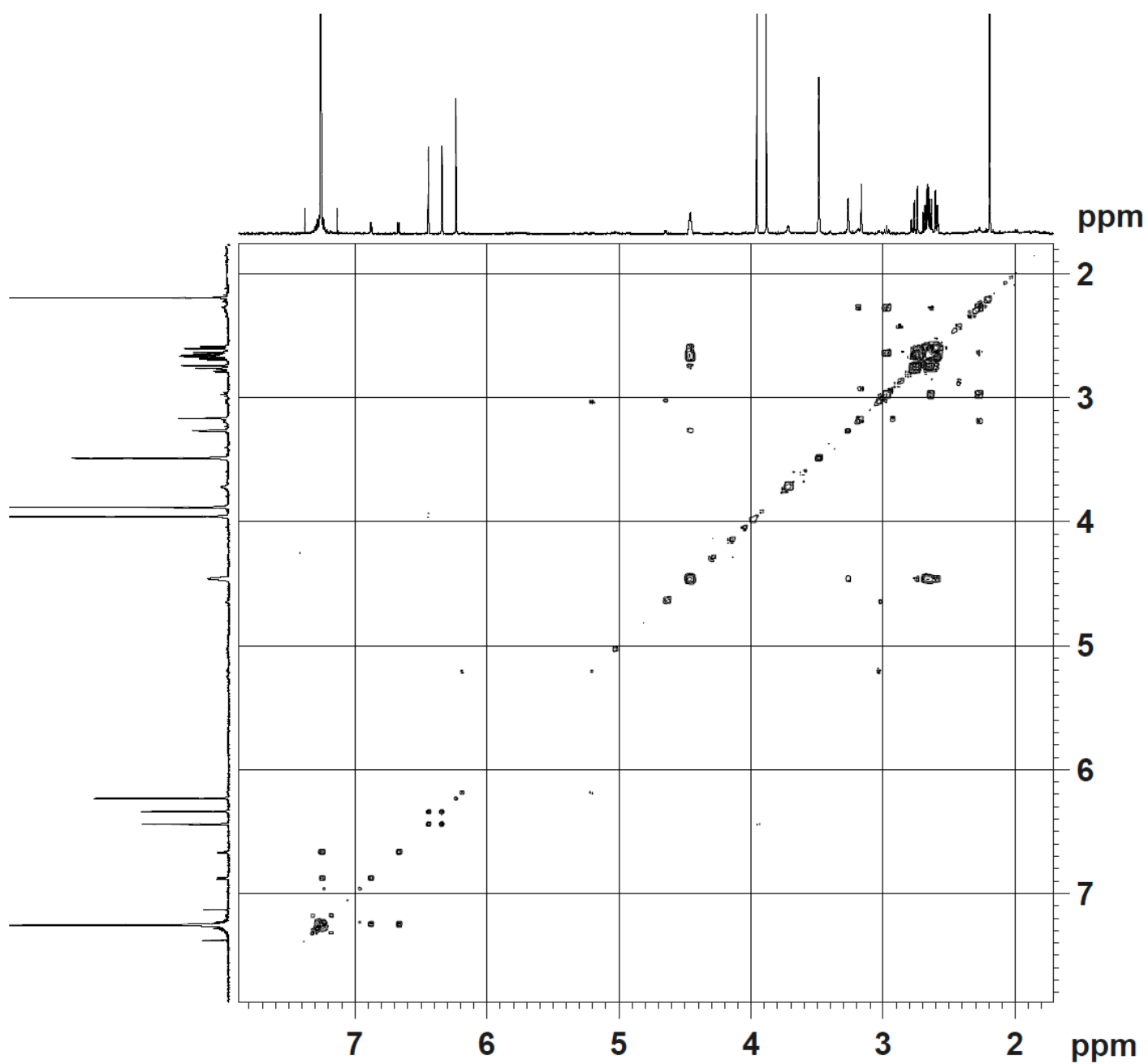


Figure S25. HSQC spectrum of (-)-6,8-di-*O*-methylocitreoisocoumarin (**4**) (CDCl₃).

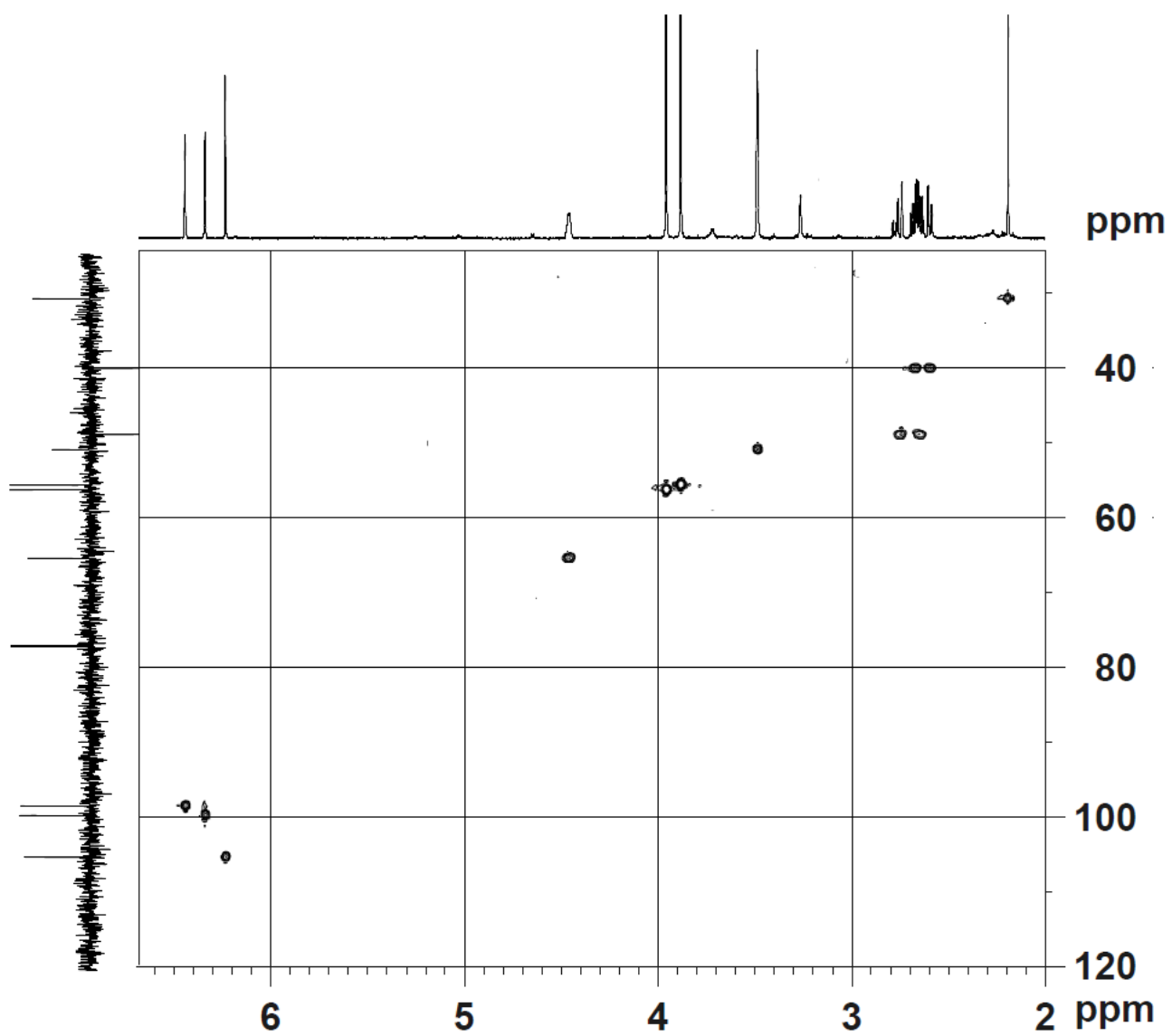


Figure S26. HMBC spectrum of (-)-6,8-di-*O*-methylcitreisocoumarin (**4**) (CDCl₃).

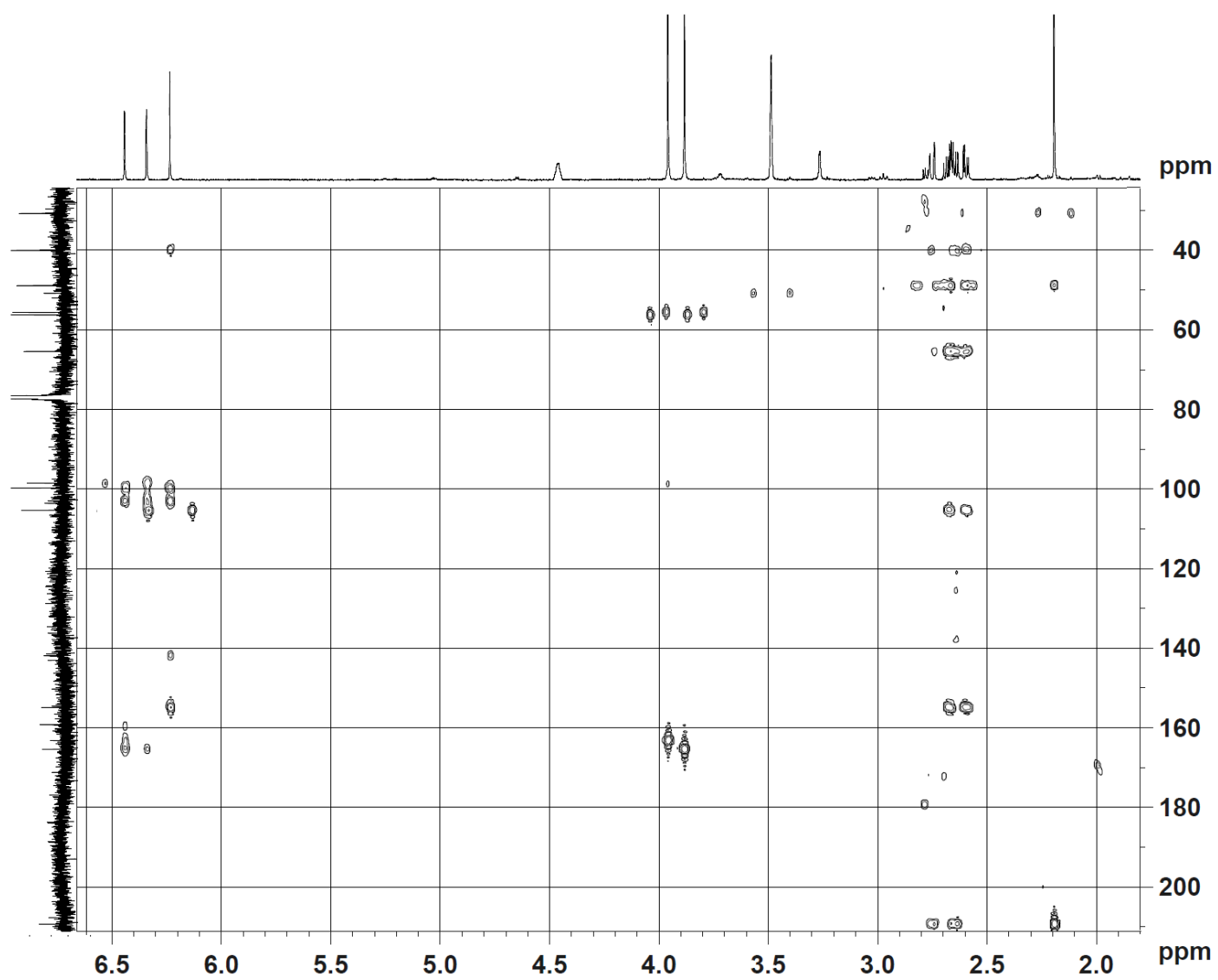


Figure 27. HPLC chromatograms of L-FDLA derivatized standard amino acid and the hydrolysates of compounds **1** and **2**.

