

Supporting Information

Gracilosulfates A-G, Monosulfated Polyoxygenated Steroids from the Marine Sponge *Haliclona gracilis*

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Figure S1. The ^1H NMR (700 MHz, CD_3OD) spectrum of compound **1**

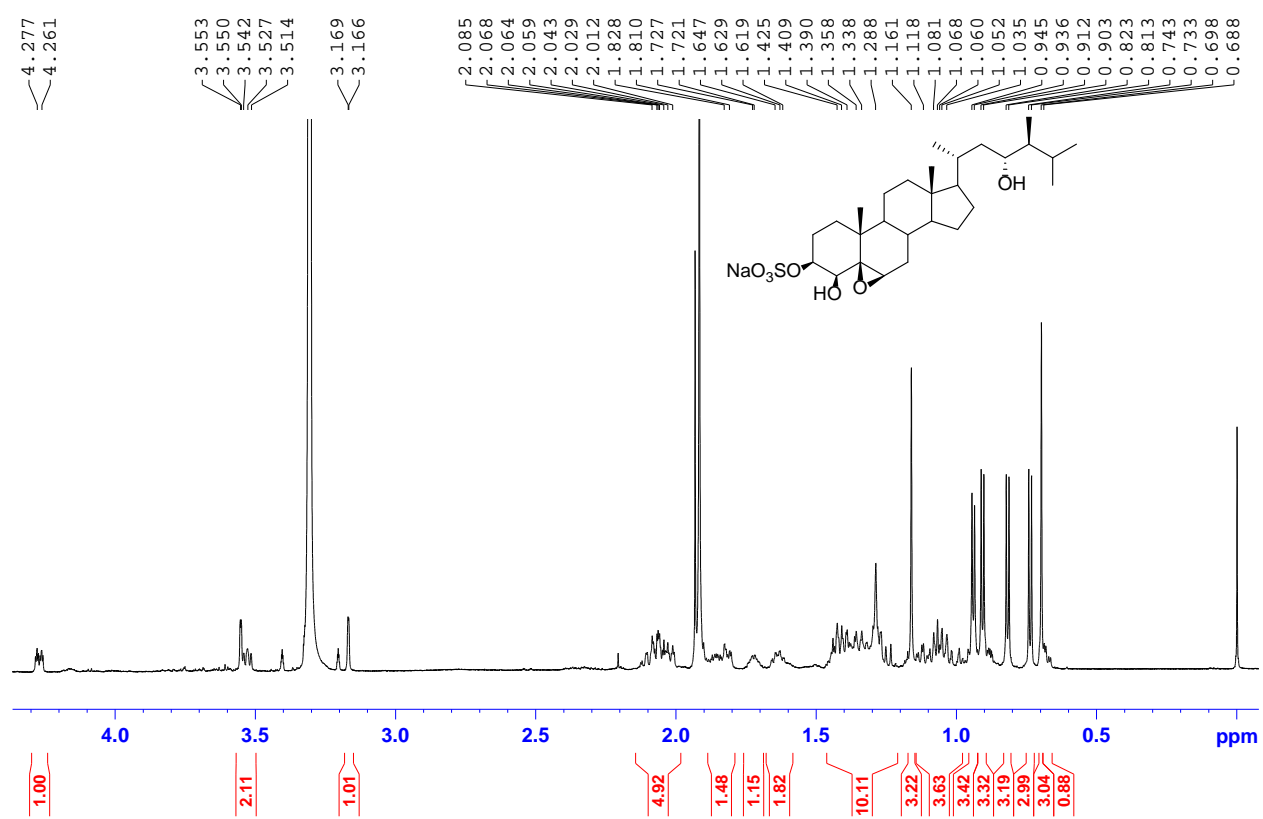


Figure S2. The ^{13}C NMR(125 MHz, CD_3OD) spectrum of compound **1**

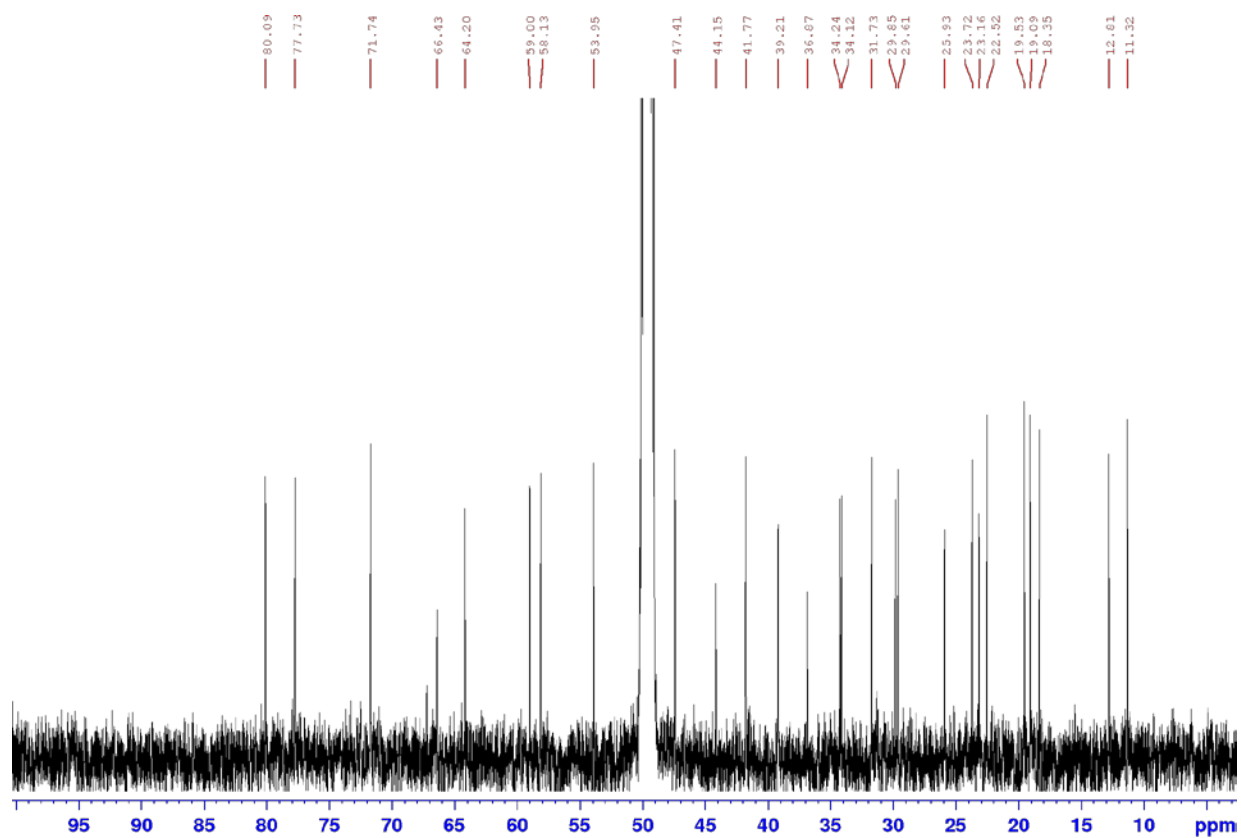


Figure S3. The COSY (700 MHz, CD₃OD) spectrum of compound **1**

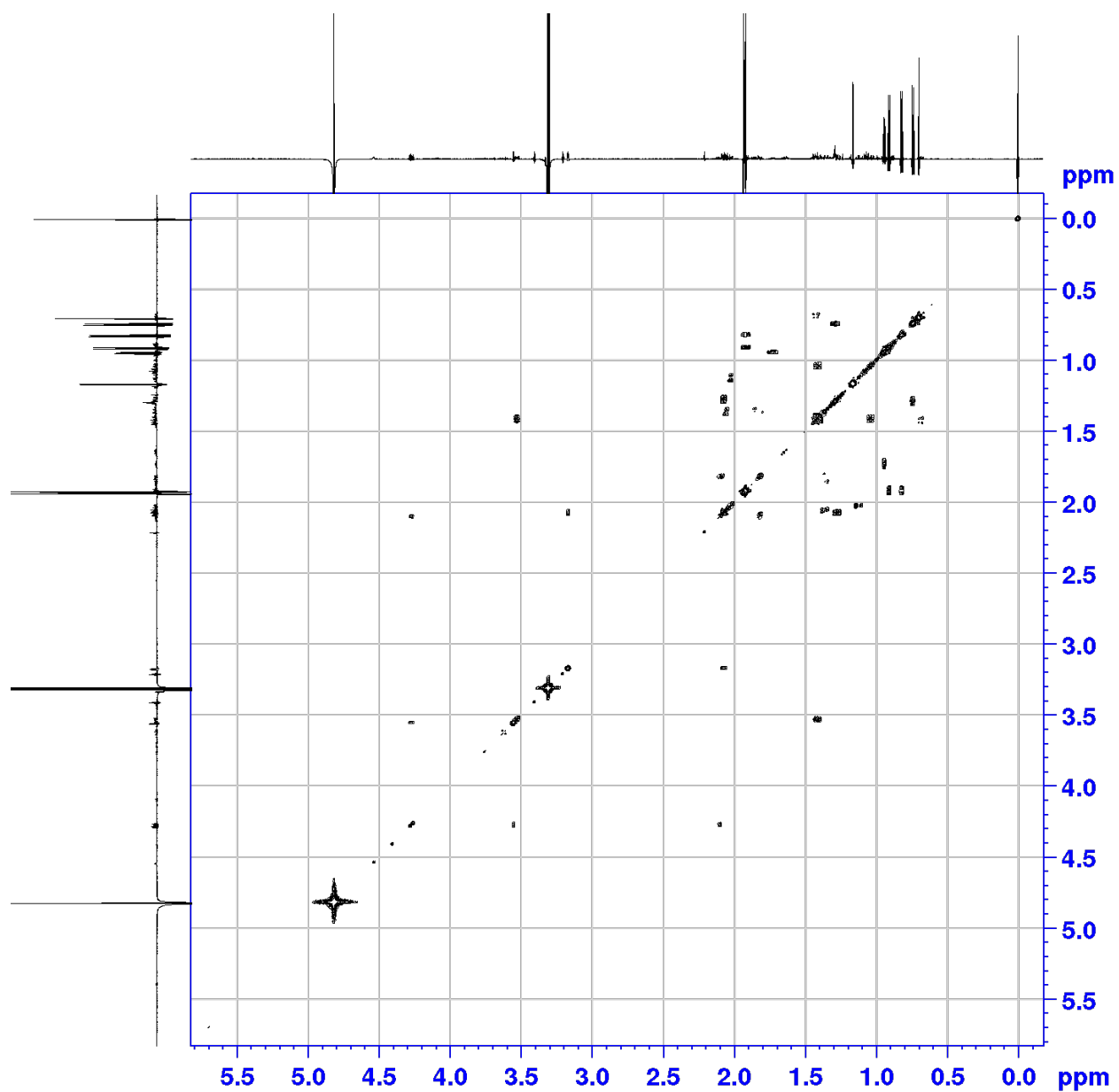


Figure S4. The HSQC (500/125 MHz, CD₃OD) spectrum of compound **1**

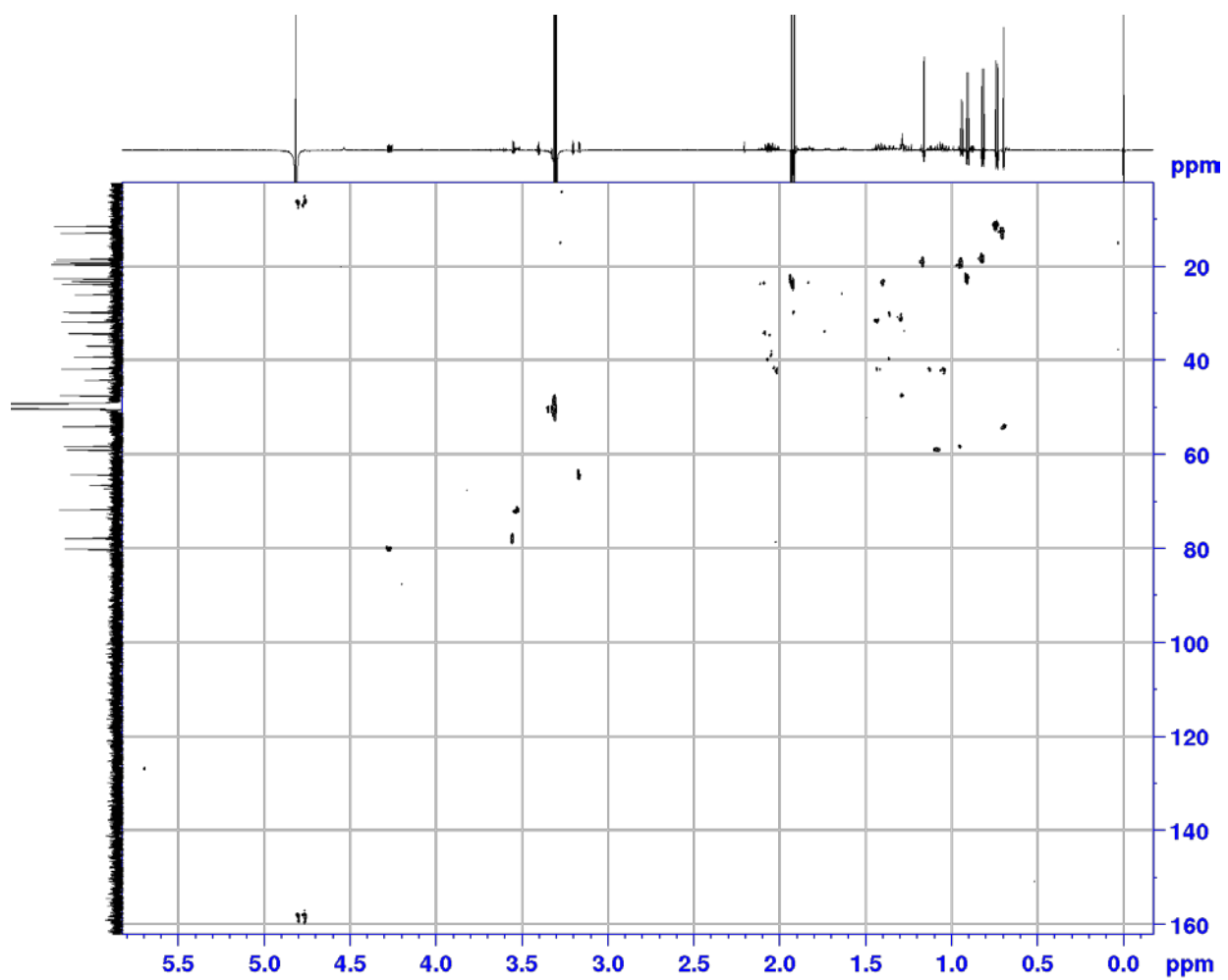


Figure S5. The HMBC (500/125 MHz, DMSO-*d*₆) spectrum of compound **1**

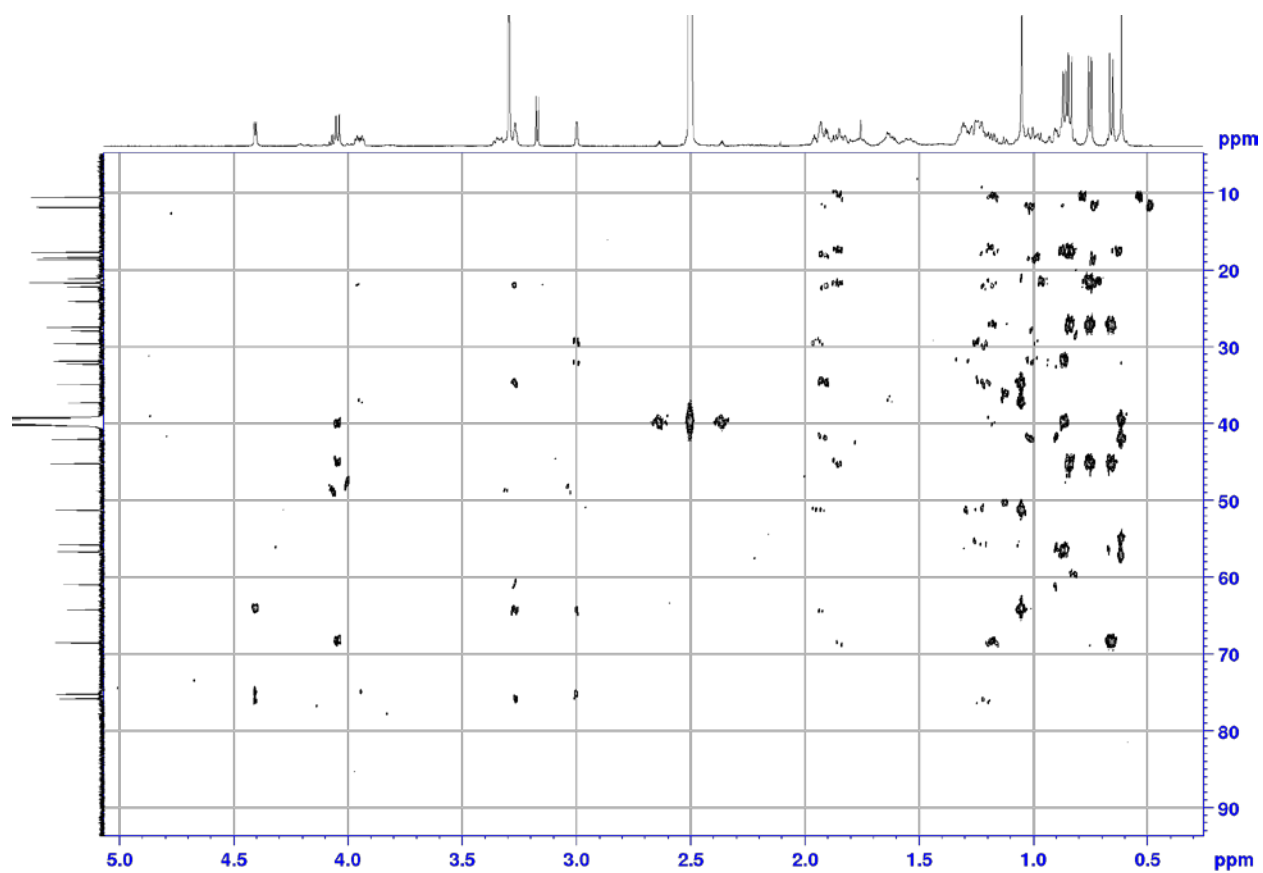


Figure S6. The NOESY (700 MHz, CD₃OD) spectrum of compound **1**

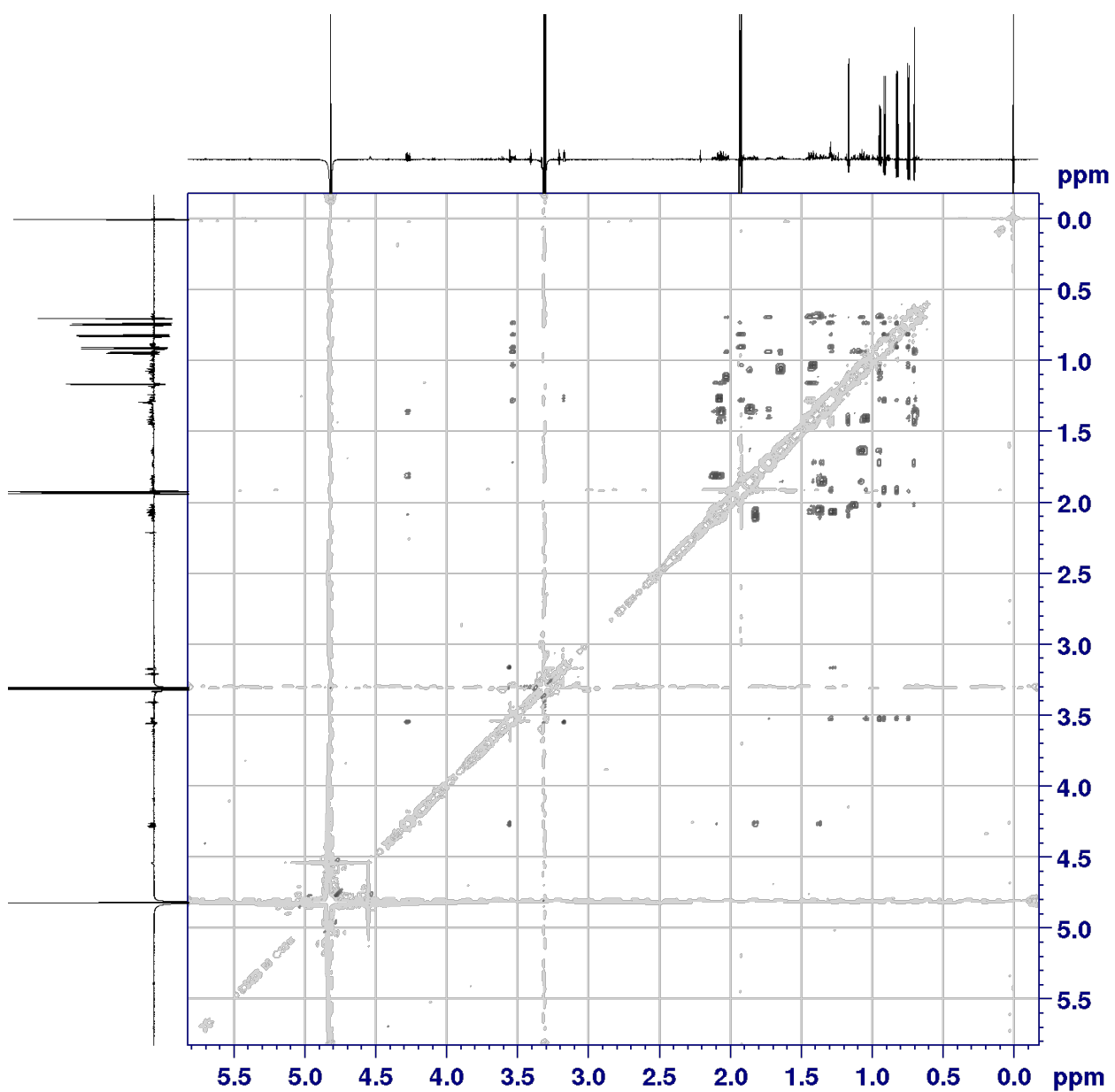


Figure S7. The HRESIMS and MS/MS spectra of compound **1**

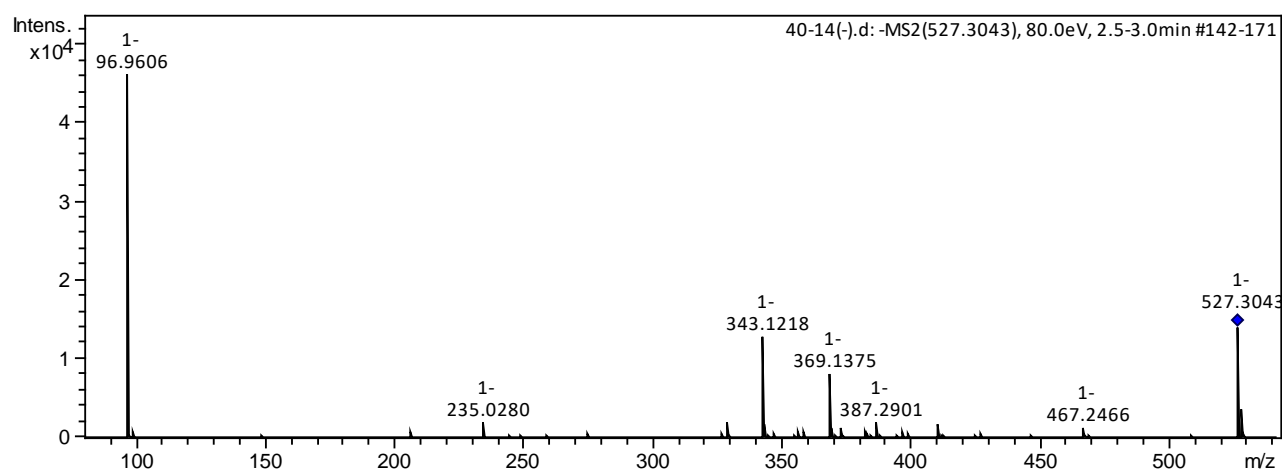
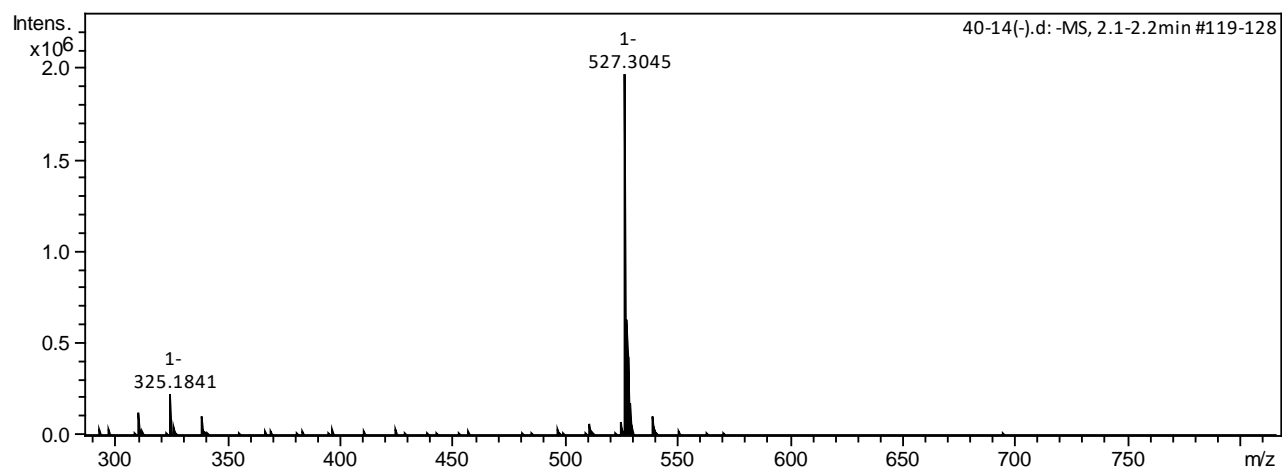


Figure S8. The ^1H NMR (700 MHz, CD_3OD) chemical shift differences between **1S** (red) and **1R** (blue)

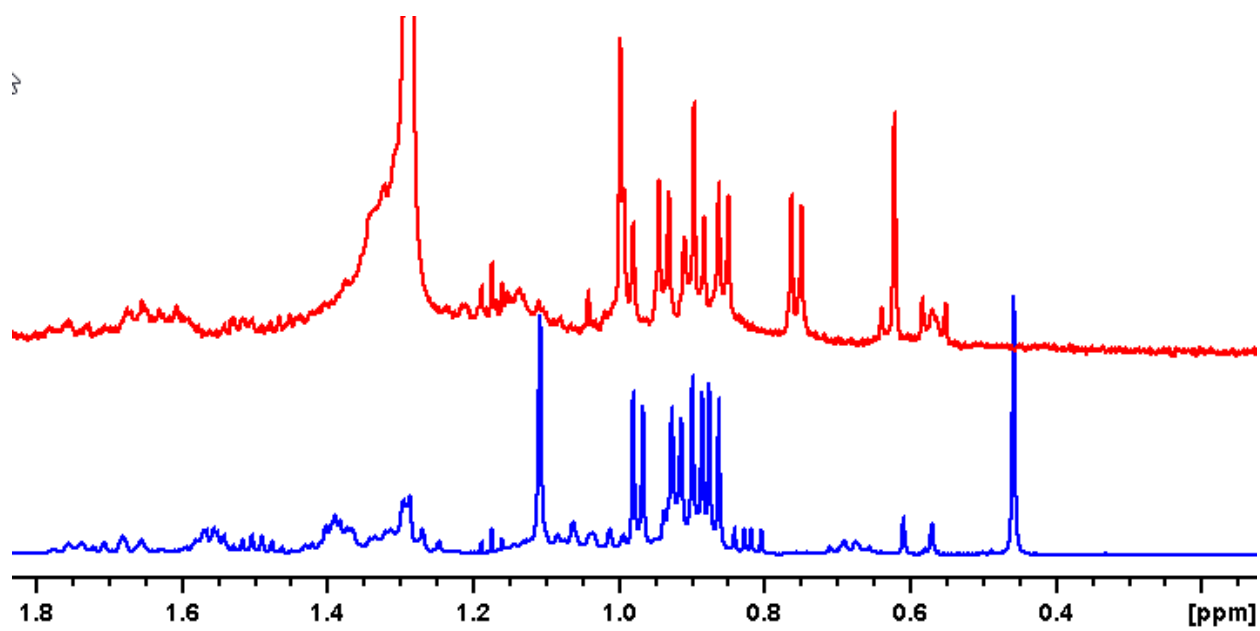


Figure S9. The HRESIMS spectrum of compound **1S (1R)**

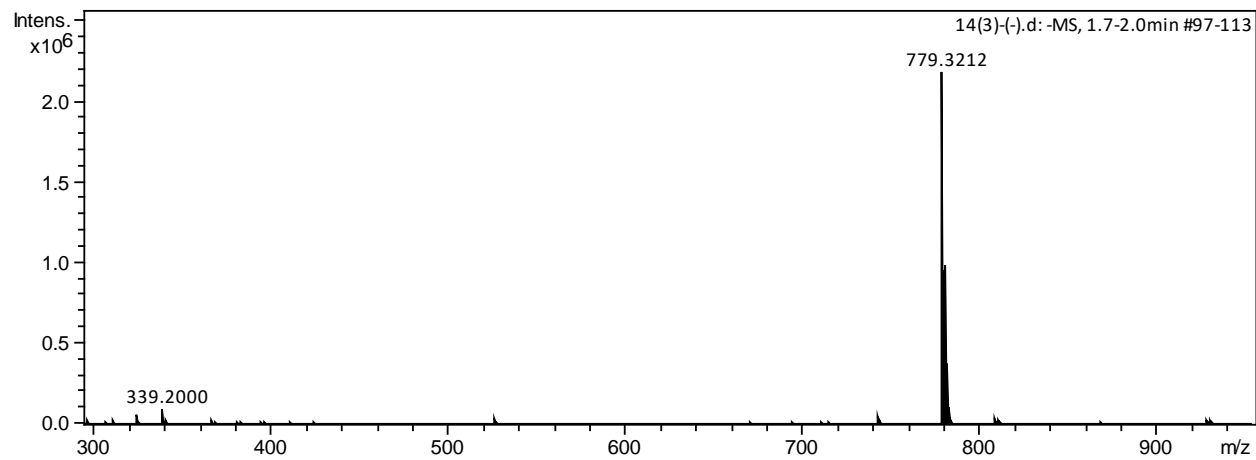


Figure S10. The ^1H NMR (700 MHz, $\text{DMSO-}d_6$) spectrum of compound **1**

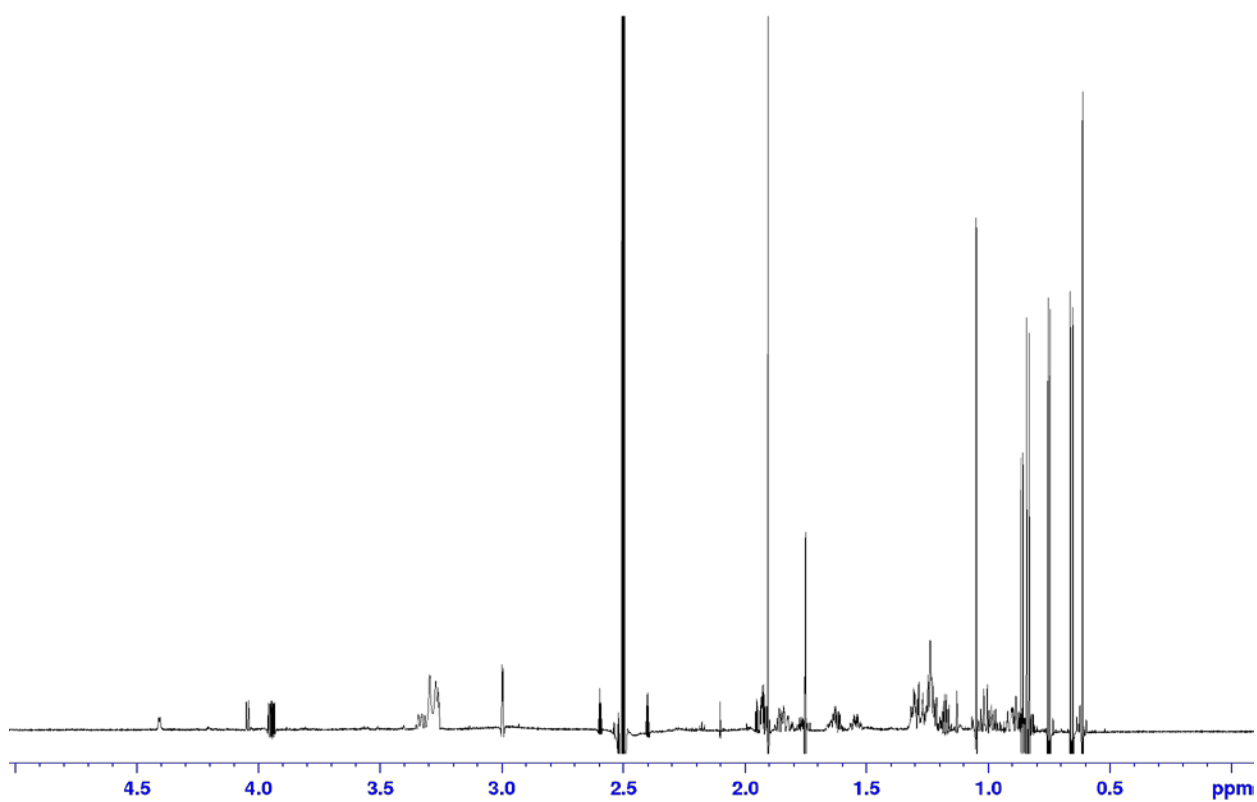


Figure S11. The COSY (700 MHz, DMSO-*d*₆) spectrum of compound **1**

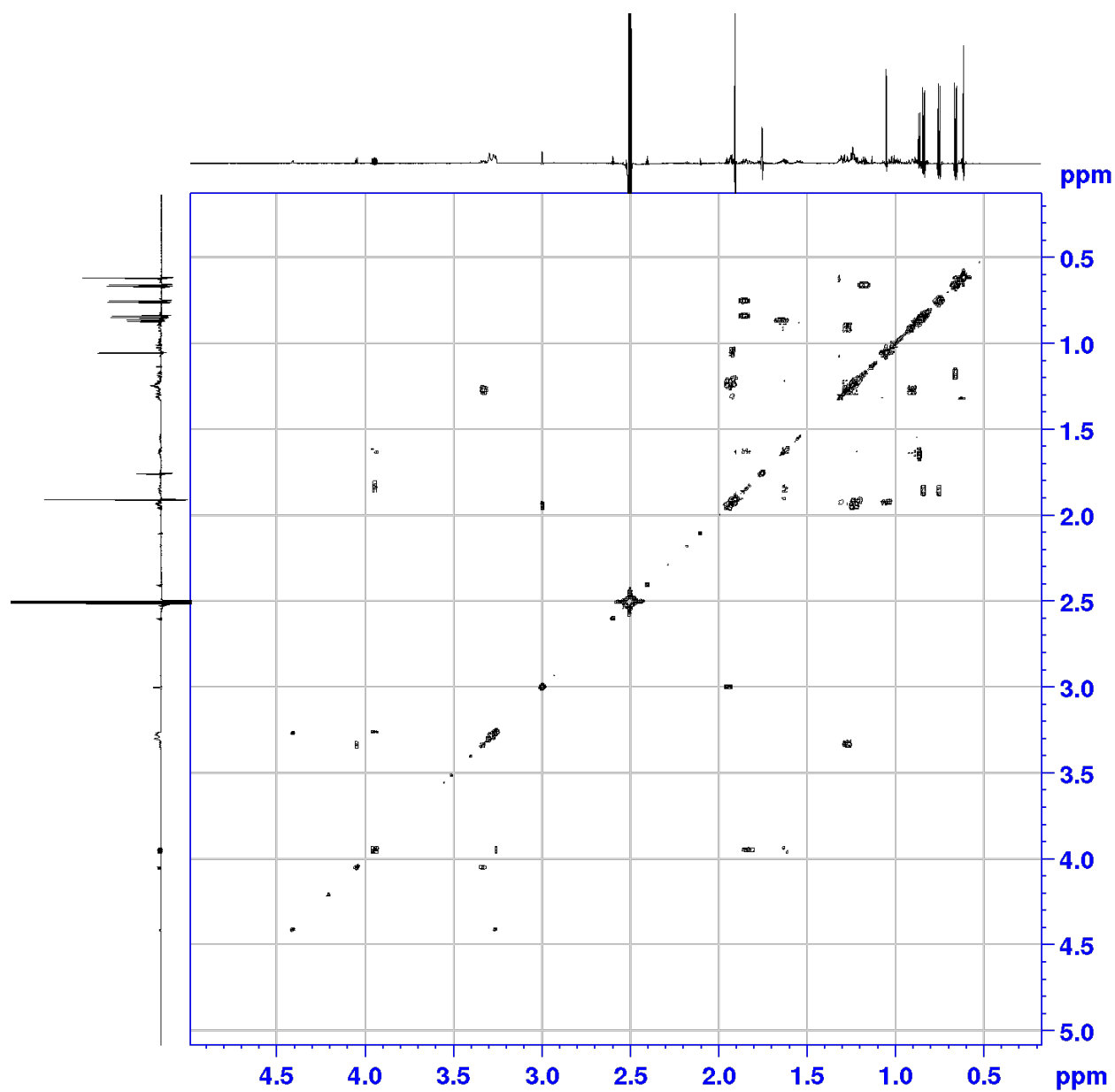


Figure S12. The ^1H NMR (500 MHz, CD_3OD) spectrum of compound **2**

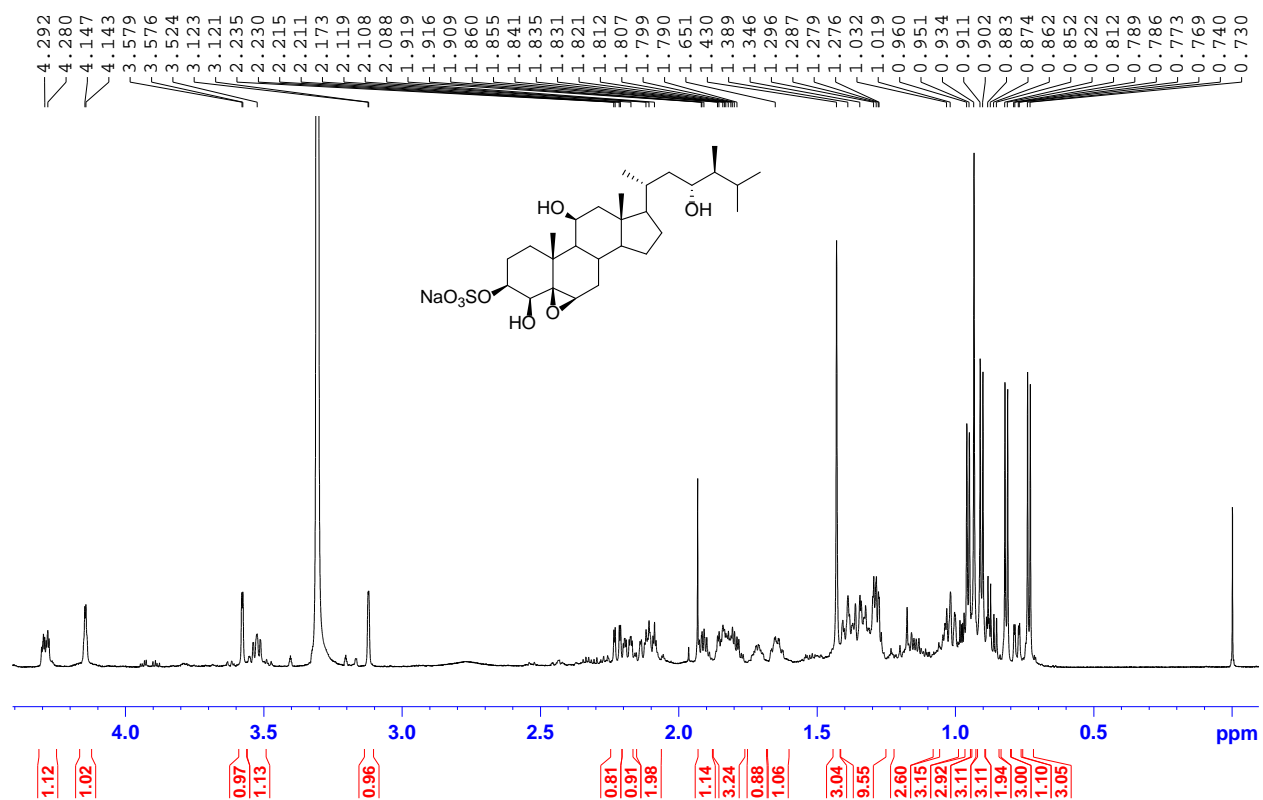


Figure S13. The ^{13}C NMR (125 MHz, CD_3OD) spectrum of compound **2**

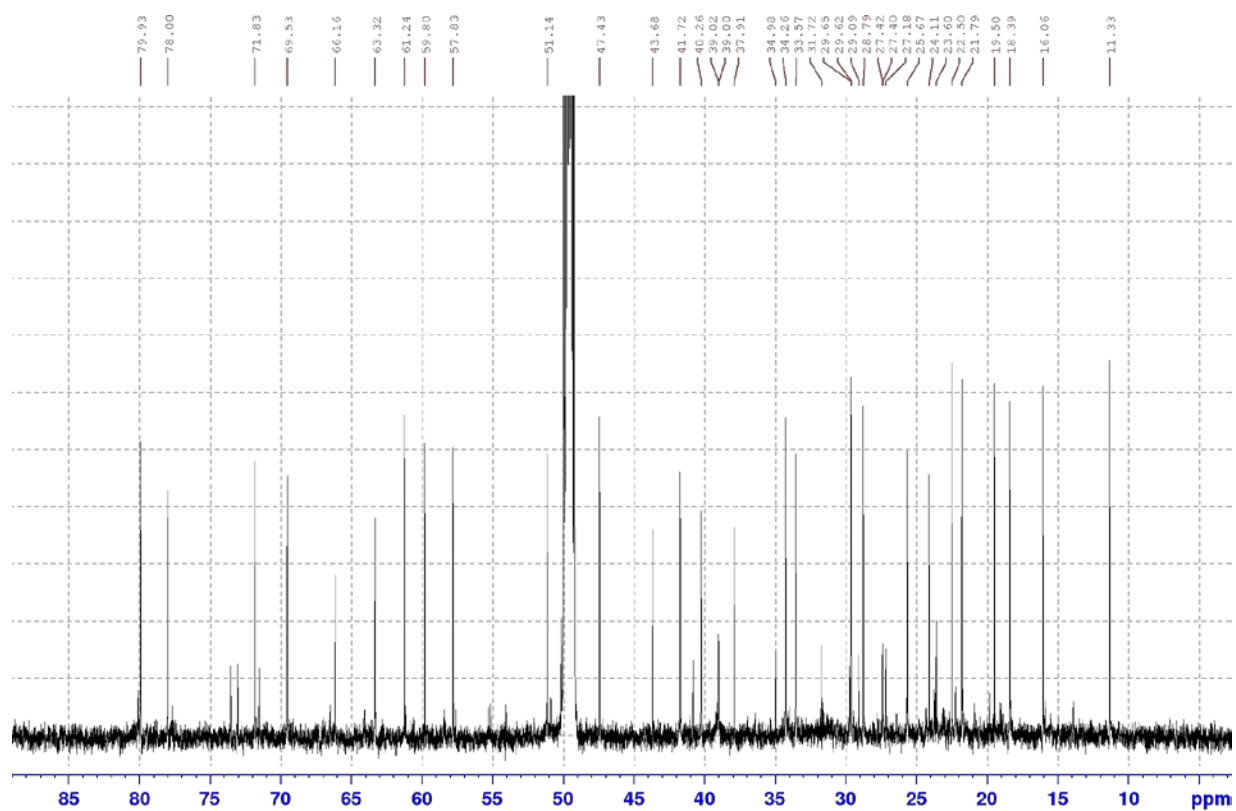


Figure S14. The COSY (500 MHz, CD₃OD) spectrum of compound 2

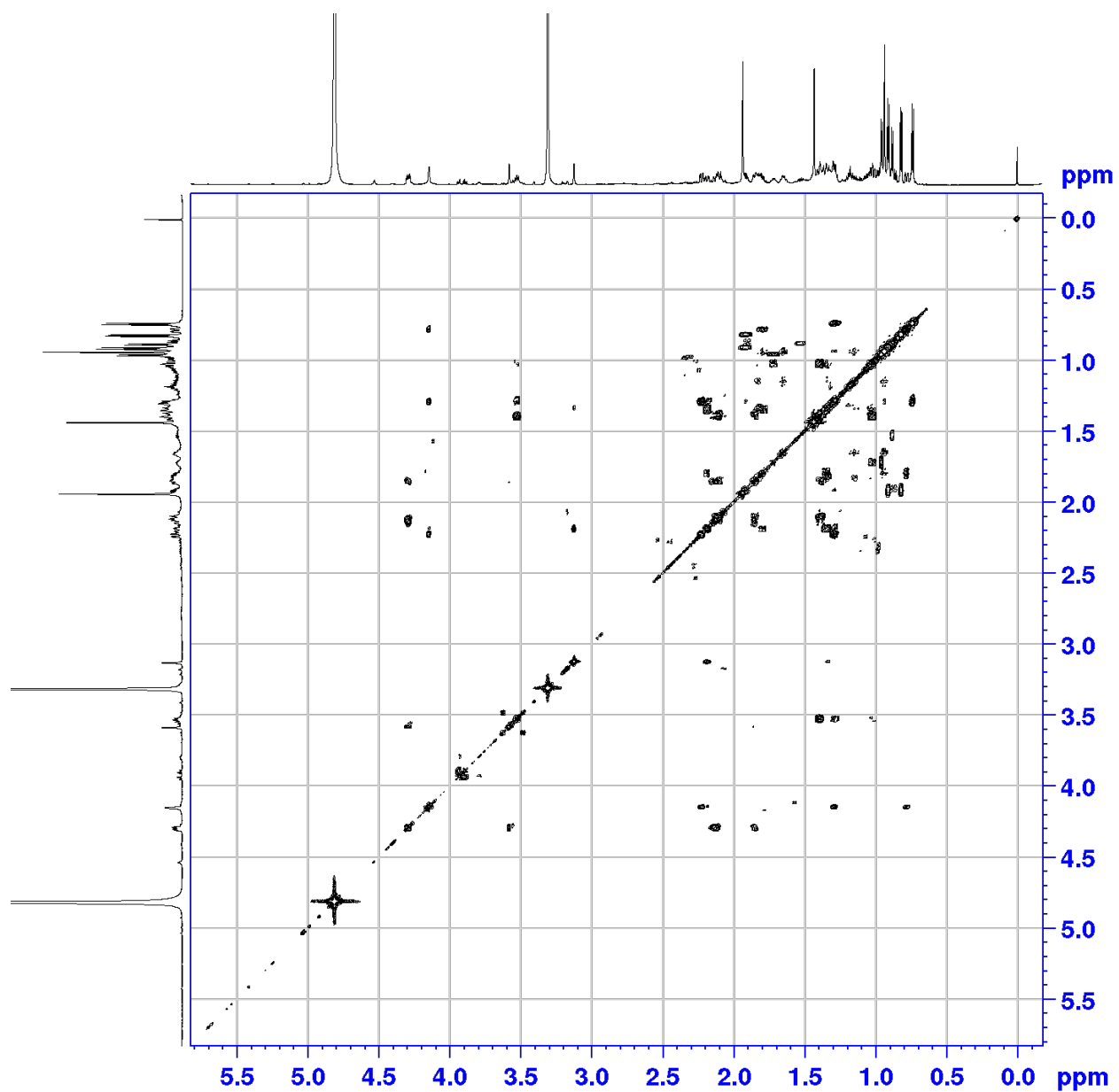


Figure S15. The HSQC (500/125 MHz, CD₃OD) spectrum of compound **2**

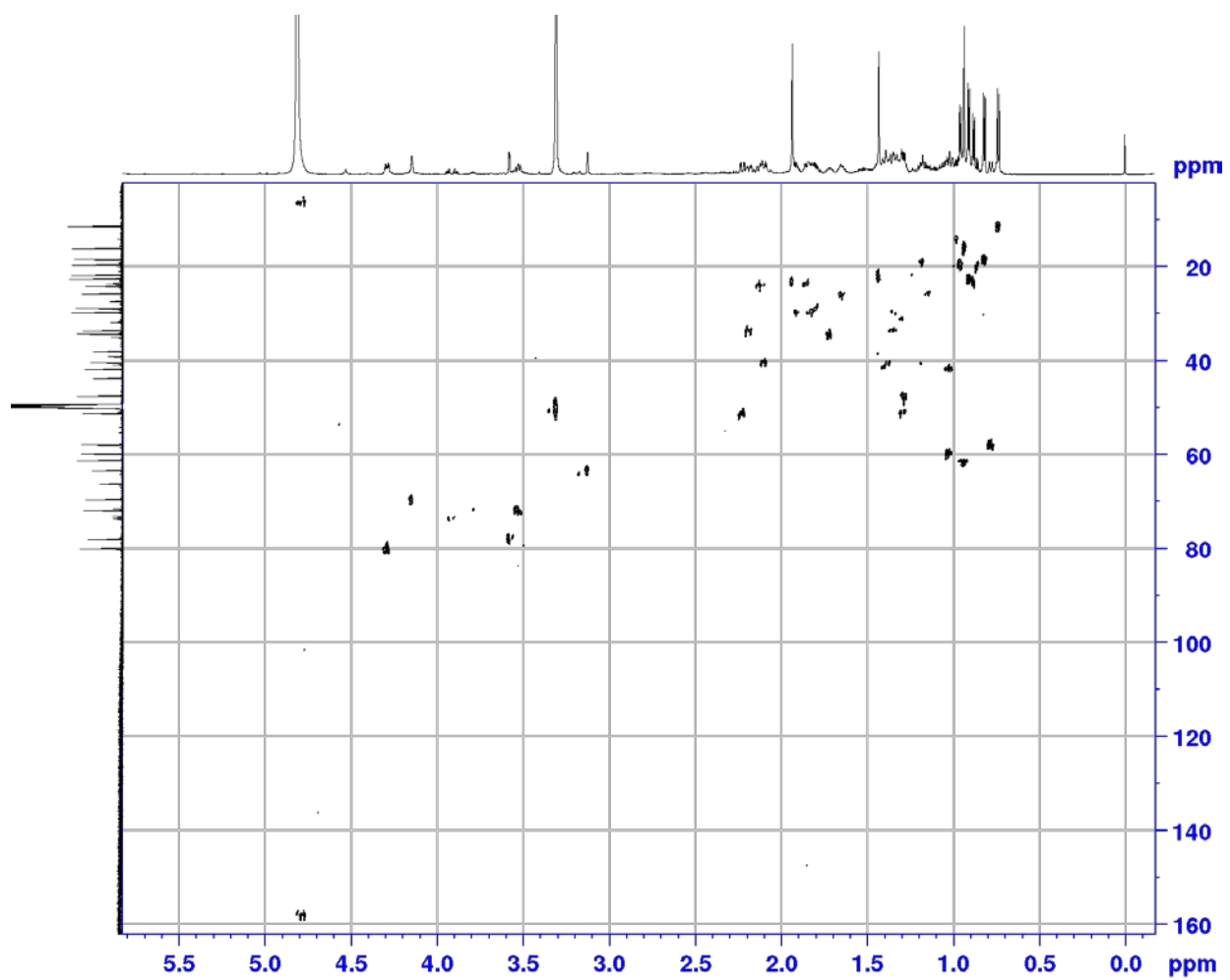


Figure S16. The HMBC (500/125 MHz, CD₃OD) spectrum of compound 2

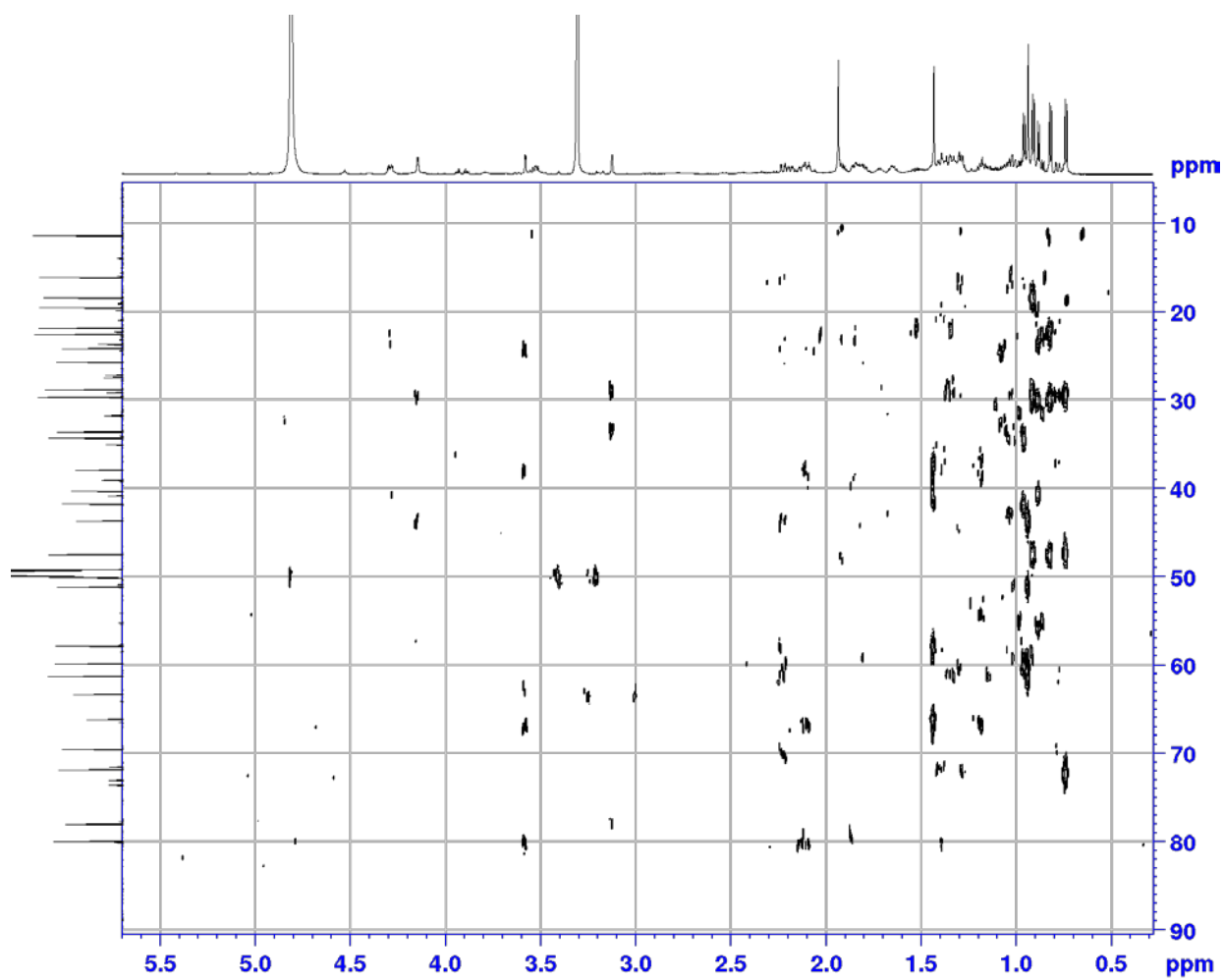


Figure S17. The NOESY (700 MHz, CD₃OD) spectrum of compound 2

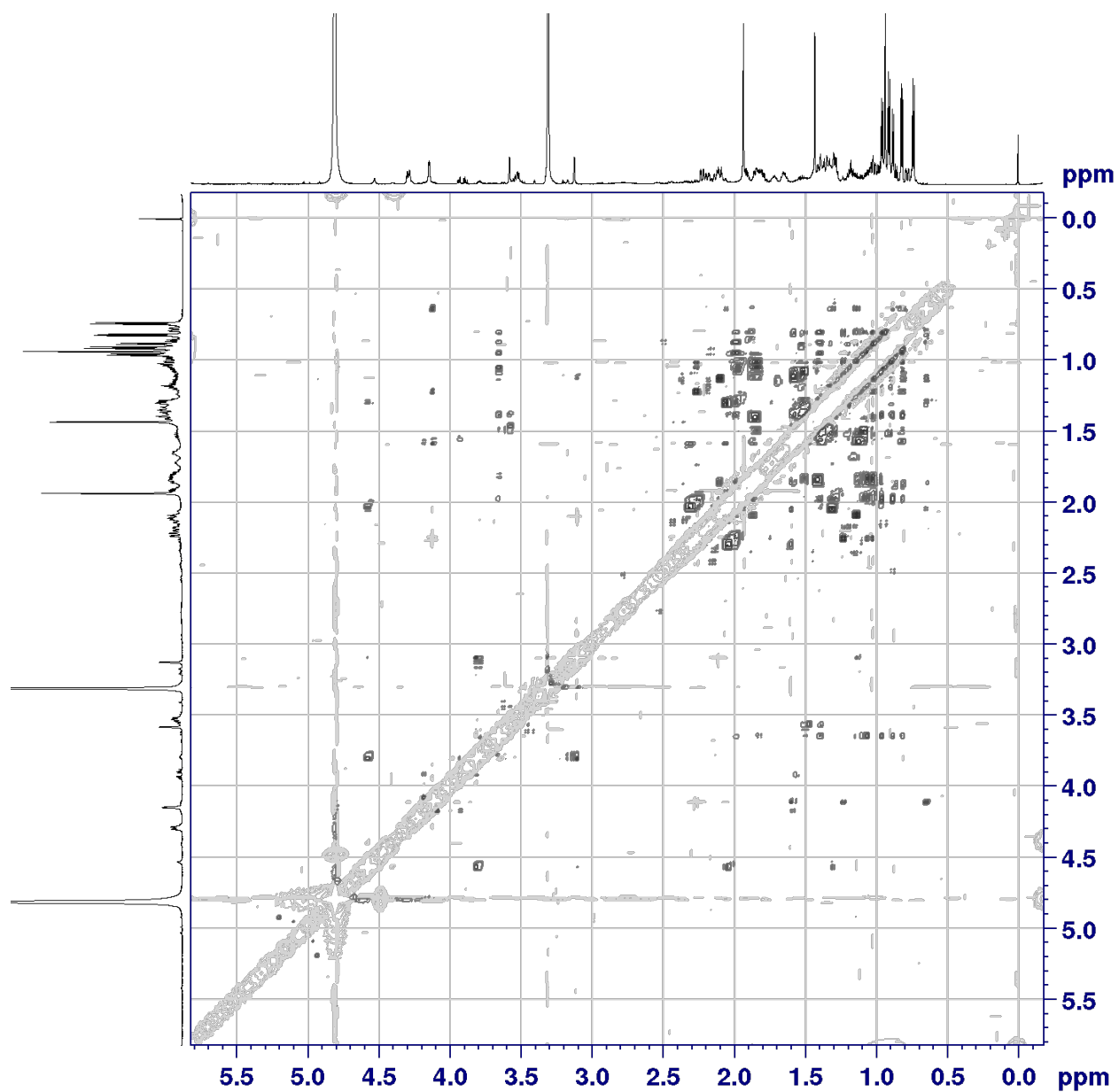


Figure S18. The HRESIMS spectrum of compound **2**

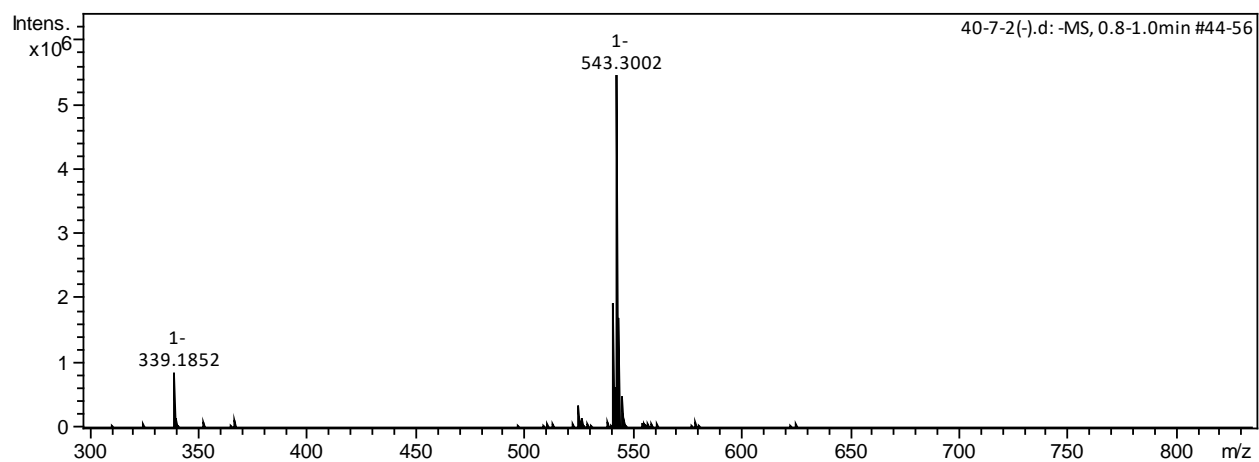


Figure S19. The ^1H (700 MHz, CD_3OD) spectrum of compound **3**

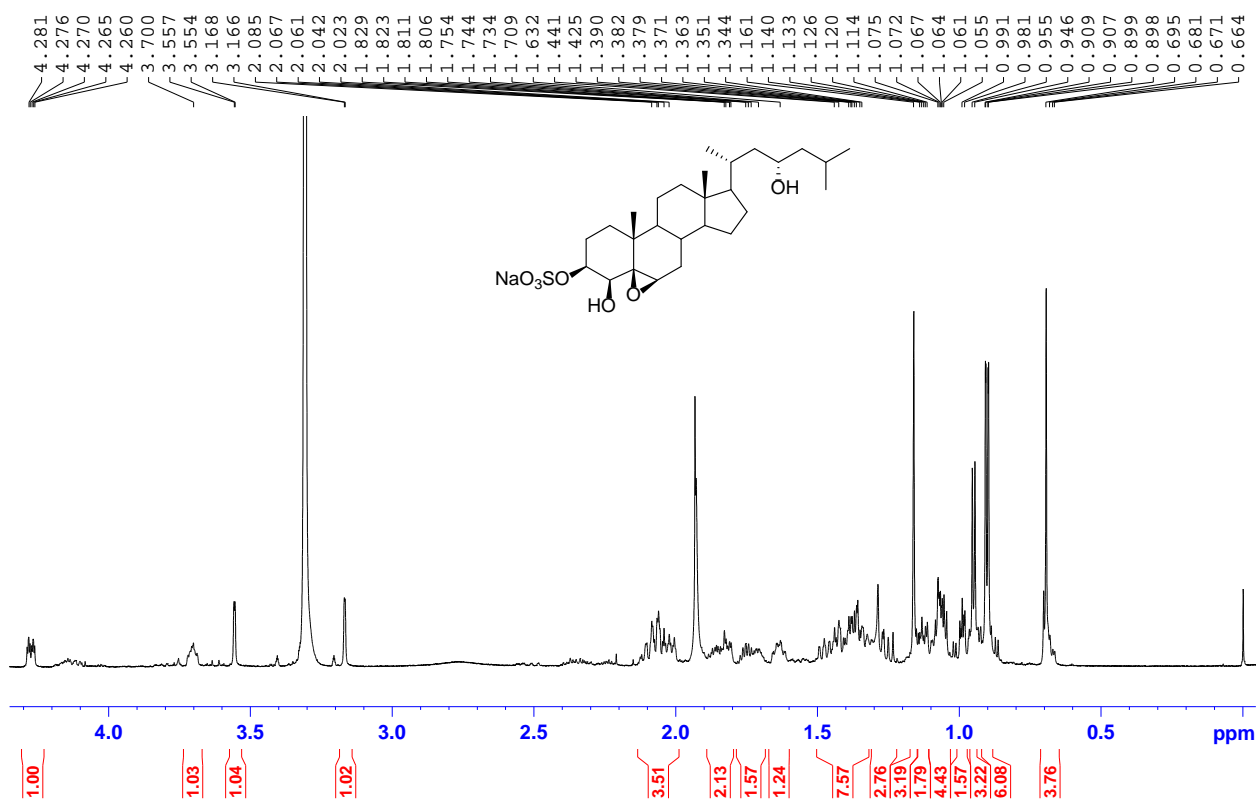


Figure S20. The ^{13}C (175 MHz, CD_3OD) spectrum of compound **3**

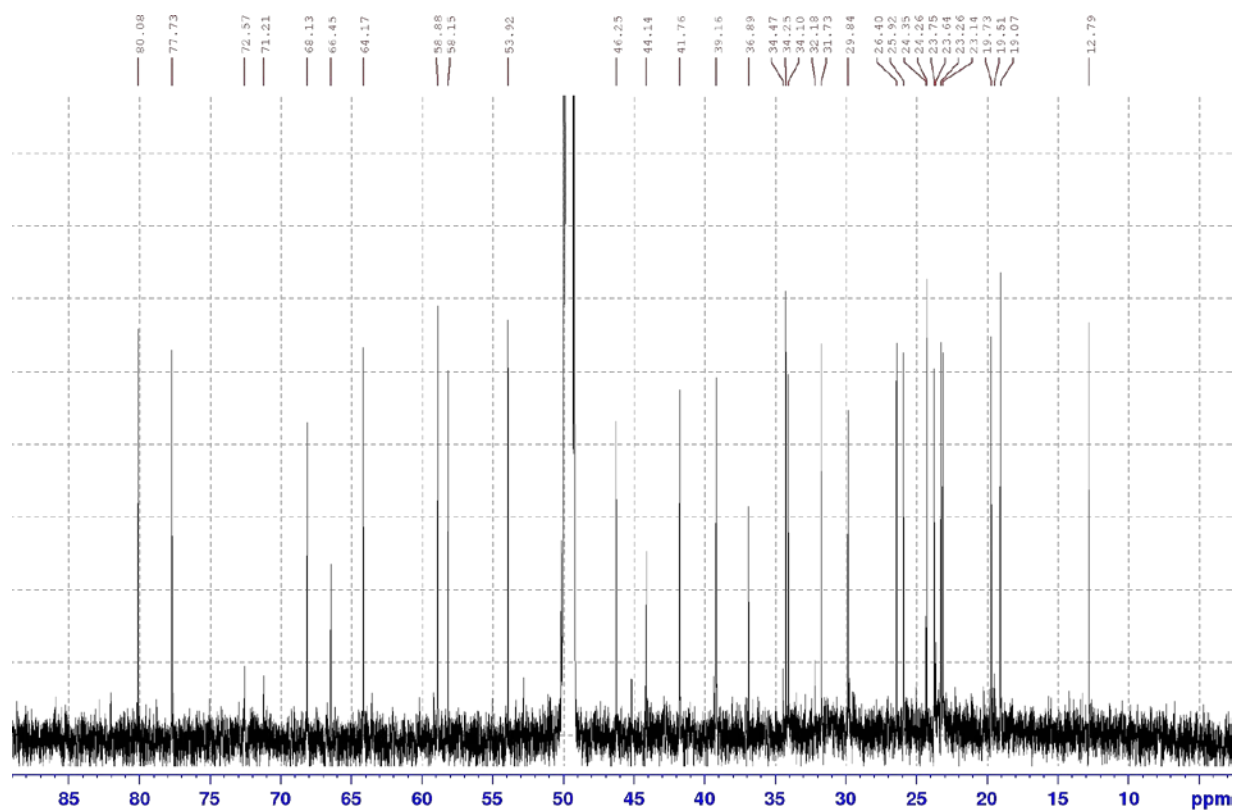


Figure S21. The COSY (700 MHz, CD₃OD) spectrum of compound **3**

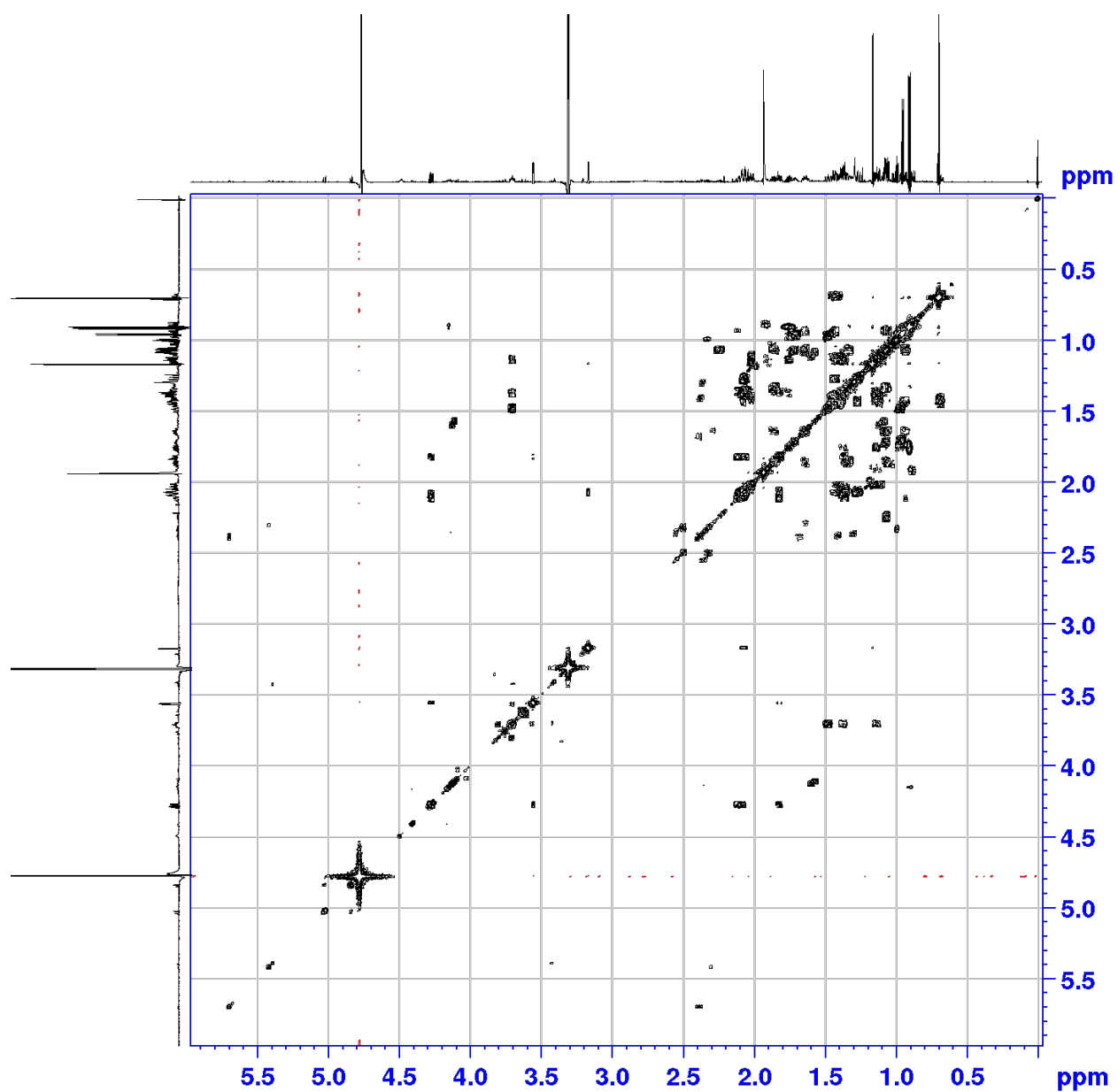


Figure S22. The HSQC (700/175 MHz, CD₃OD) spectrum of compound **3**

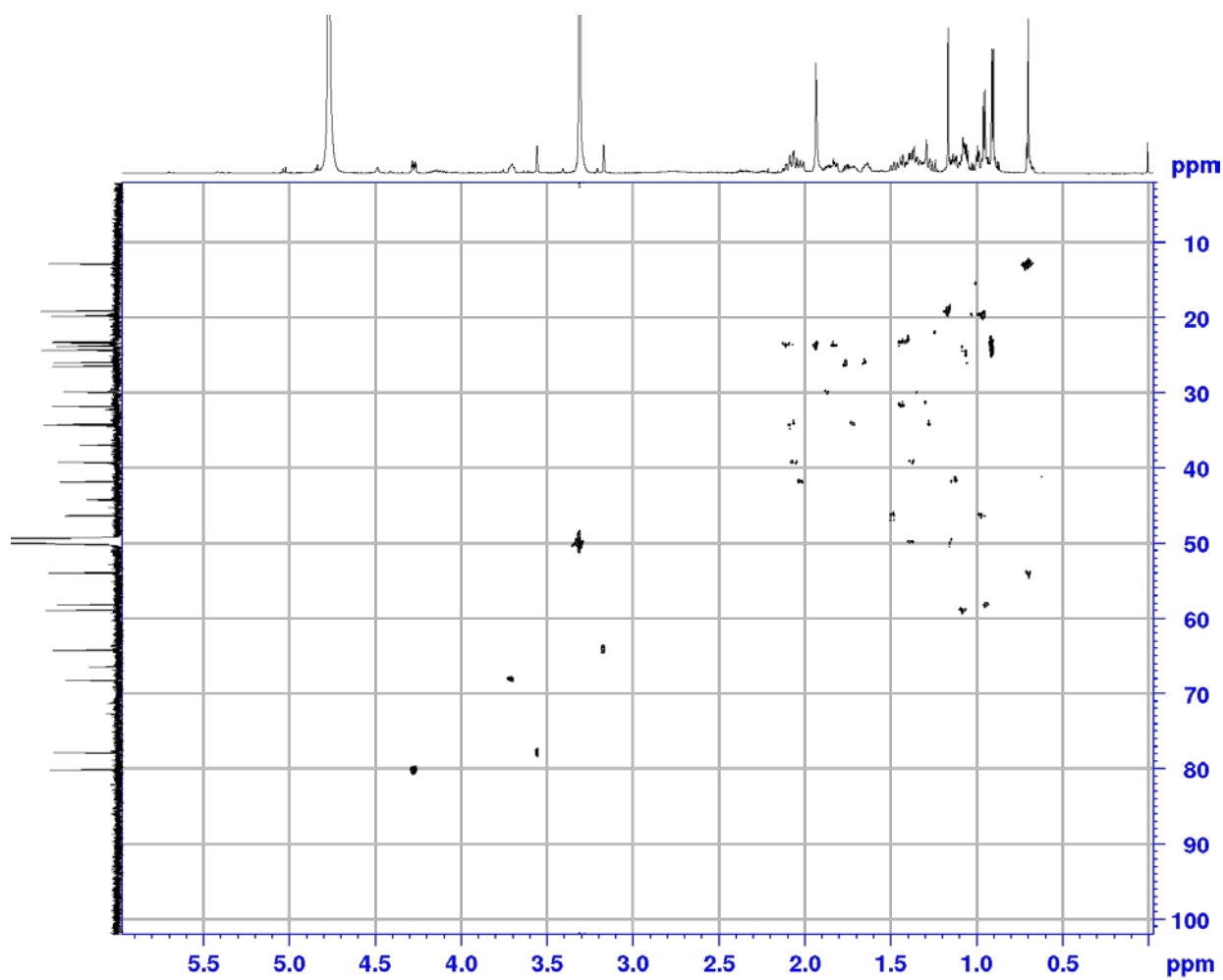


Figure S23. The HMBC (700/175 MHz, CD₃OD) spectrum of compound **3**

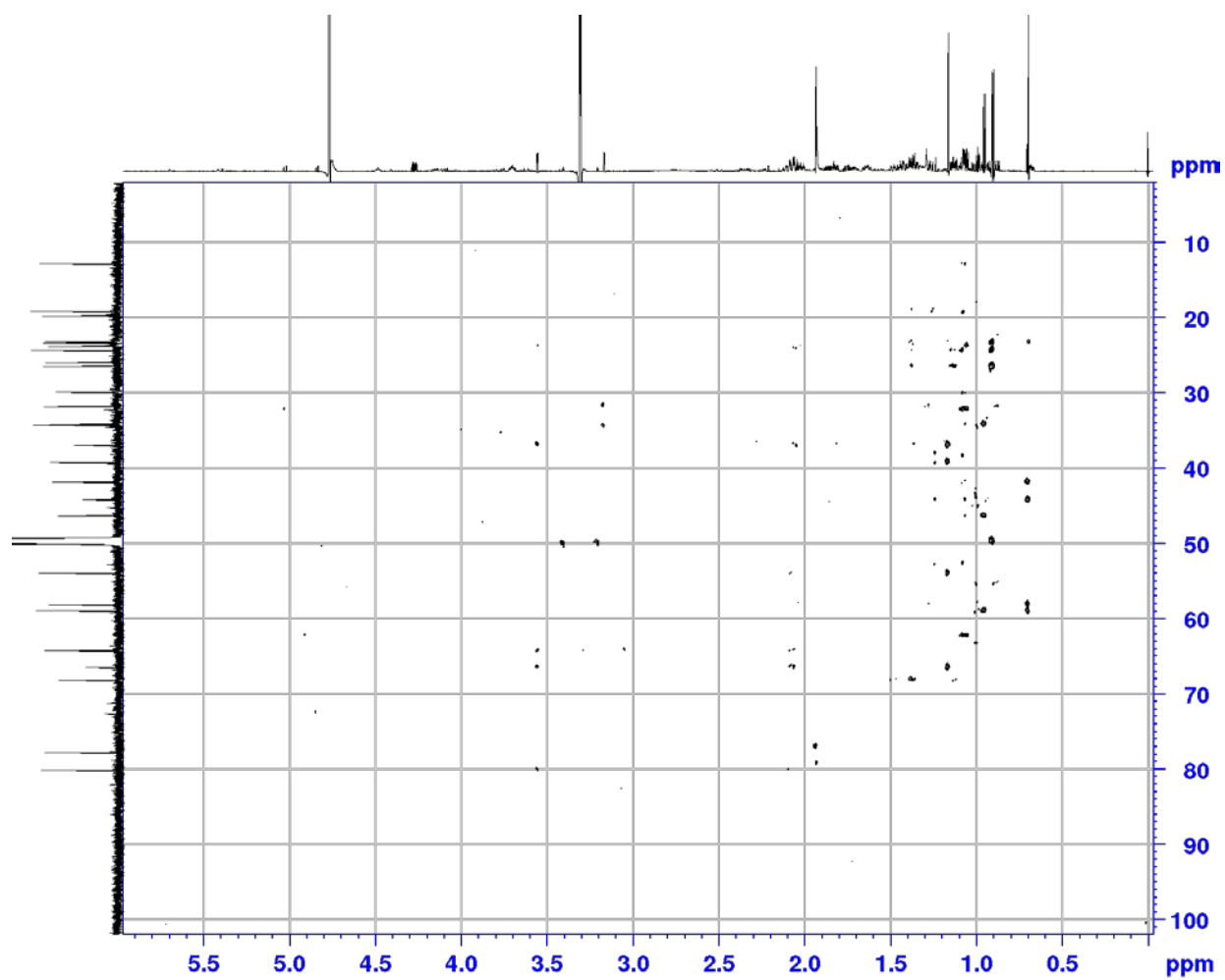


Figure S24. The NOESY (700 MHz, CD₃OD) spectrum of compound **3**

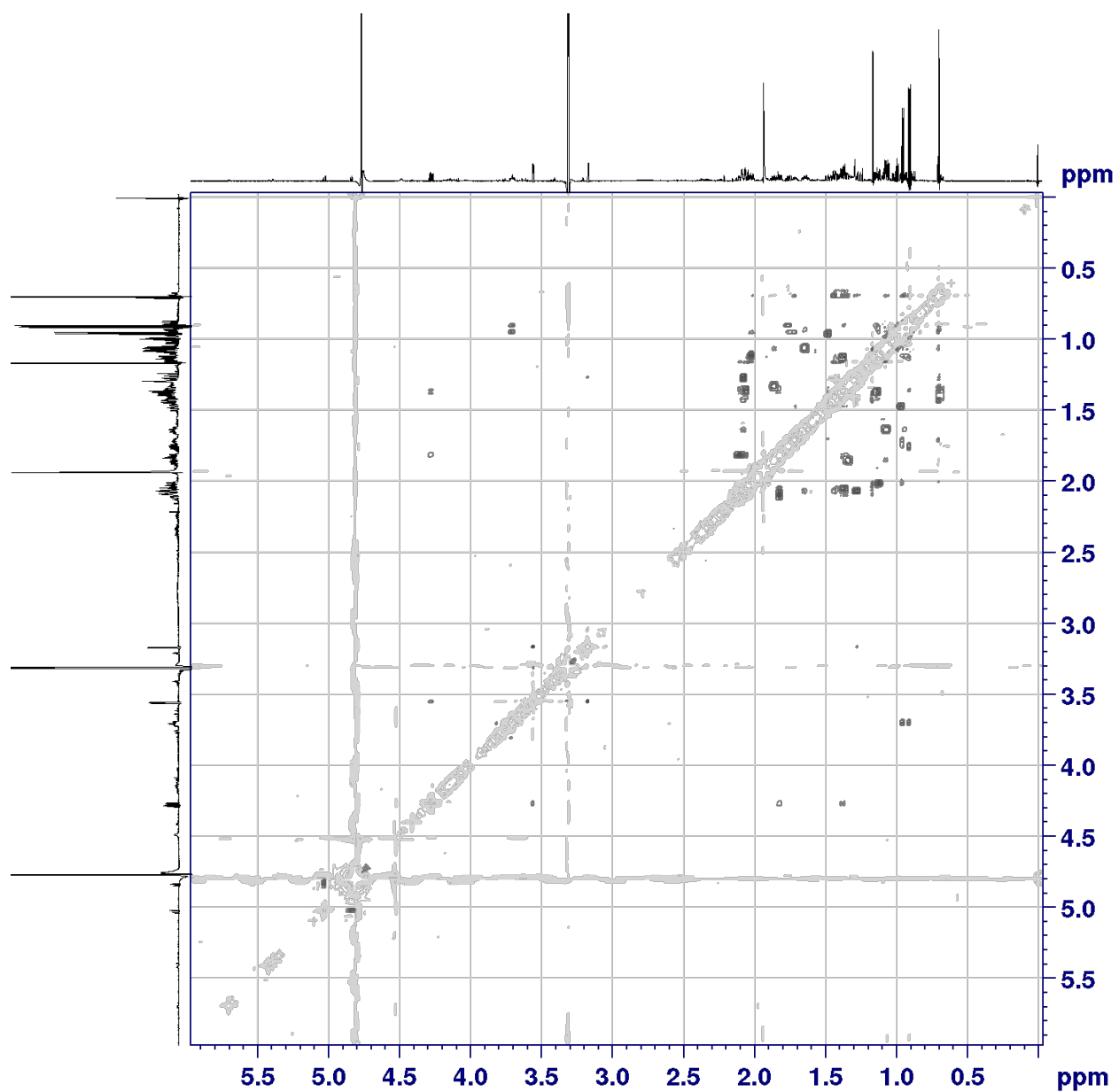


Figure S25. The HRESIMS spectrum of compound **3**

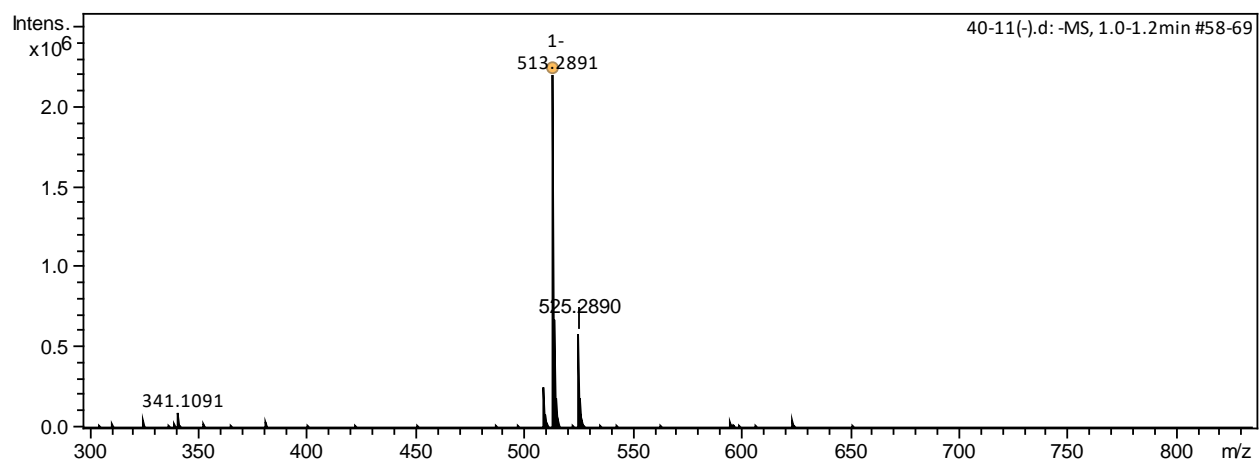


Figure S26. ^1H NMR chemical shift differences between **3S** (red) and **3R** (blue)

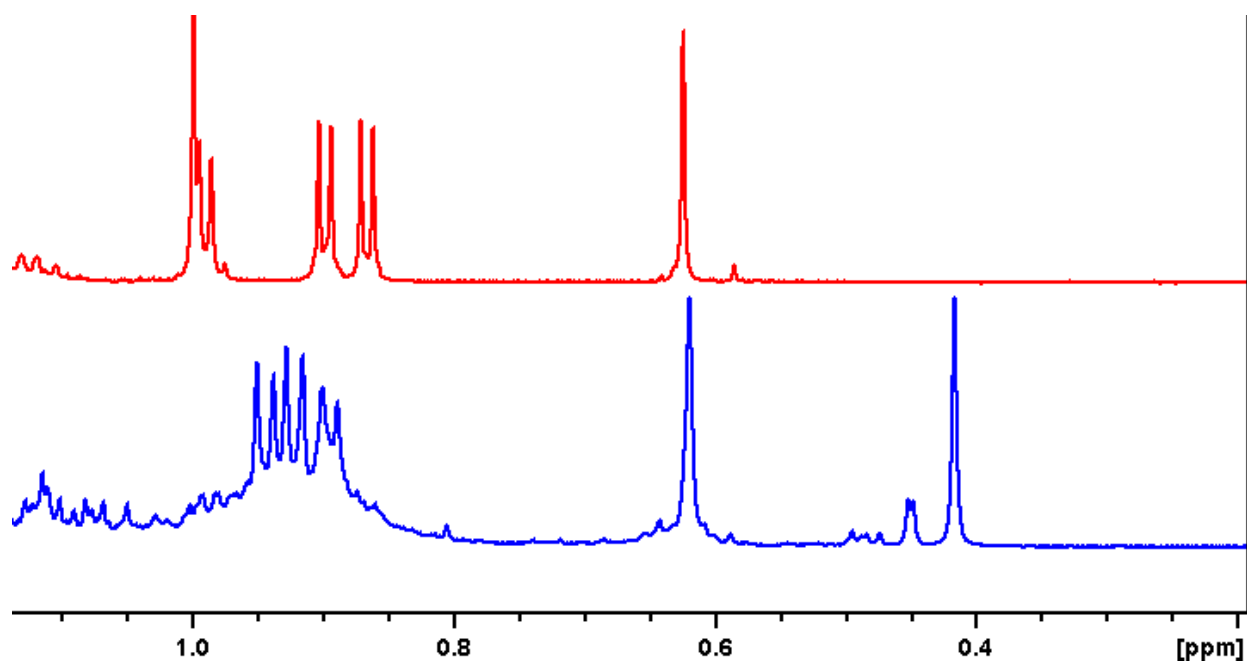


Figure S27. The HRESIMS spectrum of compound **3R (3S)**

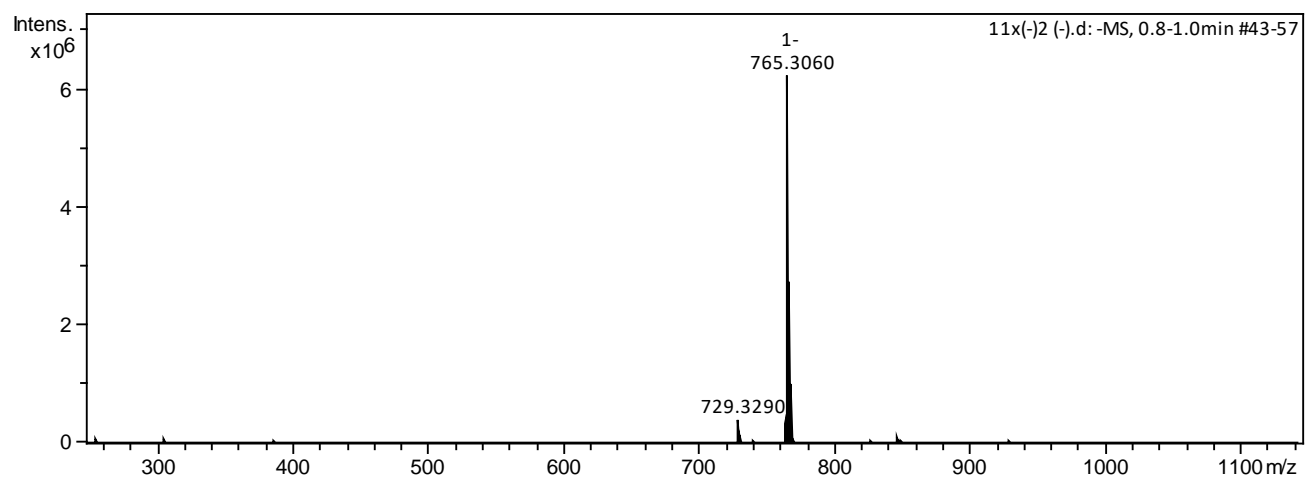


Figure S28. The ^1H (700 MHz, CD_3OD) spectrum of compound 4

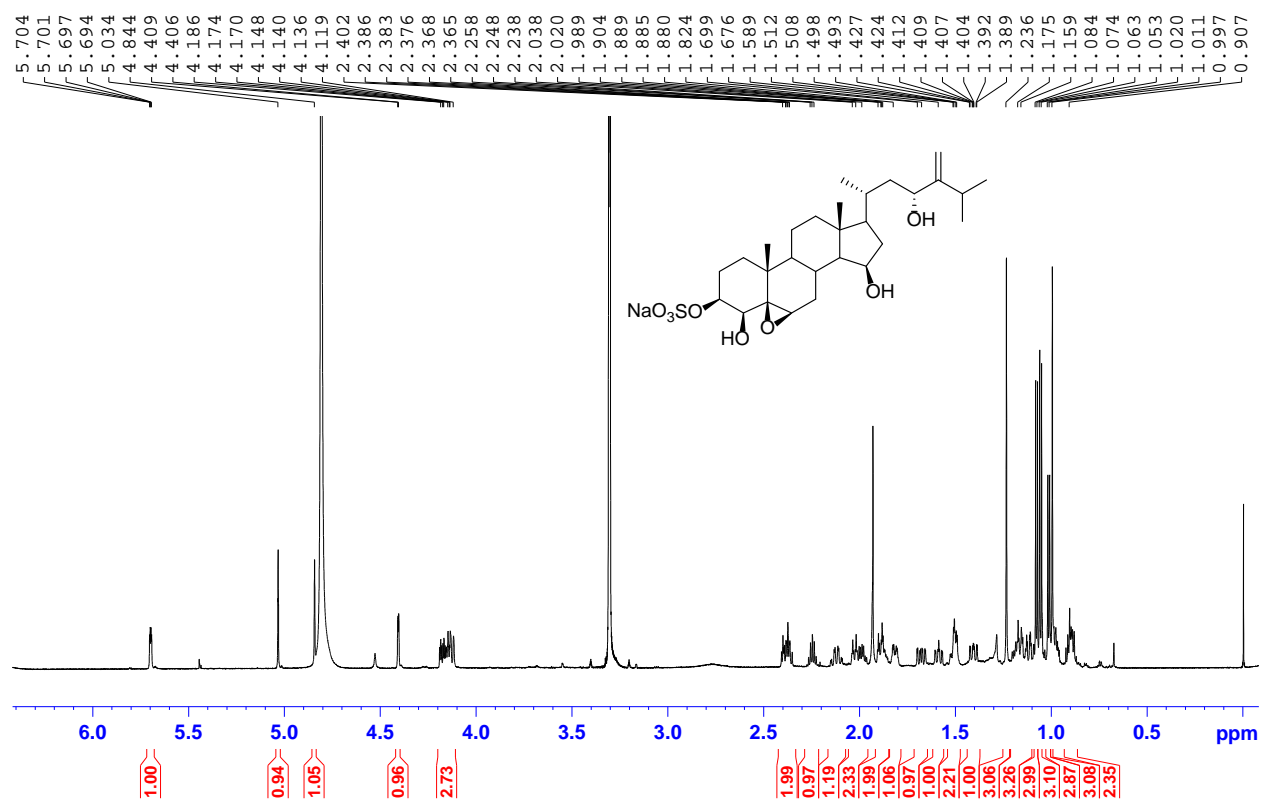


Figure S29. The ^{13}C (175 MHz, CD_3OD) spectrum of compound **4**

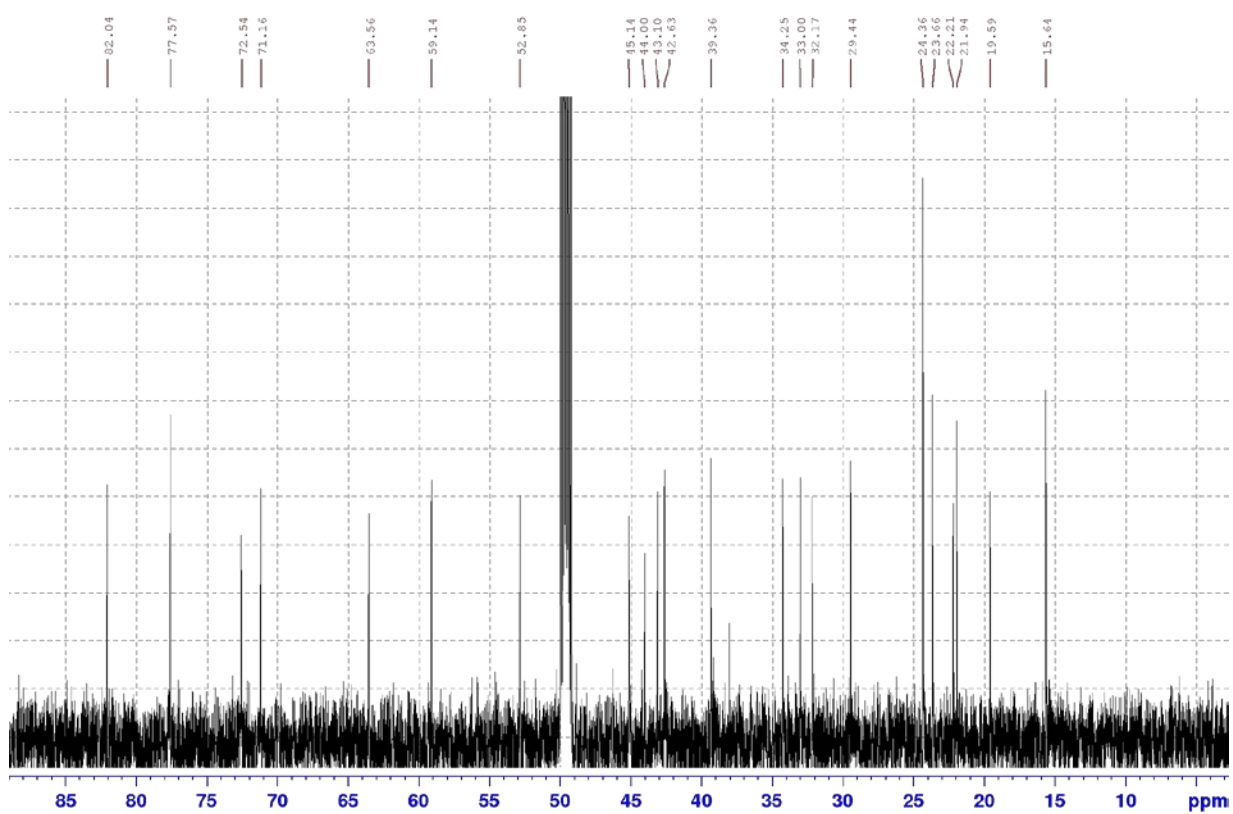


Figure S30. The COSY (700 MHz, CD₃OD) spectrum of compound **4**

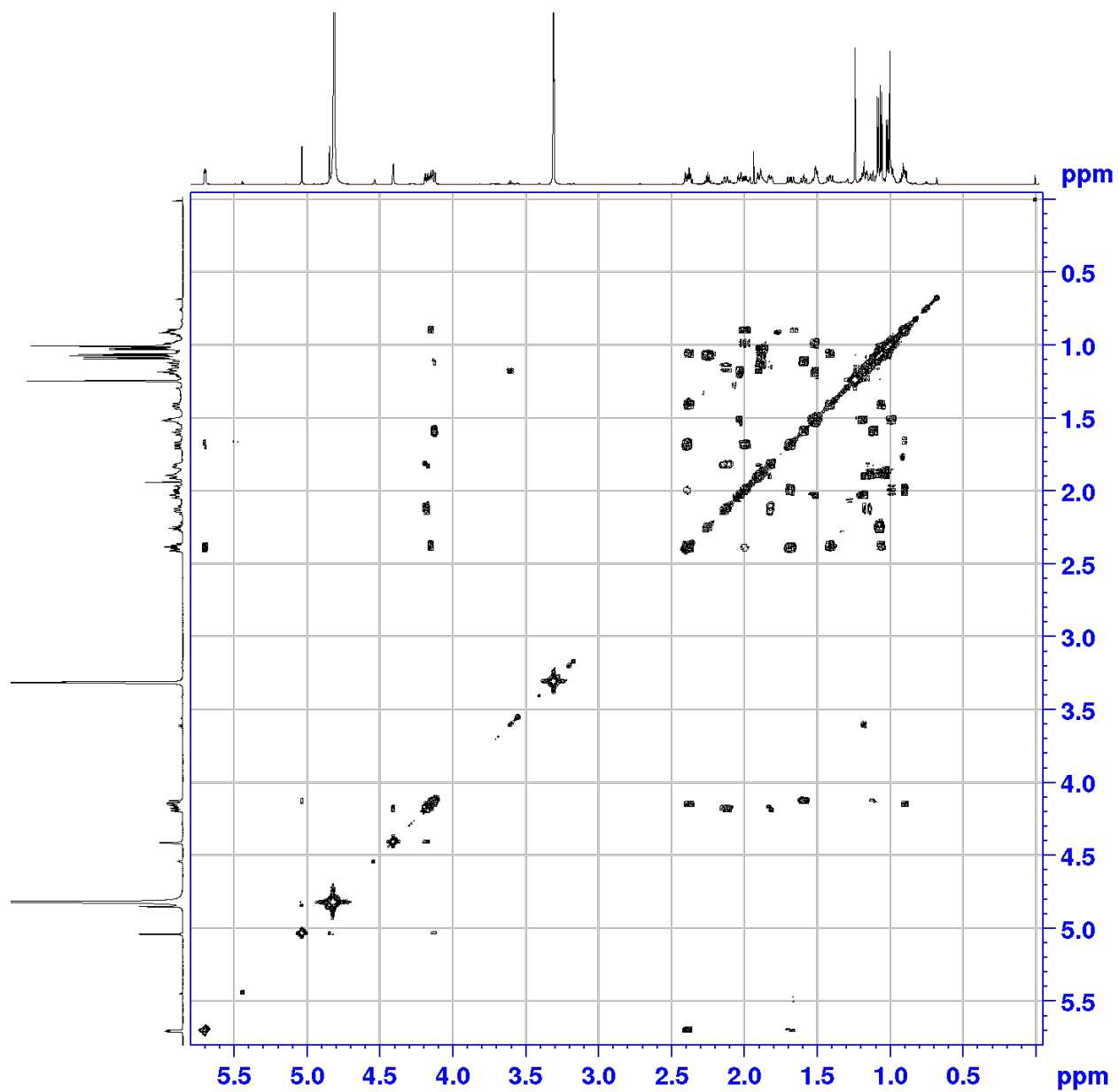


Figure S31. The HSQC (700/175 MHz, CD₃OD) spectrum of compound 4

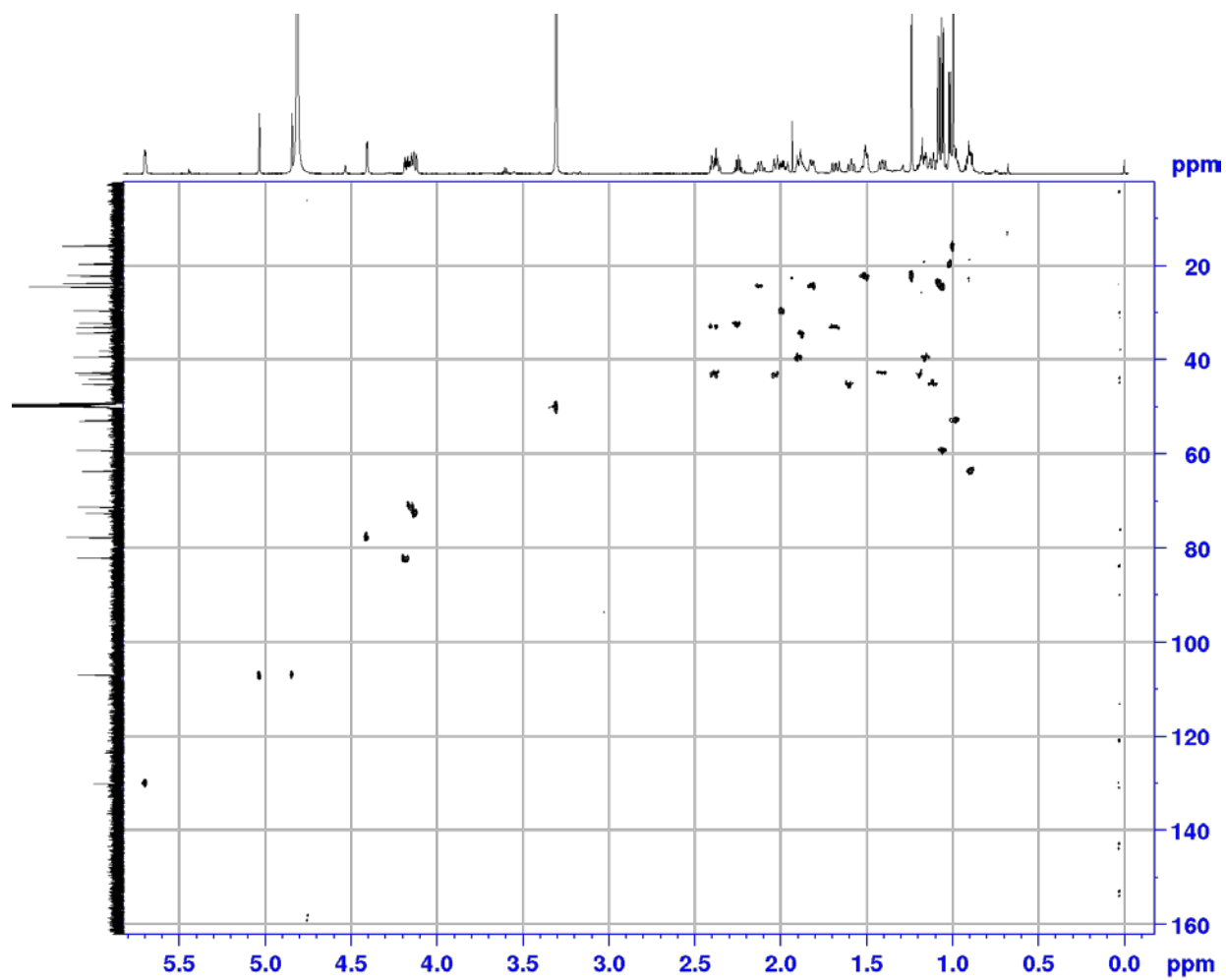


Figure S32. The HMBC (700/175 MHz, CD₃OD) spectrum of compound **4**

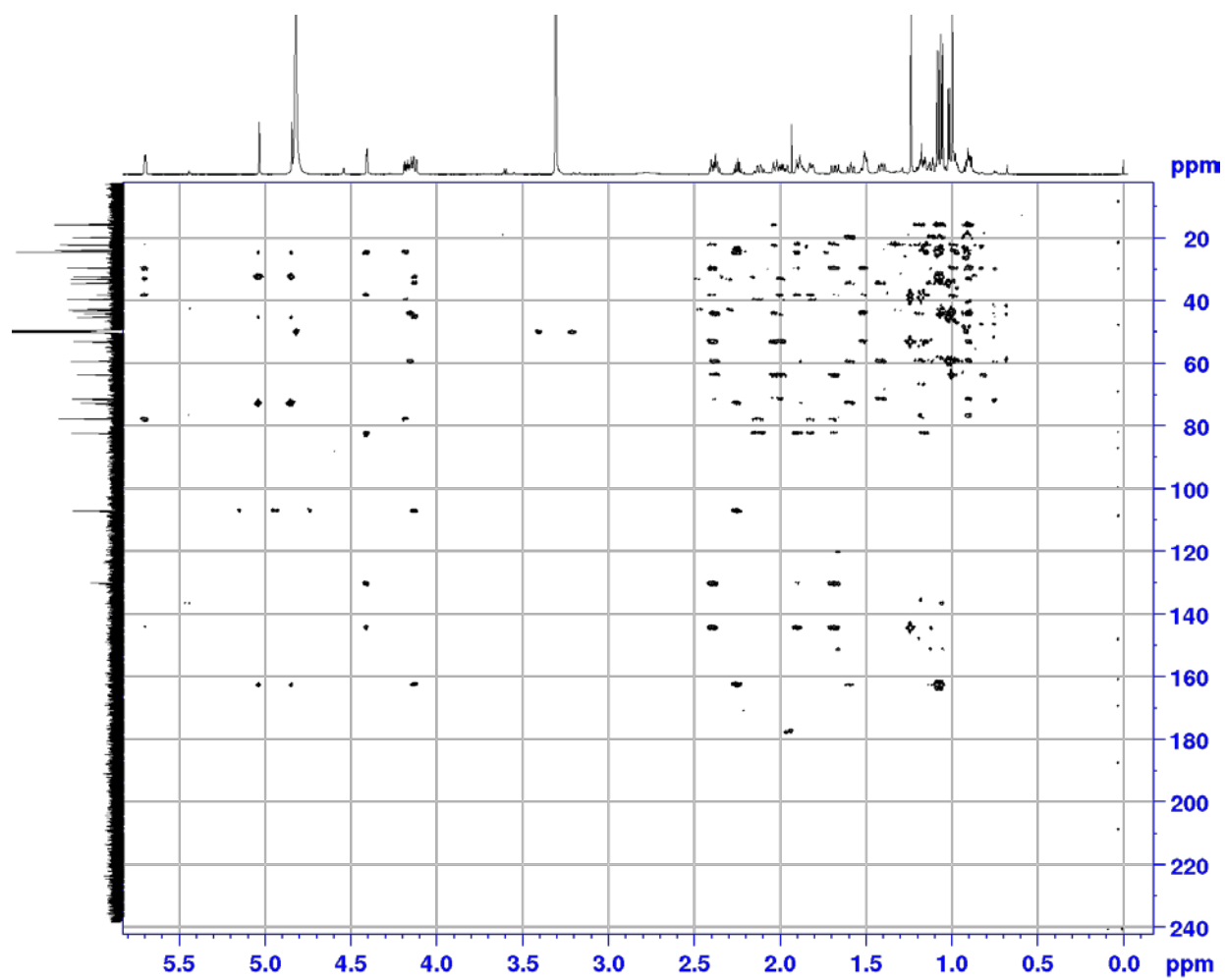


Figure S33. The NOESY (700 MHz, CD₃OD) spectrum of compound 4

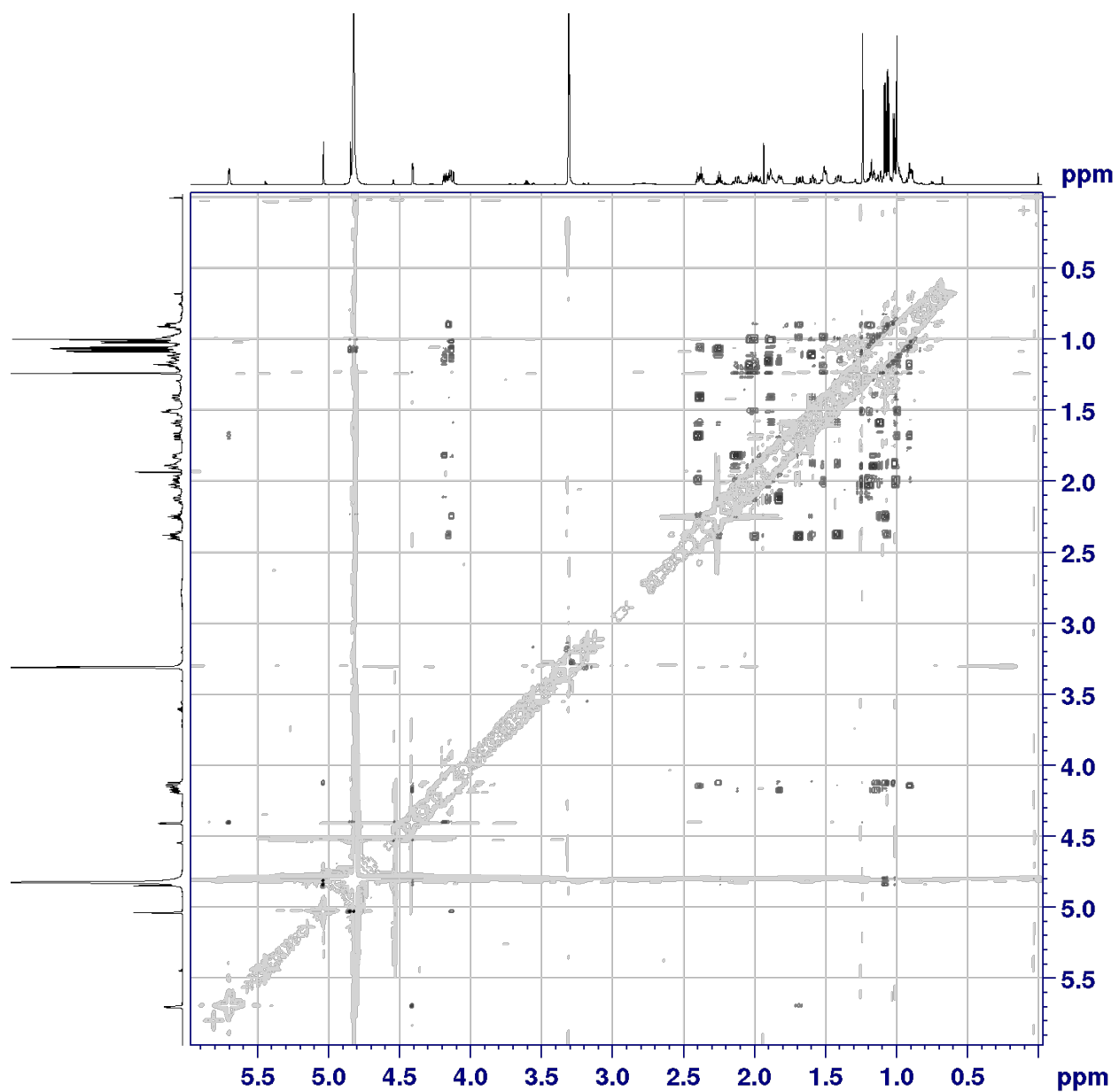


Figure S34. The HRESIMS spectrum of compound **4**

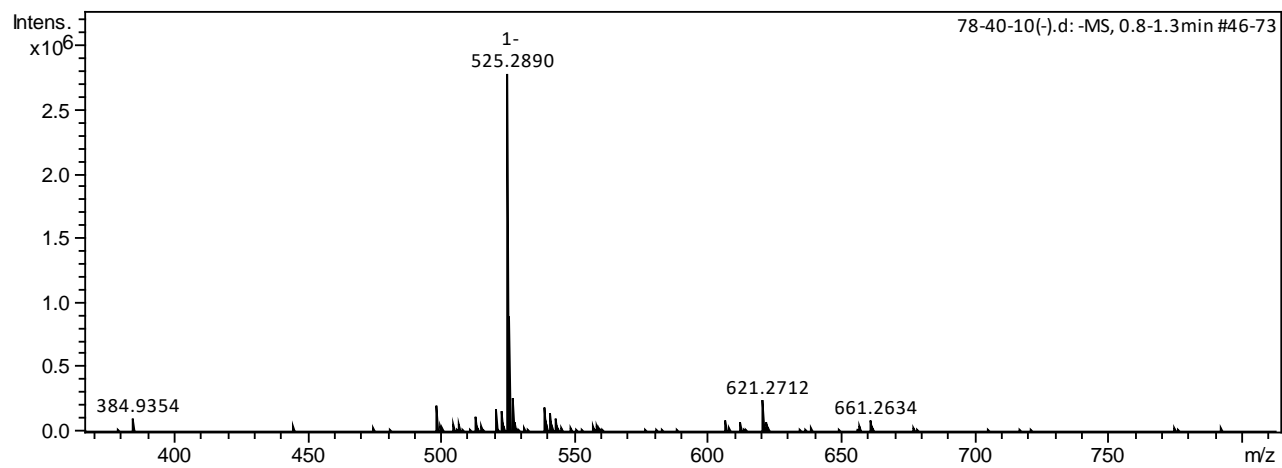


Figure S35. ^1H NMR chemical shift differences between **4S** (red) and **4R** (blue)

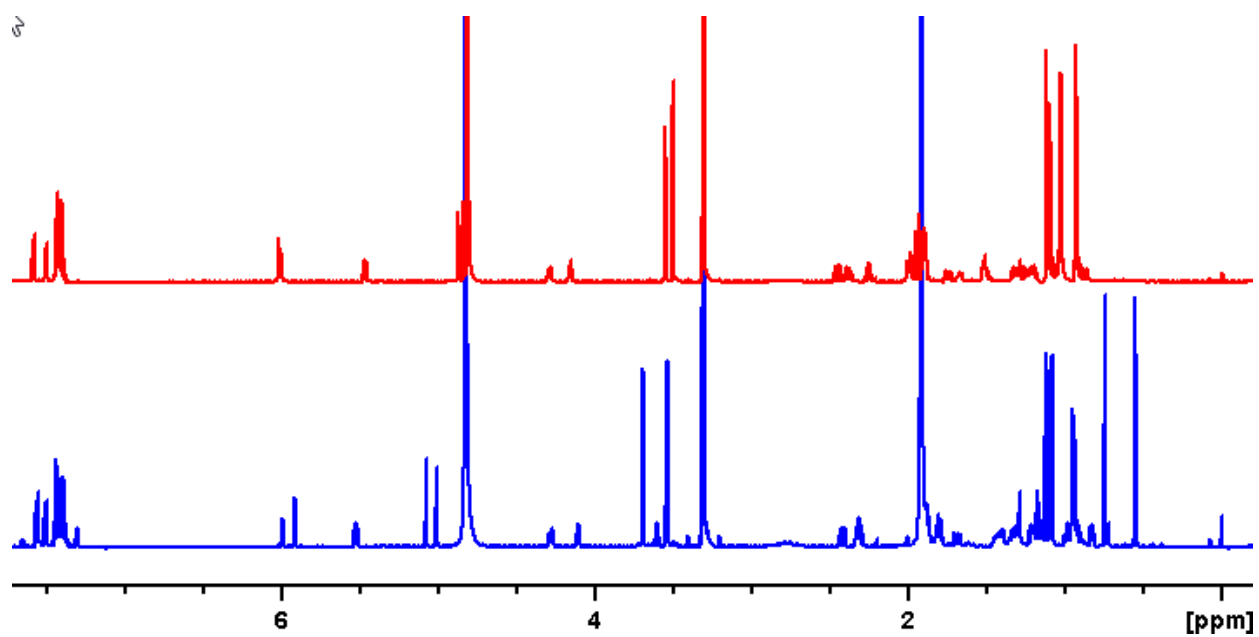


Figure S36. The HRESIMS spectrum of compound **4R (4S)**

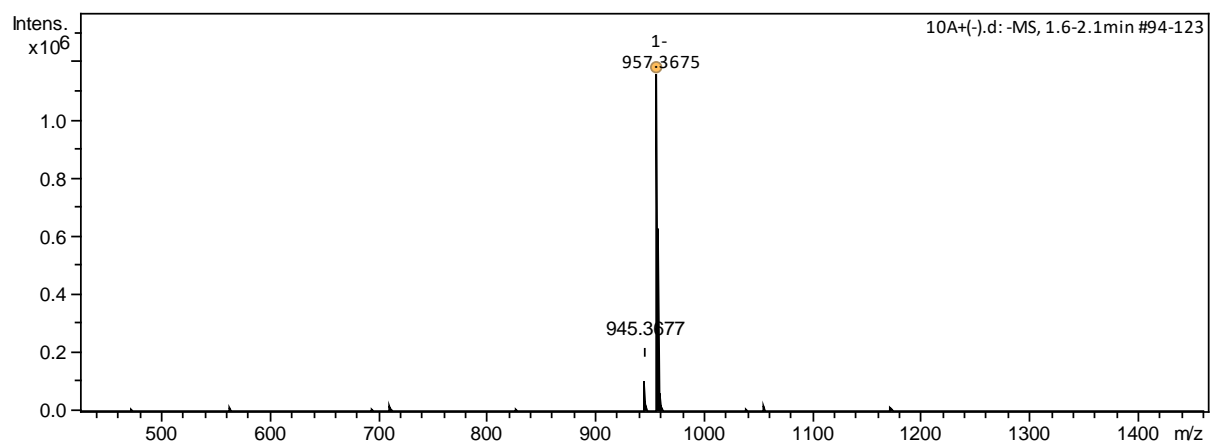


Figure S37. The ^1H NMR (500 MHz, CD_3OD) spectrum of compound **5**

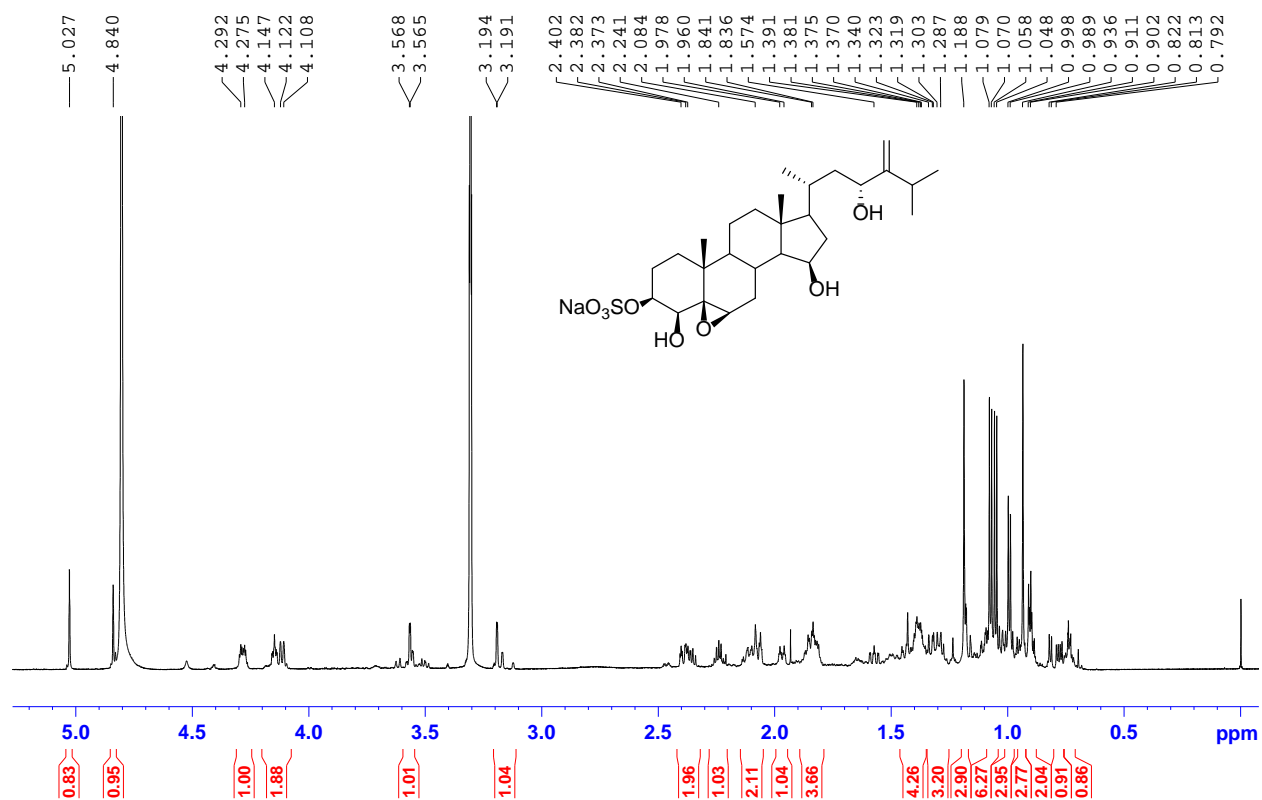


Figure S38. The ^{13}C NMR (125 MHz, CD_3OD) spectrum of compound **5**

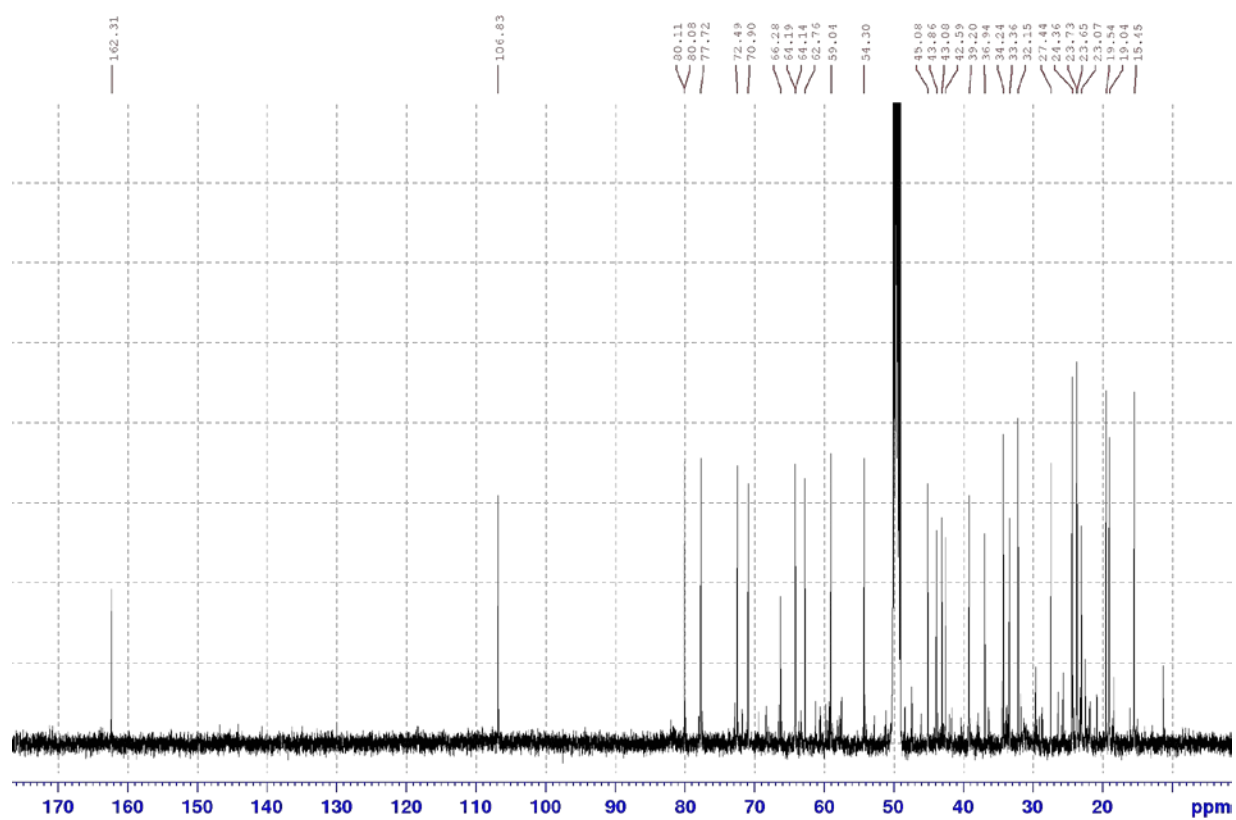


Figure S39. The COSY (500 MHz, CD₃OD) spectrum of compound 5

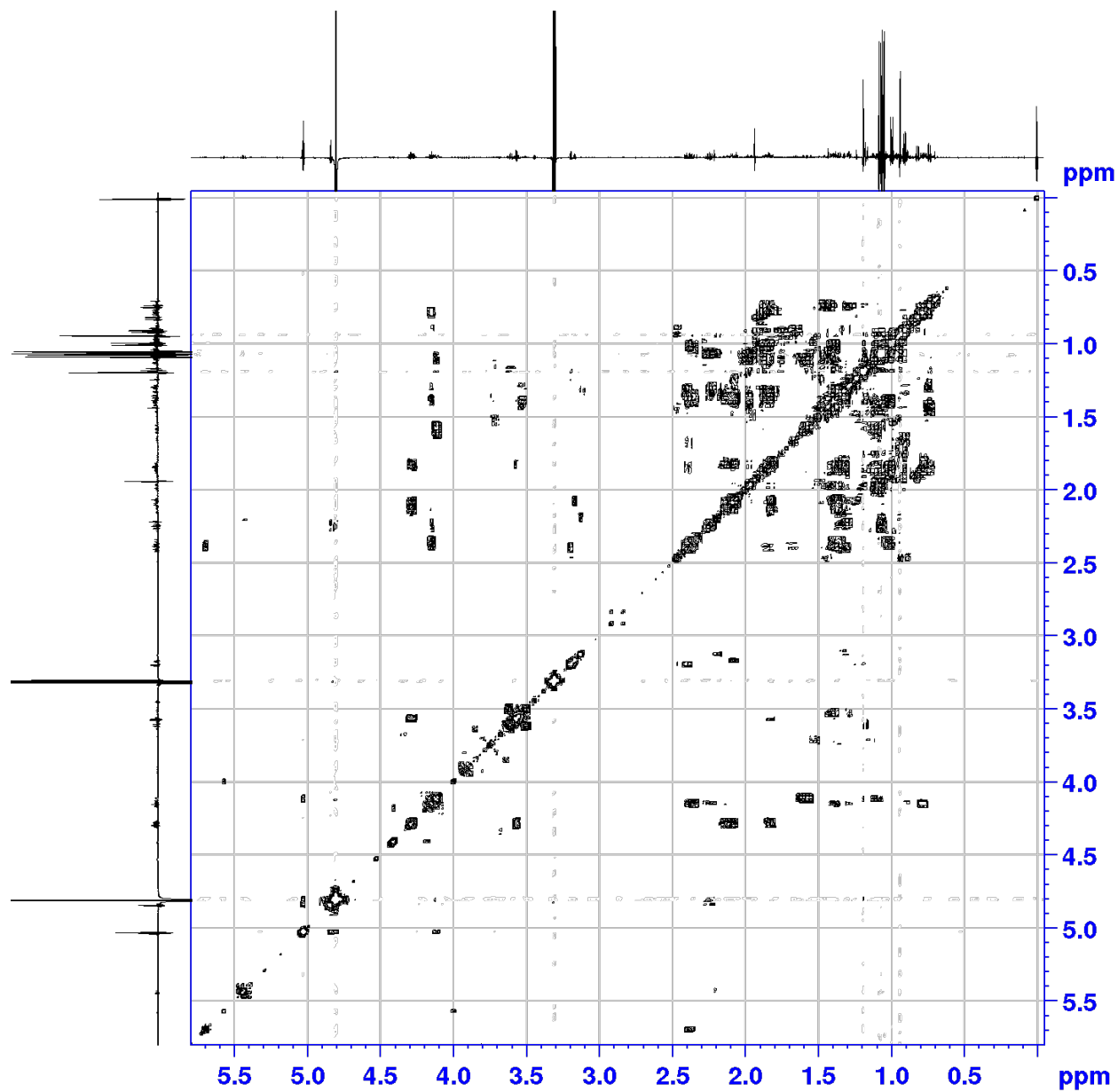


Figure S40. The HSQC (500/125 MHz, CD₃OD) spectrum of compound **5**

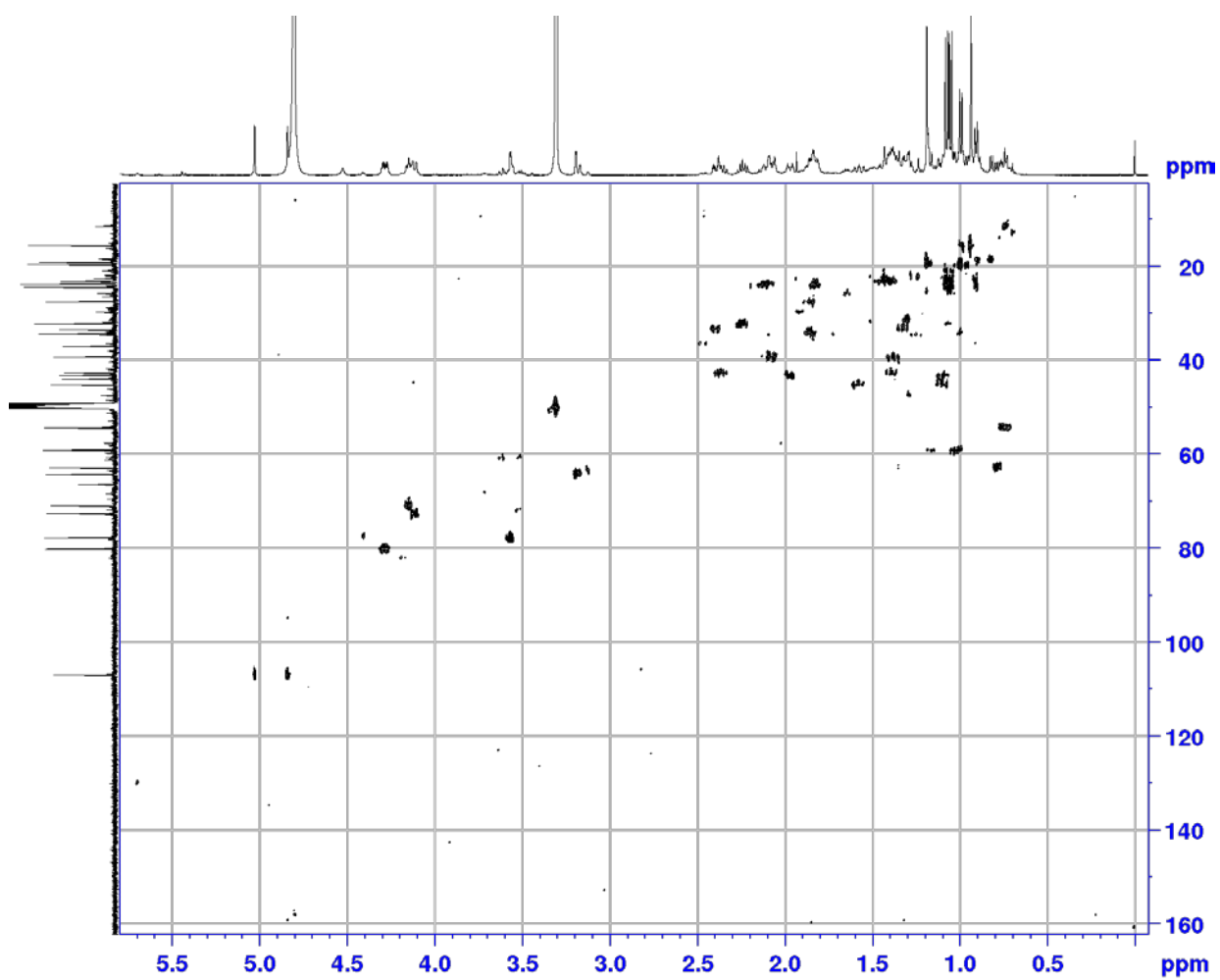


Figure S41. The HMBC (500/125 MHz, CD₃OD) spectrum of compound **5**

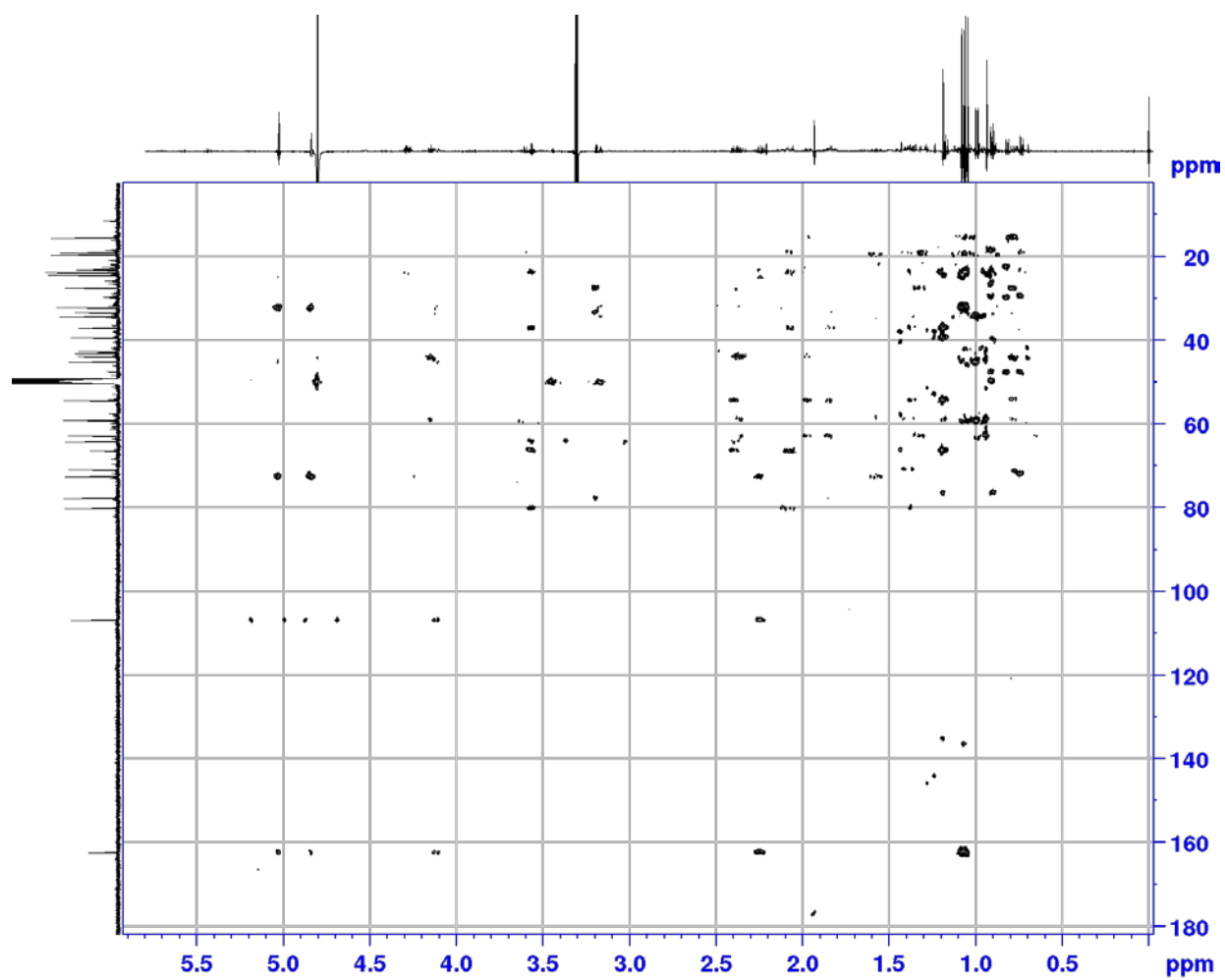


Figure S42. The NOESY (500 MHz, CD₃OD) spectrum of compound **5**

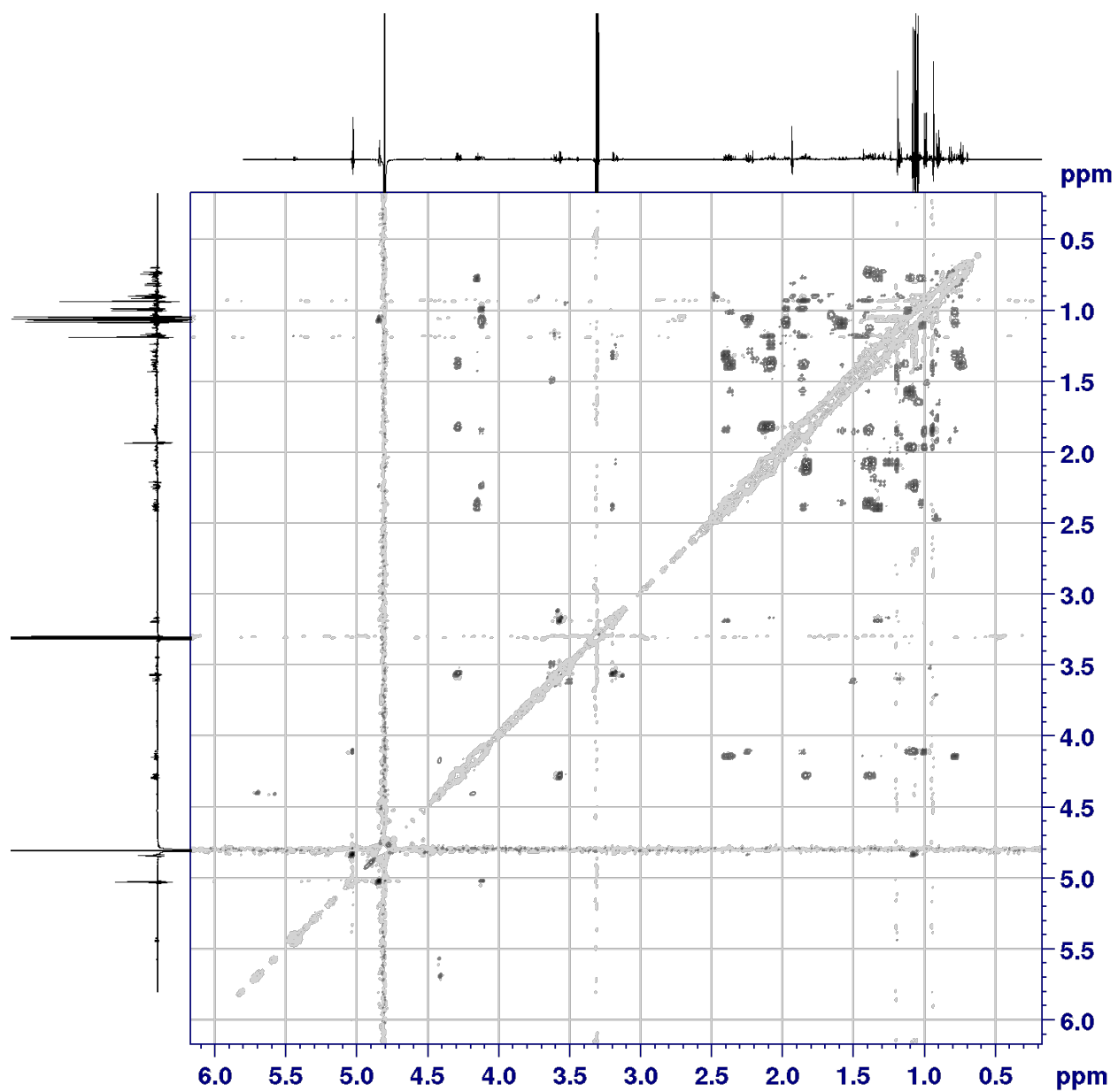


Figure S43. The HRESIMS spectrum of compound **5**

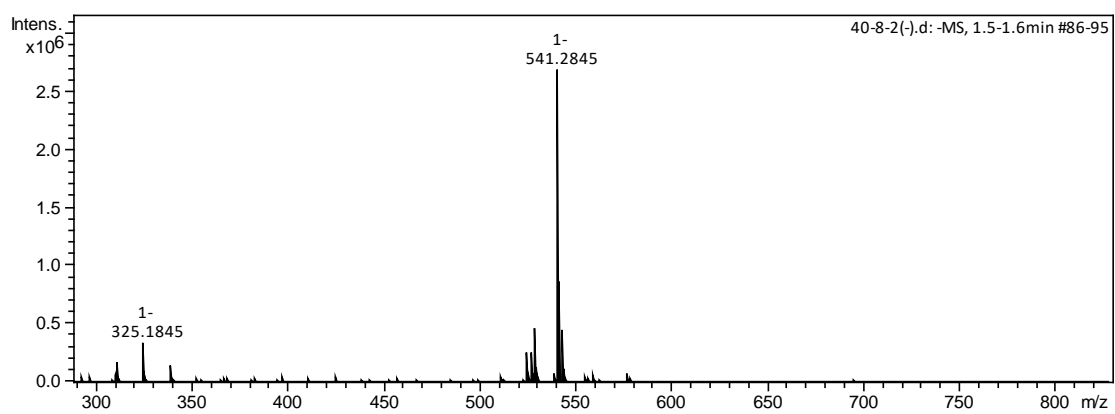


Figure S44. The ^1H NMR (700 MHz, CD_3OD) spectrum of compound **6**

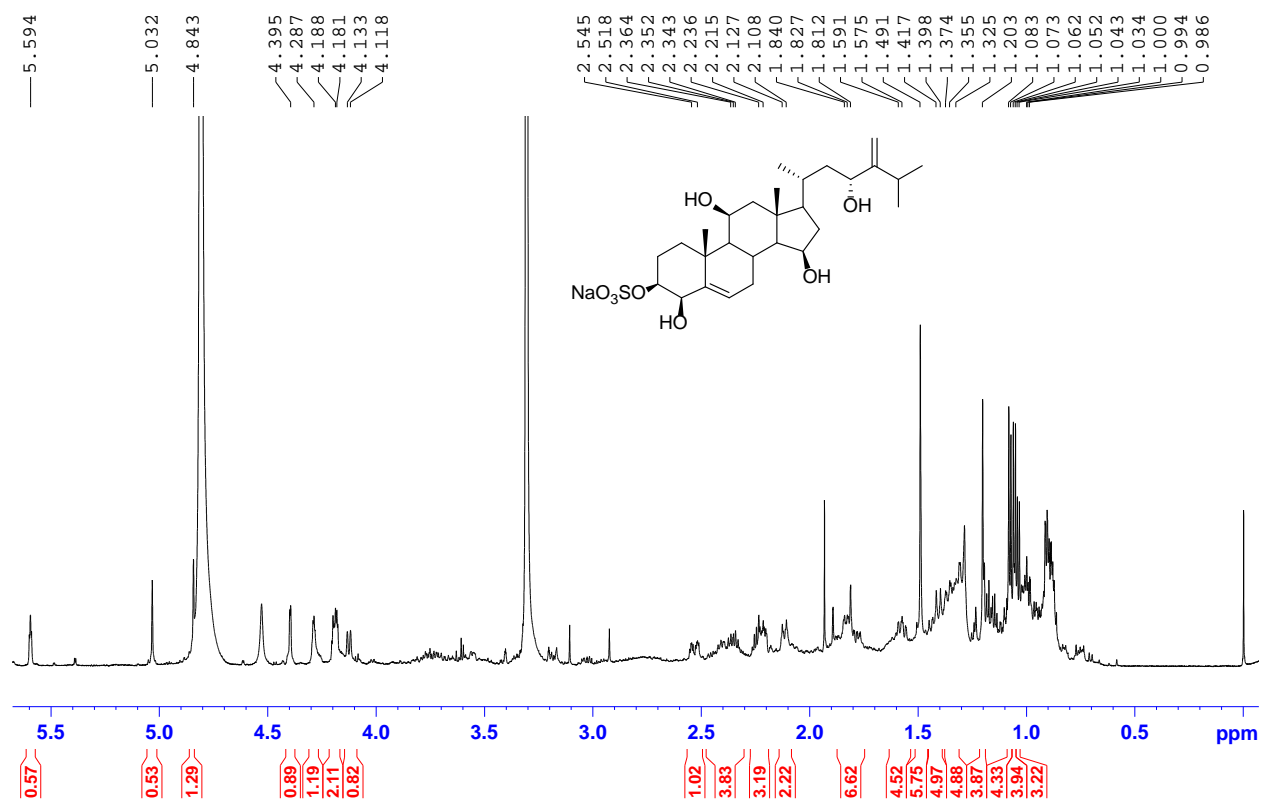


Figure S45. The ^{13}C NMR (175 MHz, CD_3OD) spectrum of compound **6**

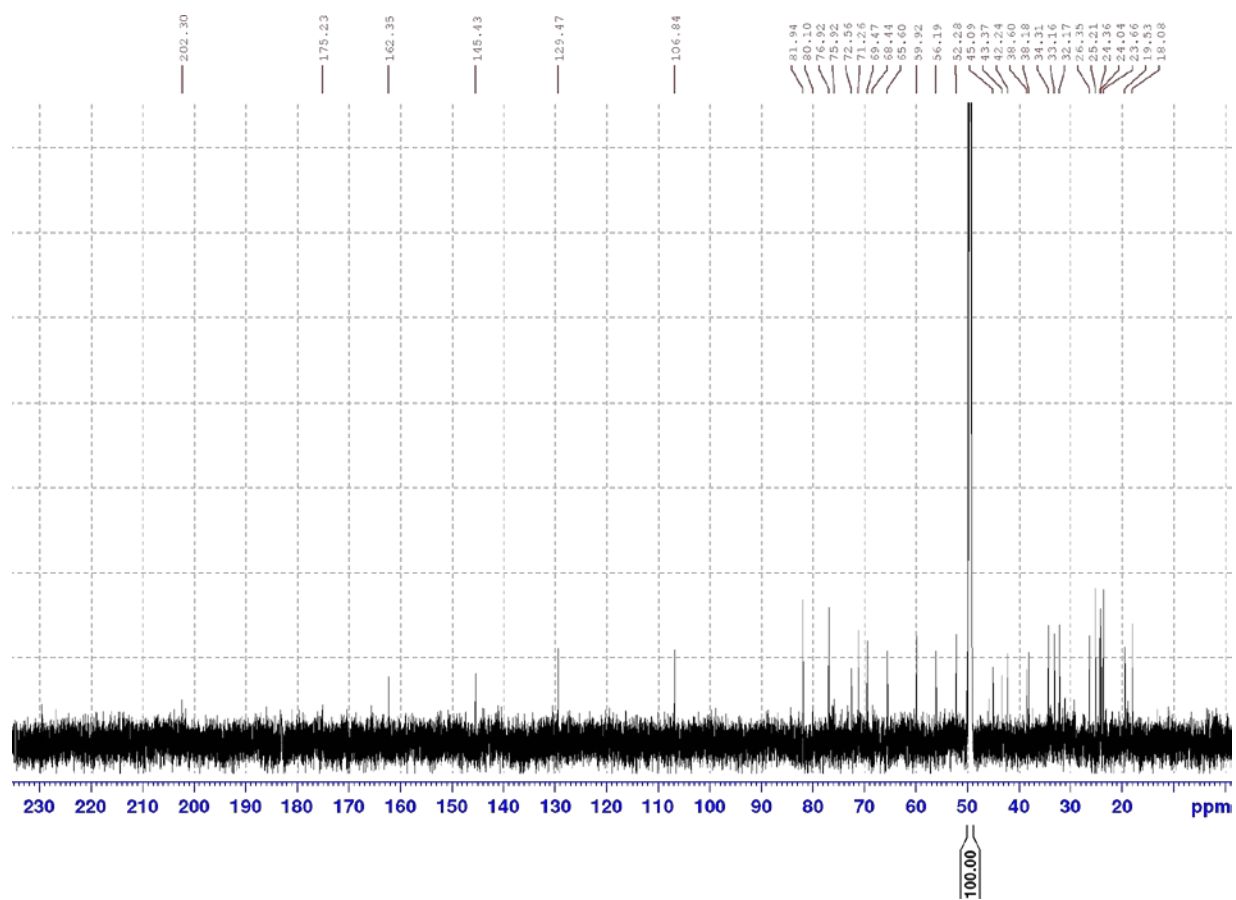


Figure S46. The COSY (700 MHz, CD₃OD) spectrum of compound **6**

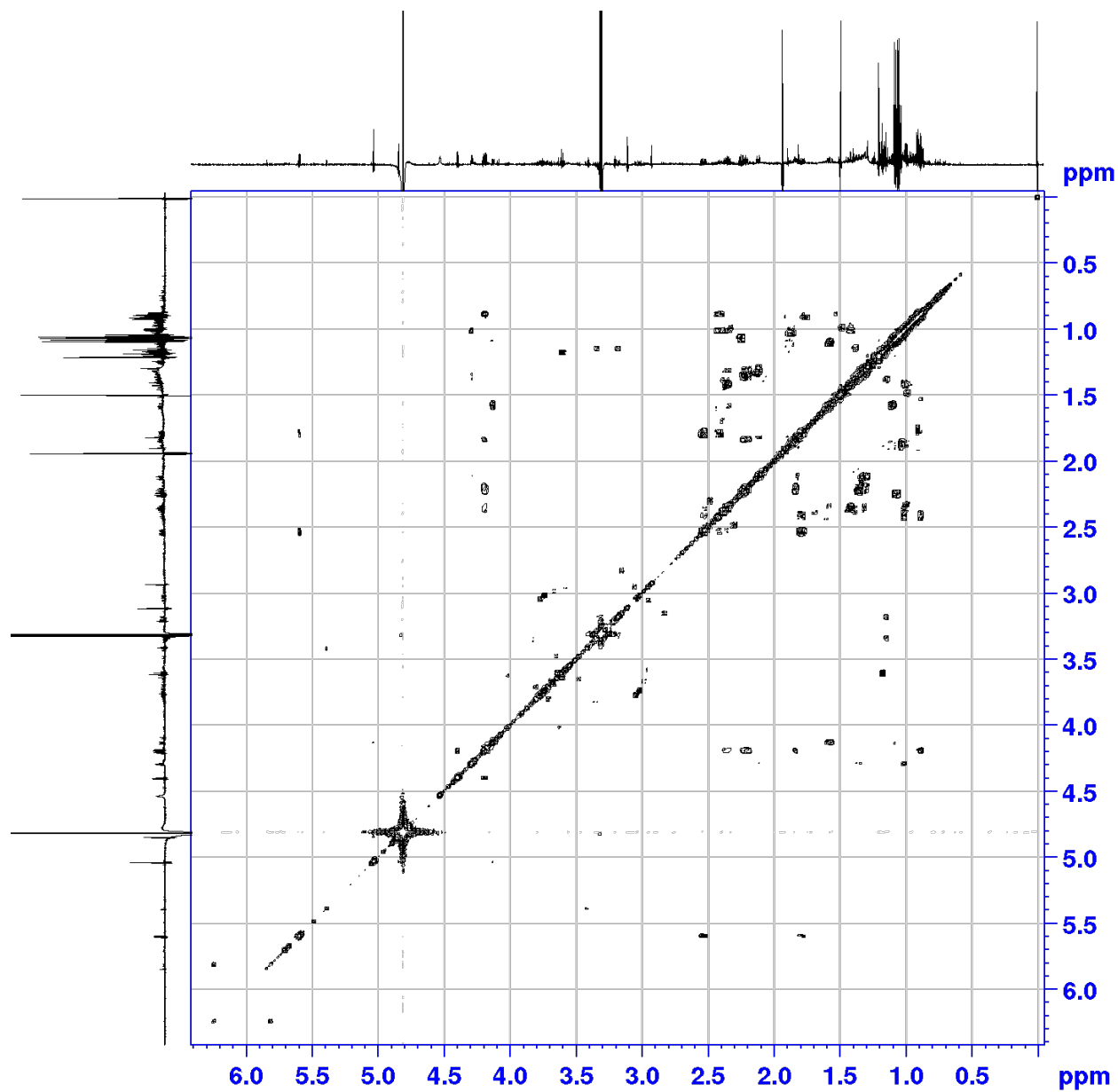


Figure S47. The HSQC (700/175 MHz, CD₃OD) spectrum of compound 6

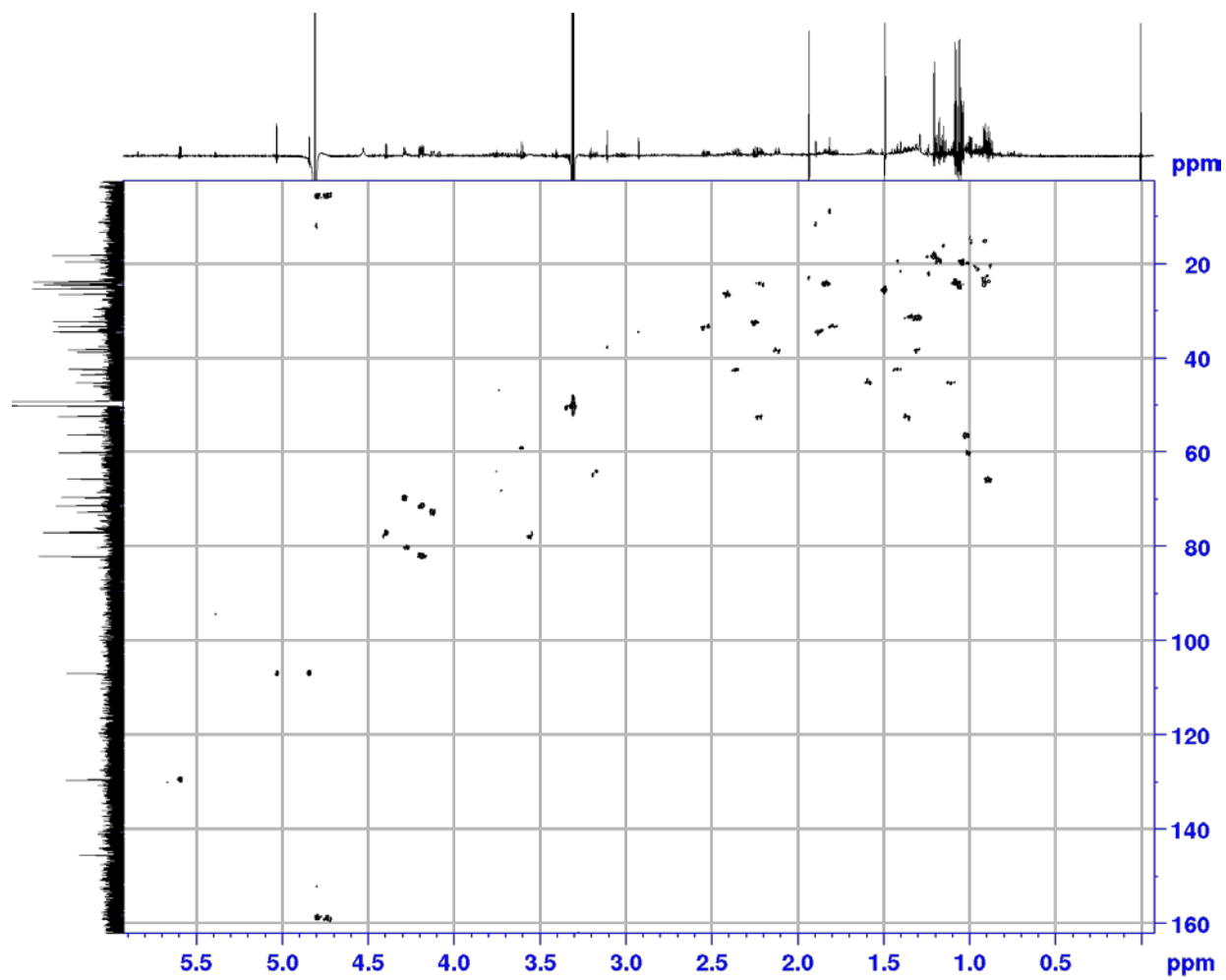


Figure S48. The HMBC (700/175 MHz, CD₃OD) spectrum of compound **6**

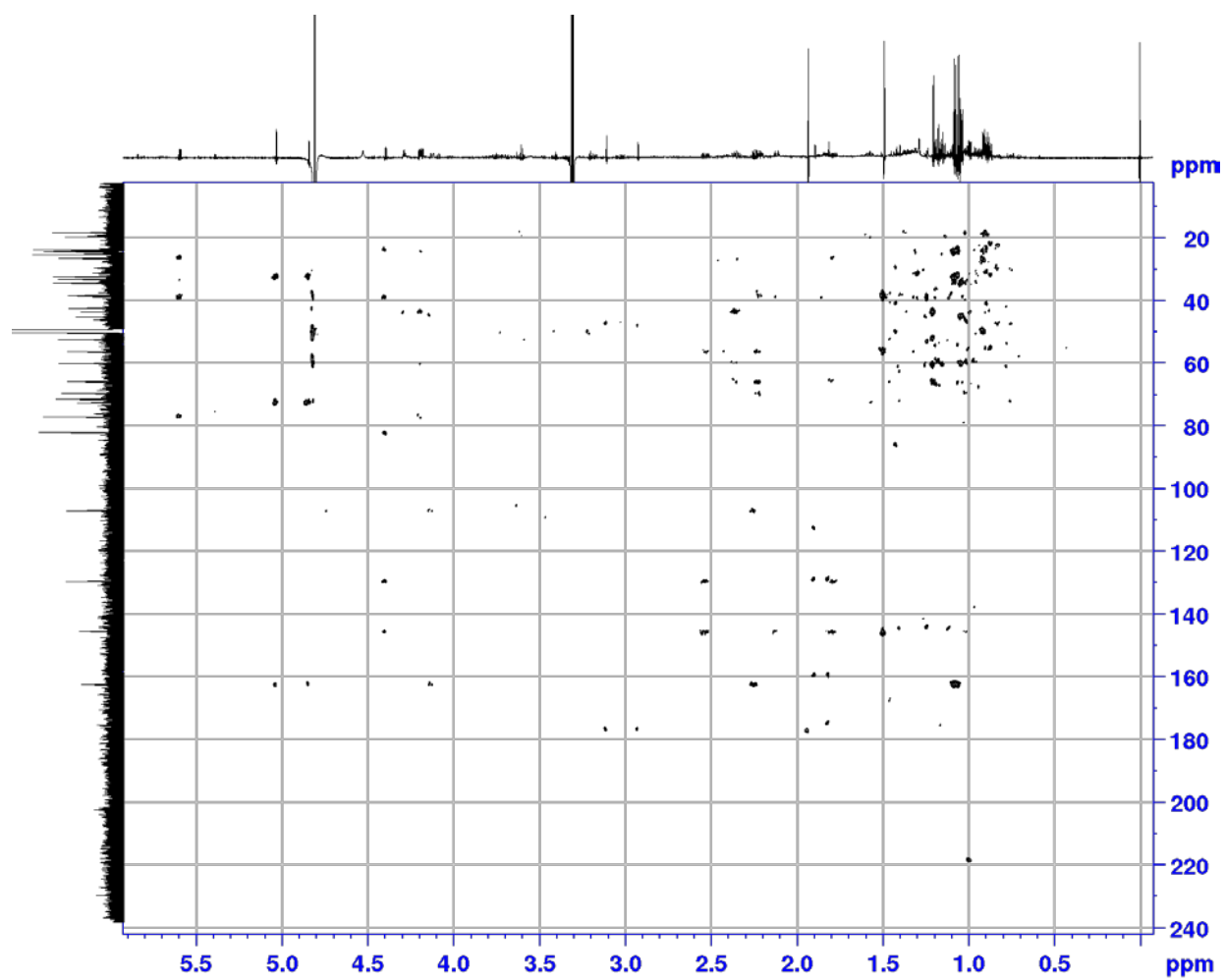


Figure S49. The NOESY (700 MHz, CD₃OD) spectrum of compound 6

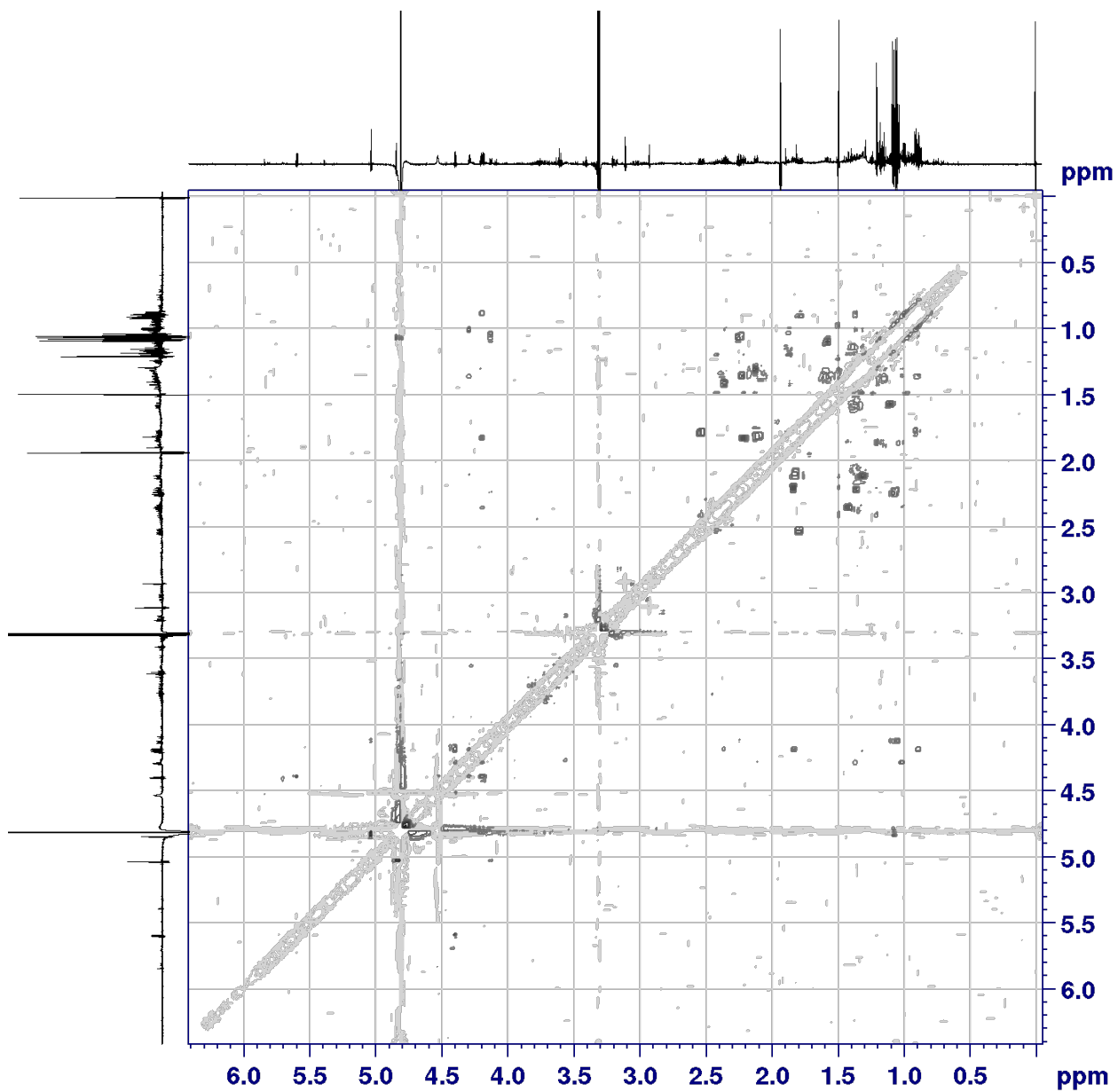


Figure S50. The HRESIMS spectrum of compound **6**

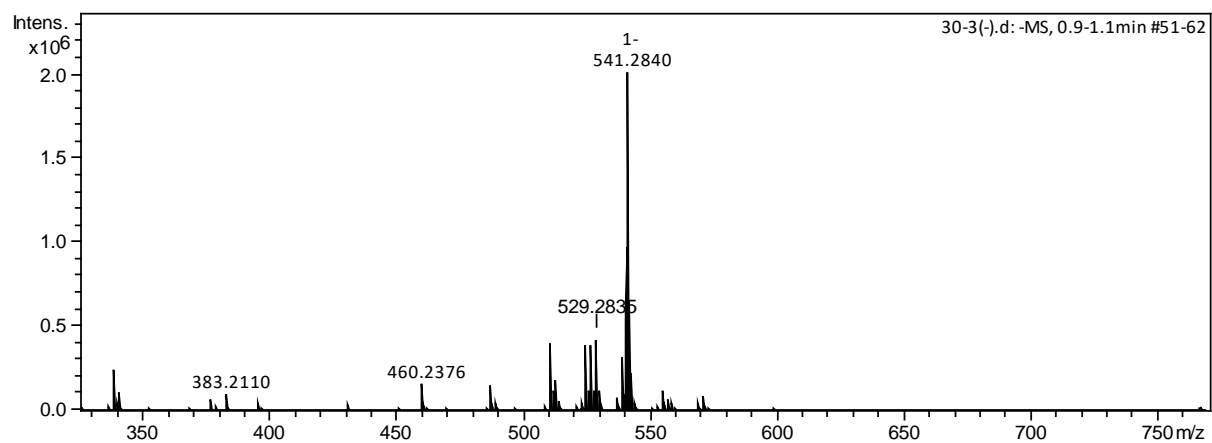


Figure S51. The ^1H NMR (700 MHz, CD_3OD) spectrum of compound 7

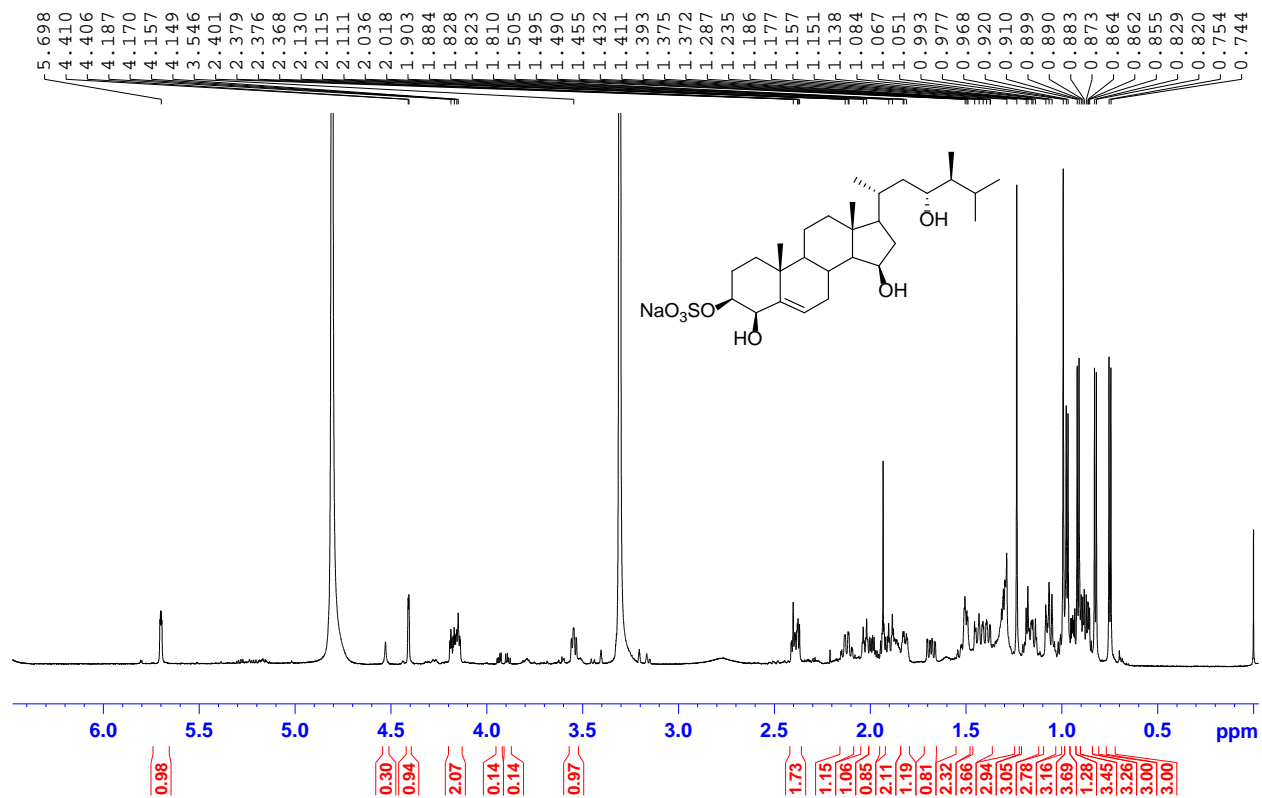


Figure S52. The ^{13}C NMR (175 MHz, CD_3OD) spectrum of compound **7**

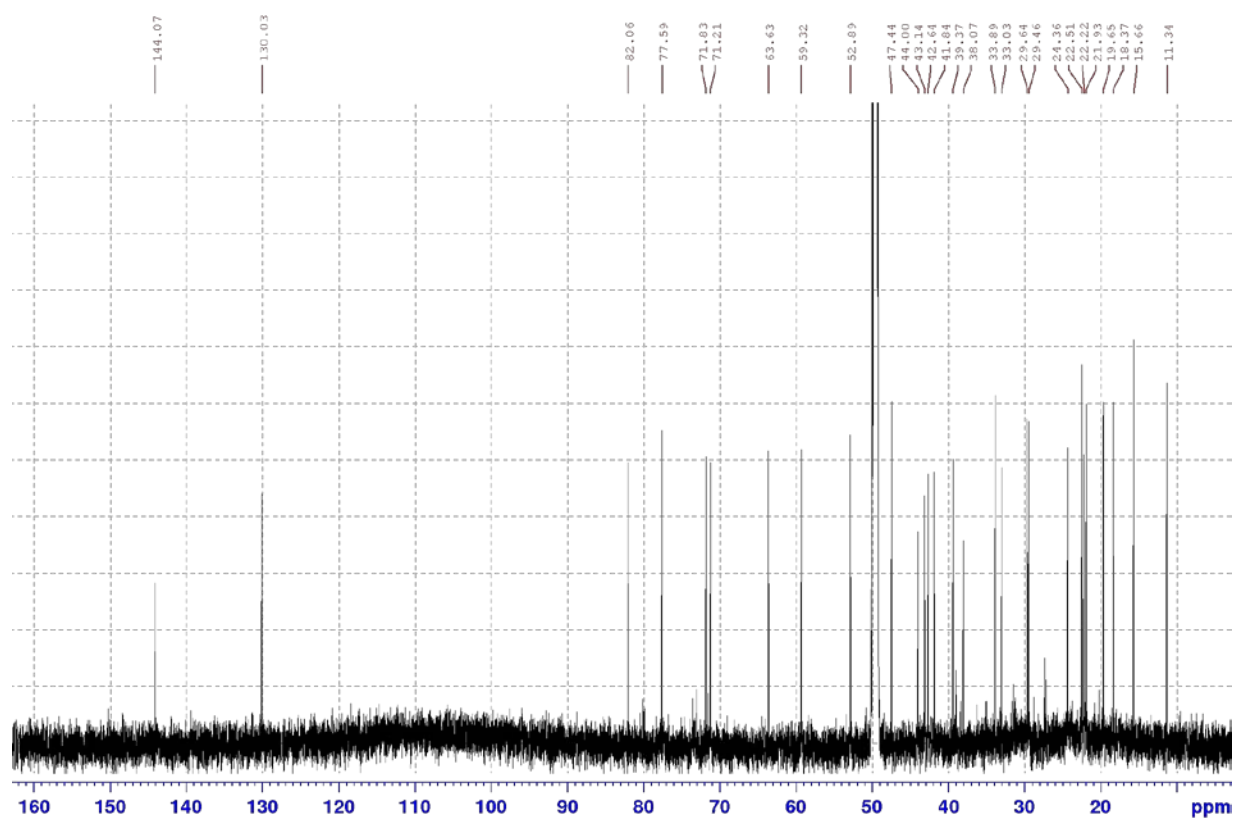


Figure S53. The COSY (700 MHz, CD₃OD) spectrum of compound 7

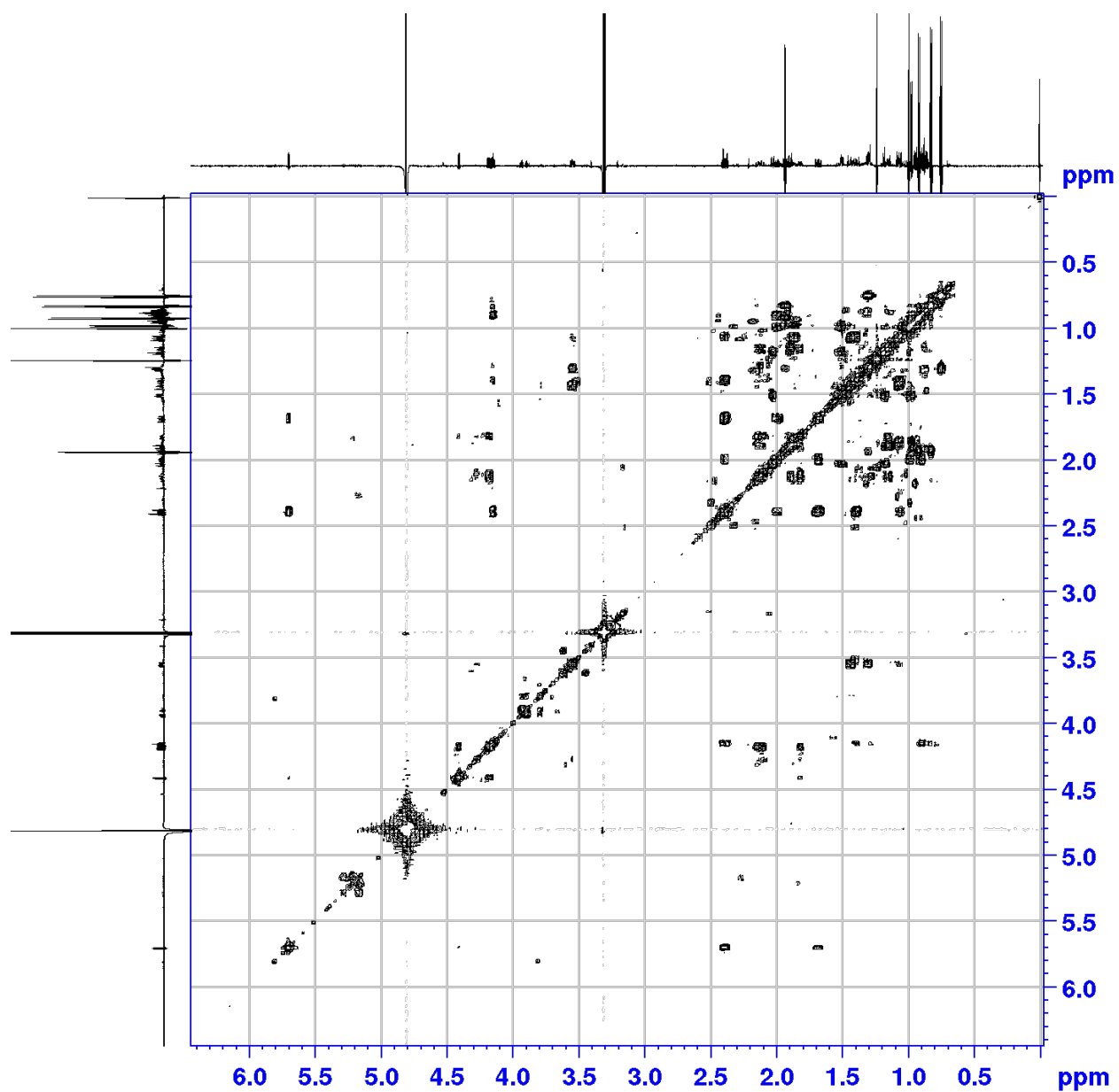


Figure S54. The HSQC (700/175 MHz, CD₃OD) spectrum of compound **7**

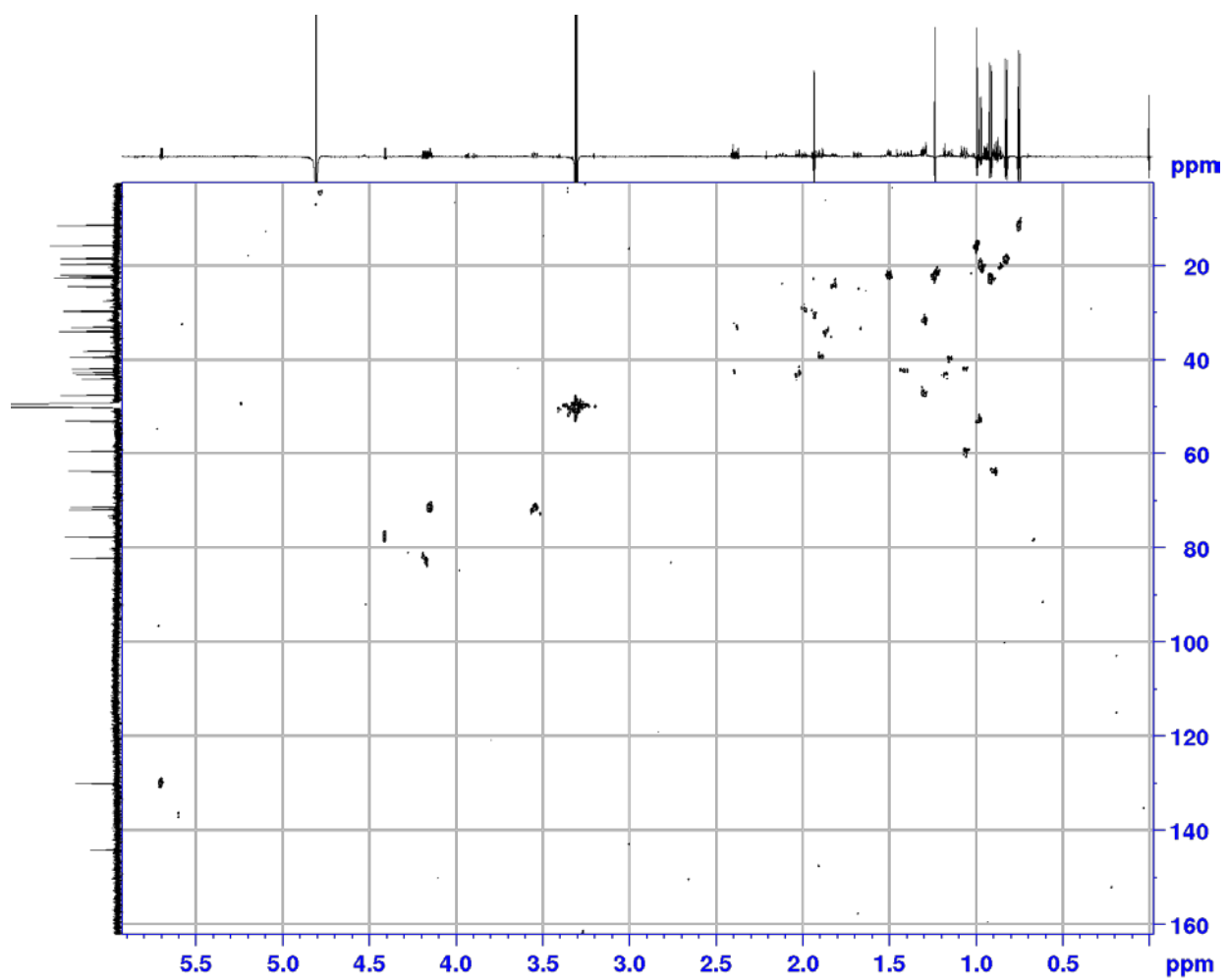


Figure S55. The HMBC (700/175 MHz, CD₃OD) spectrum of compound 7

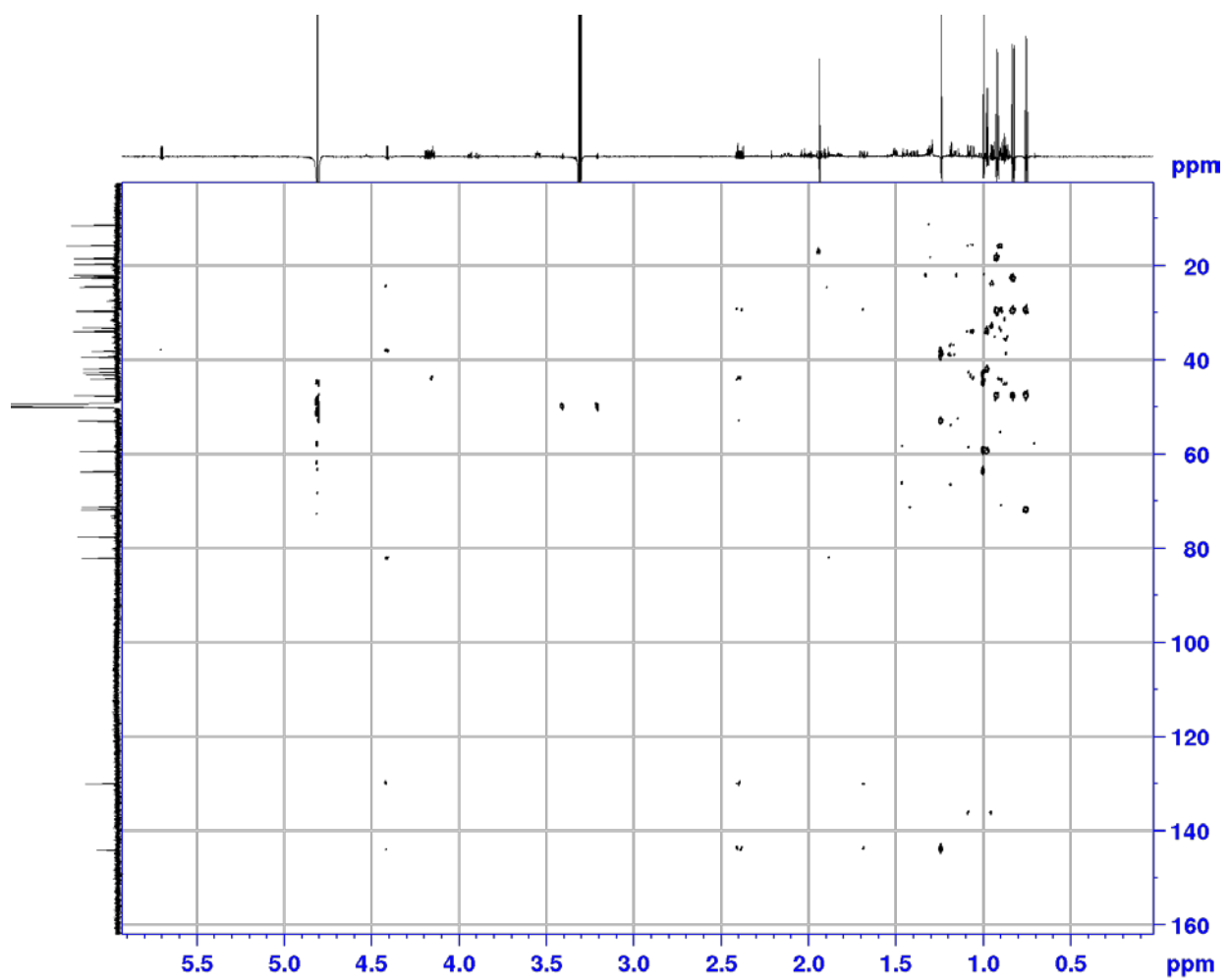


Figure S56. The NOESY (700 MHz, CD₃OD) spectrum of compound 7

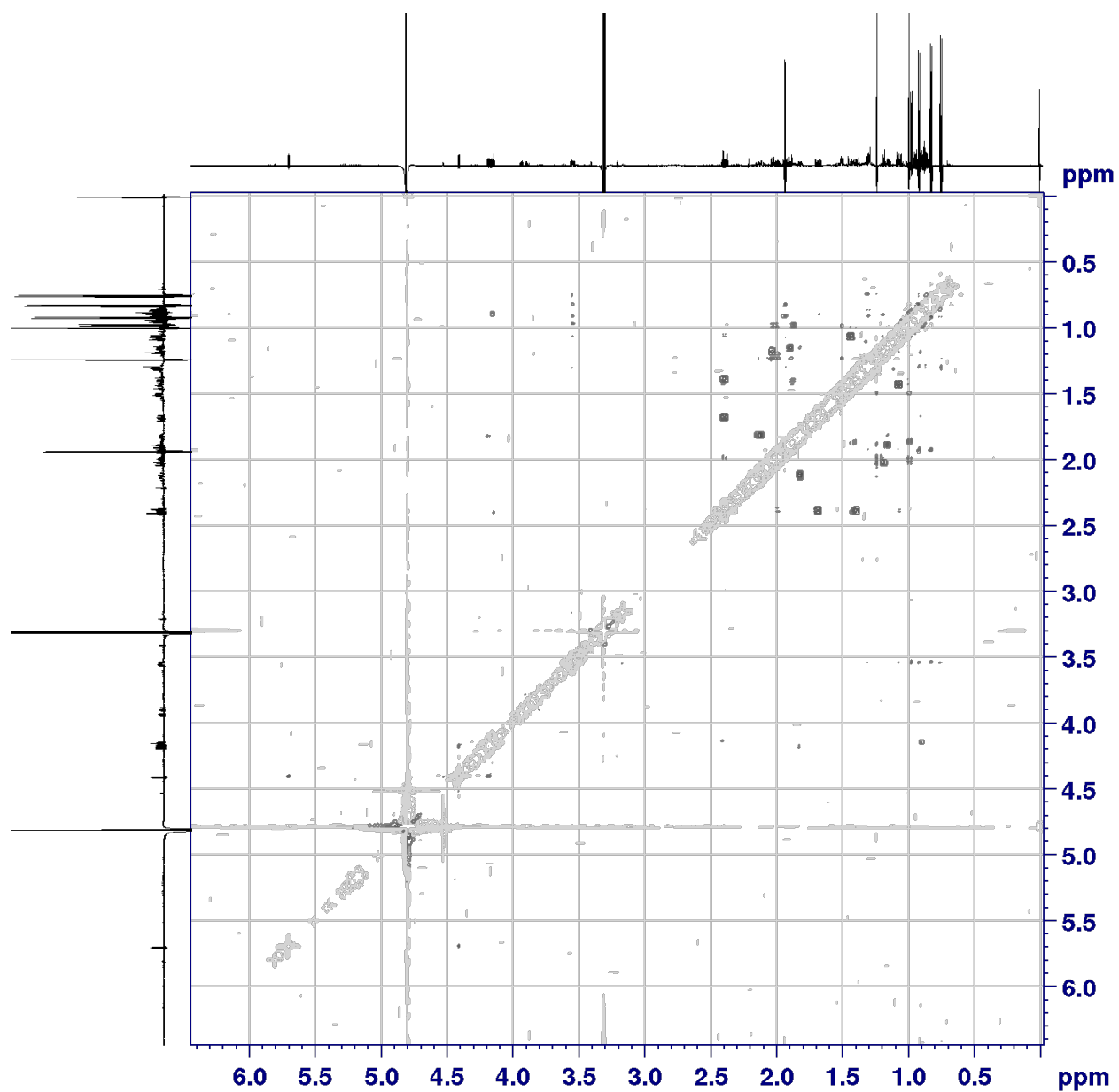


Figure S57. The HRESIMS spectrum of compound **7**

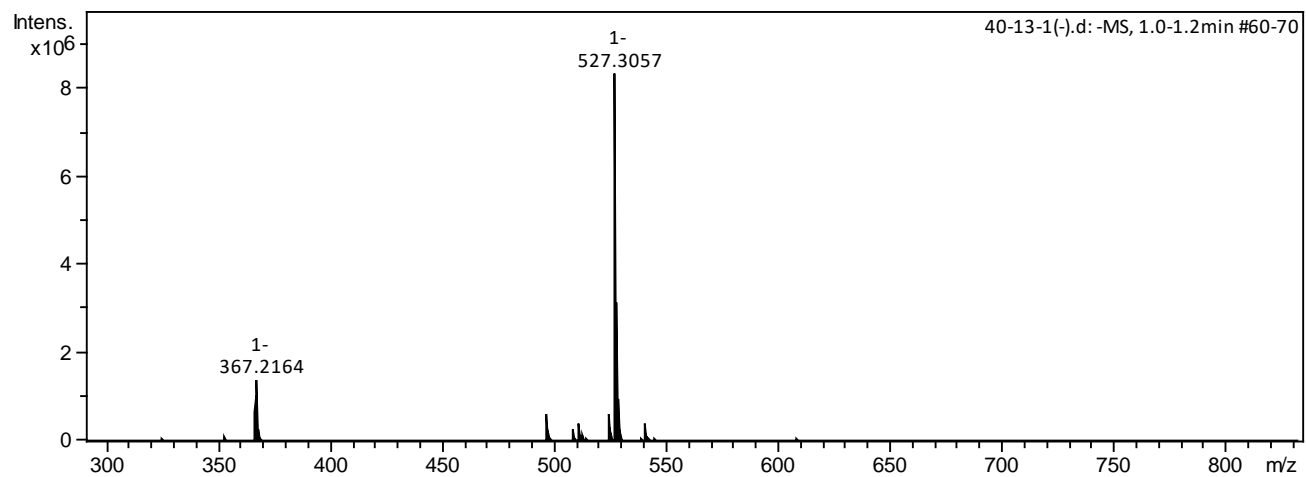


Figure S58. Photo of the sponge *Haliclona gracilis*.

