

## Supplementary data content page

**Title:** Kurilosides A<sub>1</sub>, A<sub>2</sub>, C<sub>1</sub>, D, E and F – triterpene glycosides from the Far Eastern sea cucumber *Thyonidium* (= *Duasmmodactyla*) *kurilensis* (Levin): structures with unusual non-holostane aglycones and cytotoxicities.

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Figure S58. HR-ESI-MS and ESI-MS/MS spectra of kuriloside A (**7**)

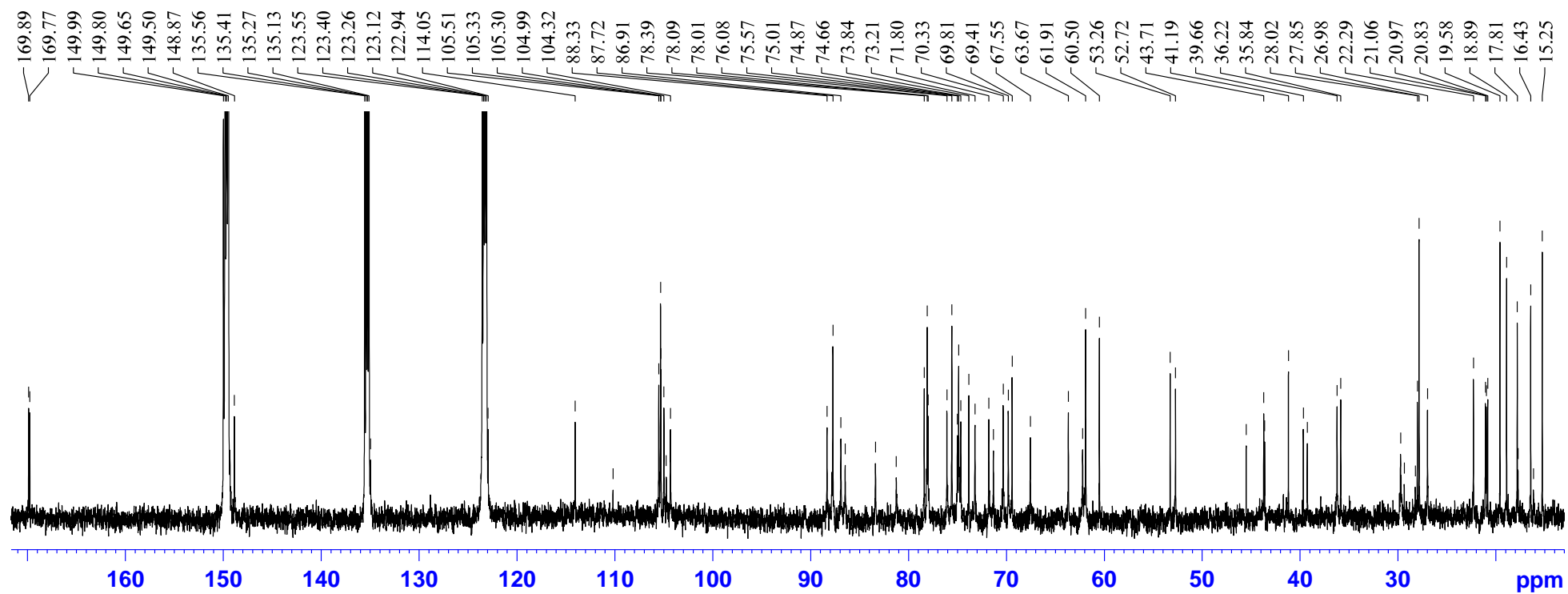


Figure S1. The  $^{13}\text{C}$  NMR (176.03 MHz) spectrum of kuriloside  $\text{A}_1$  (**1**) in  $\text{C}_5\text{D}_5\text{N}/\text{D}_2\text{O}$  (4/1)

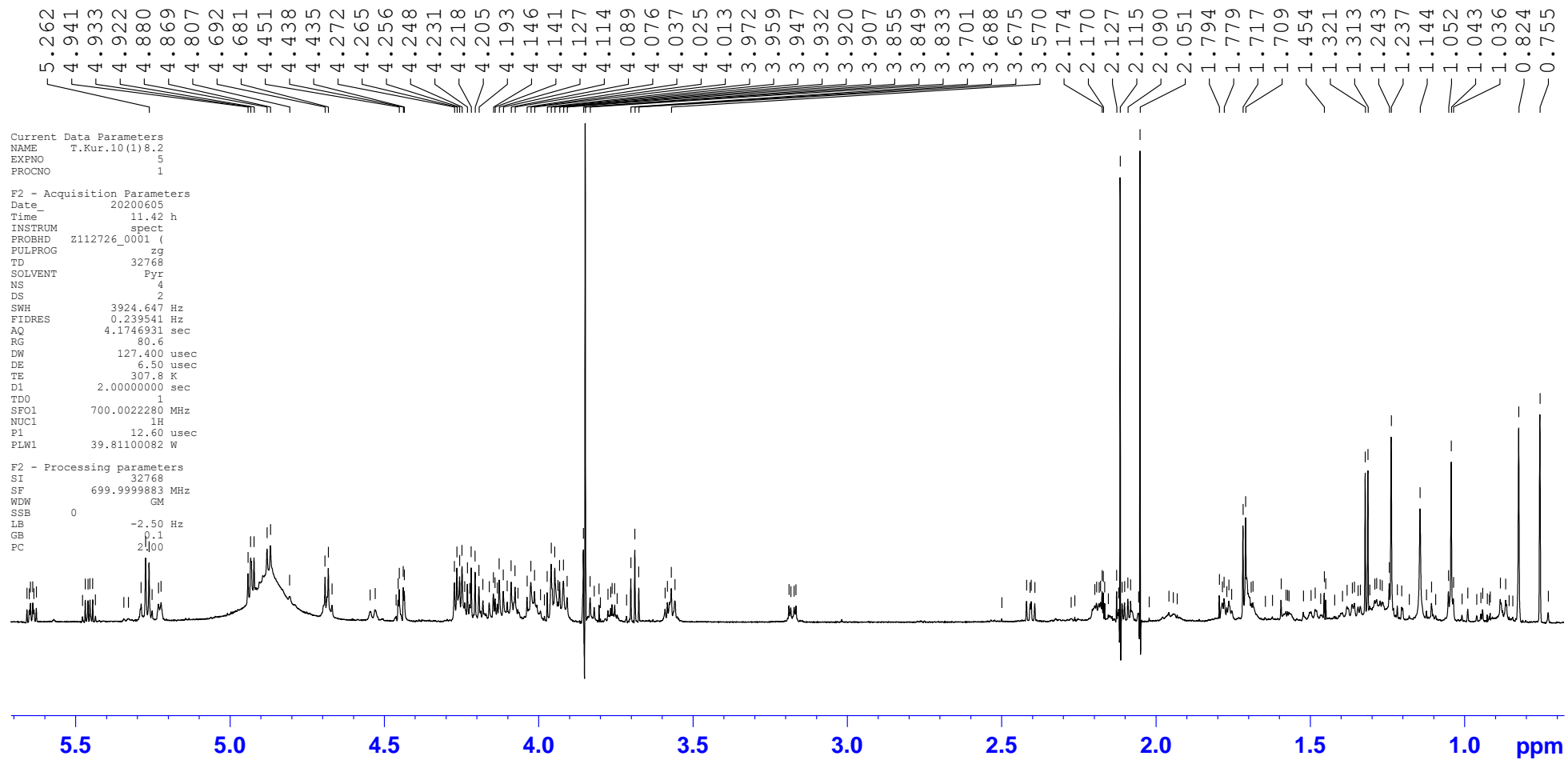


Figure S2. The  $^1\text{H}$  NMR (700.00 MHz) spectrum of kuriloside A<sub>1</sub> (**1**) in  $\text{C}_5\text{D}_5\text{N}/\text{D}_2\text{O}$  (4/1)

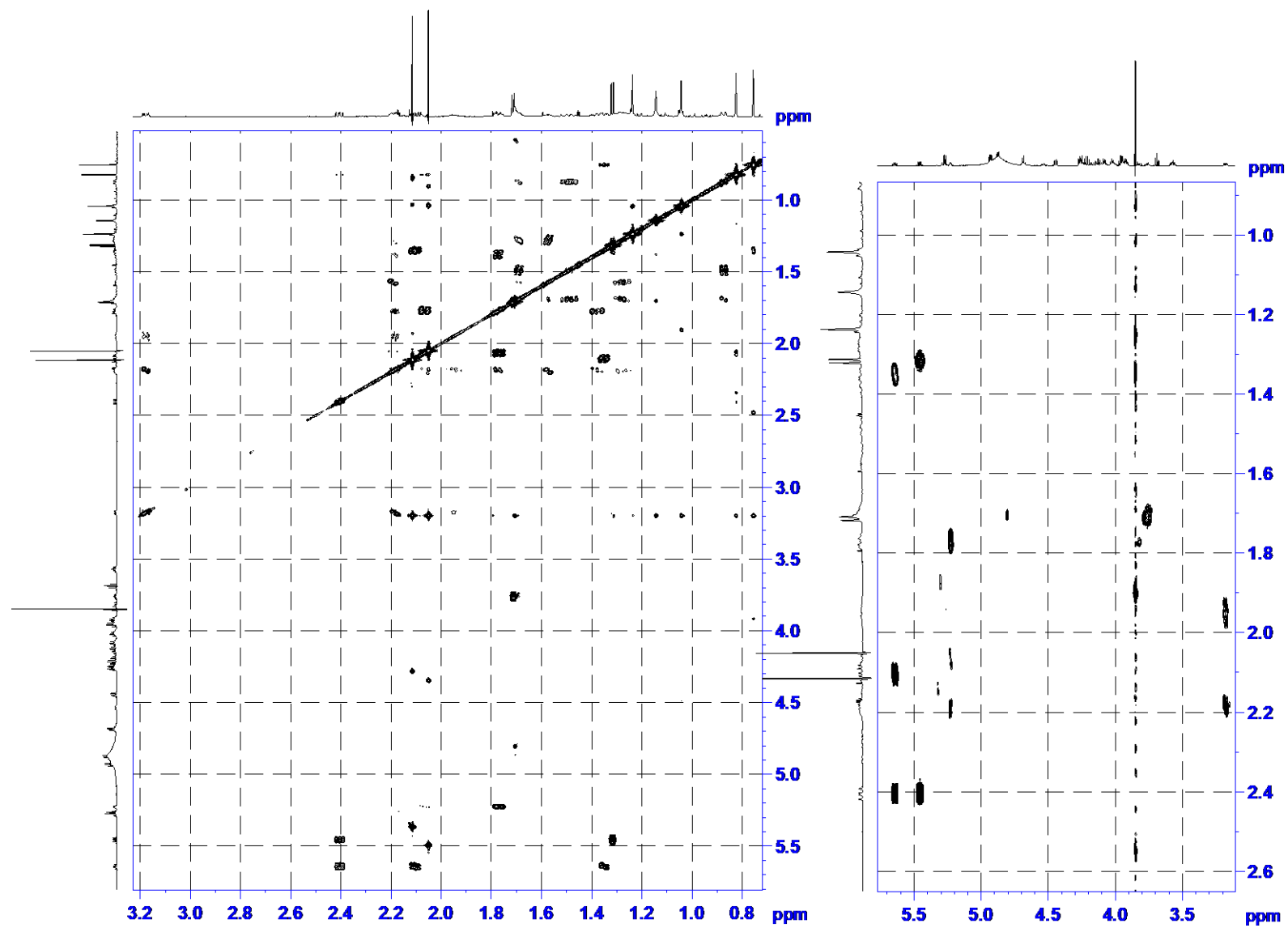


Figure S3. The COSY (700.00 MHz) spectrum of the aglycone part of kuriloside A<sub>1</sub> (**1**) in C<sub>5</sub>D<sub>5</sub>N/D<sub>2</sub>O (4/1)

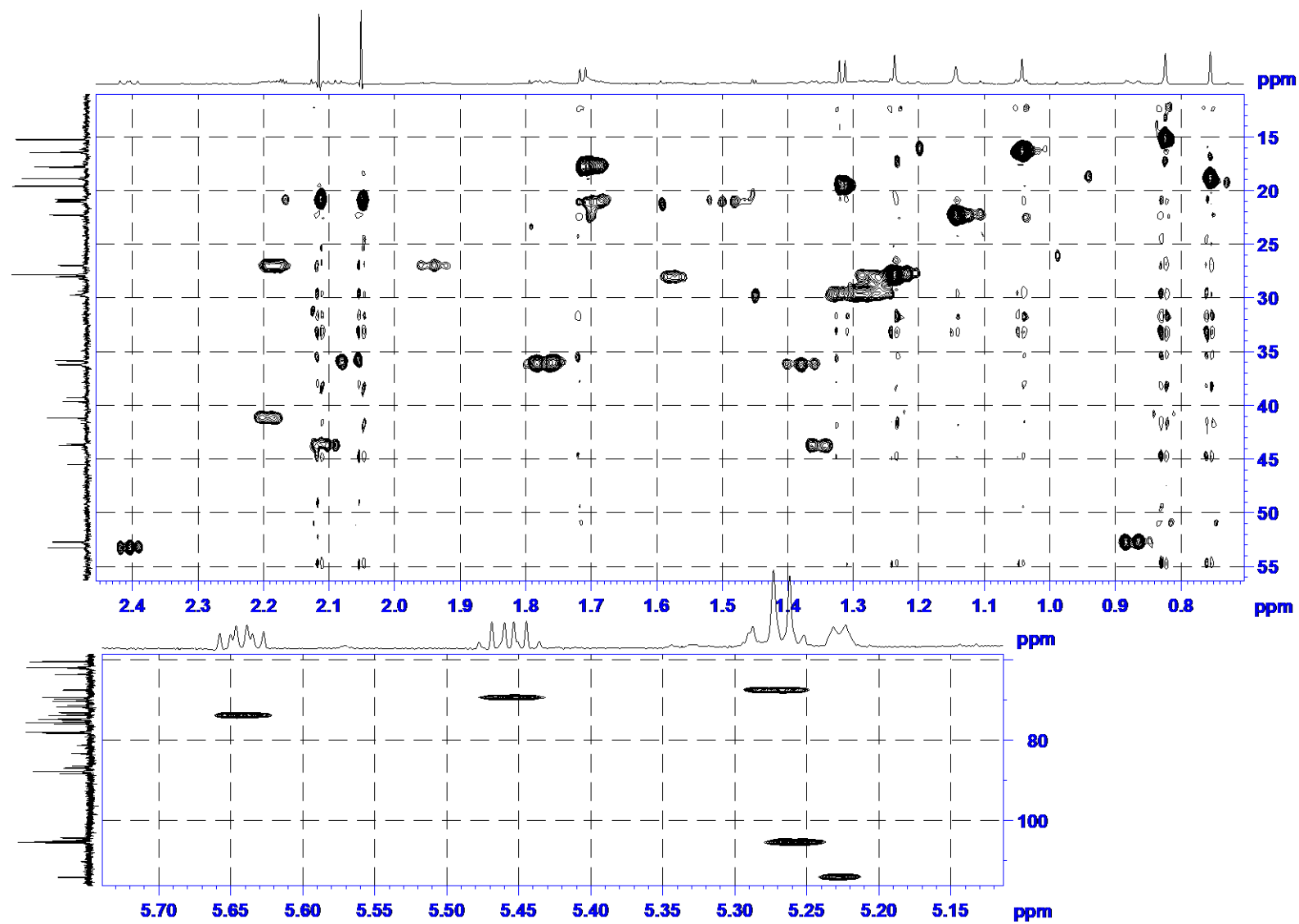


Figure S4. The HSQC (700.00 MHz) spectrum of the aglycone part of kuriloside A<sub>1</sub> (**1**) in C<sub>5</sub>D<sub>5</sub>N/D<sub>2</sub>O (4/1)



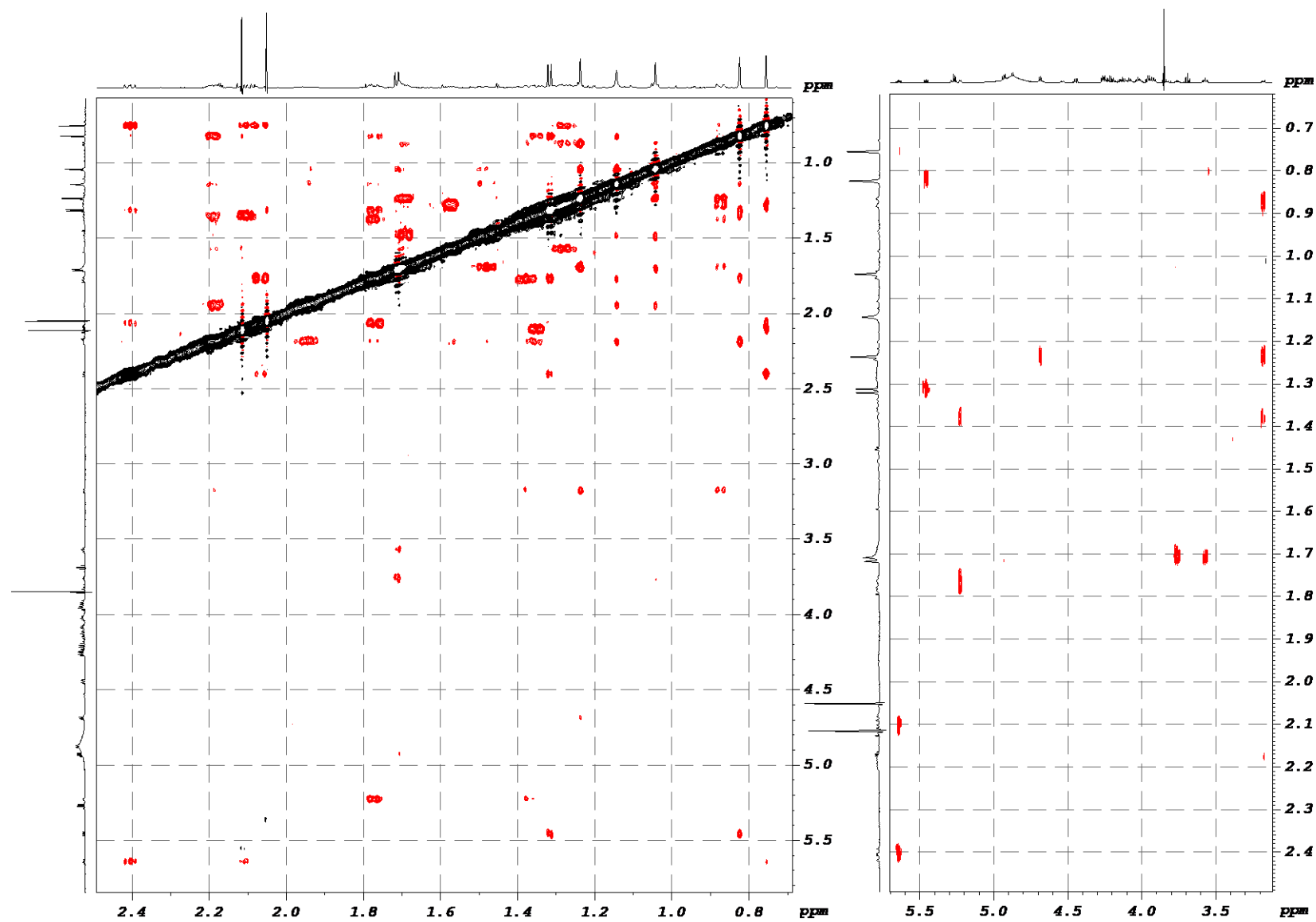


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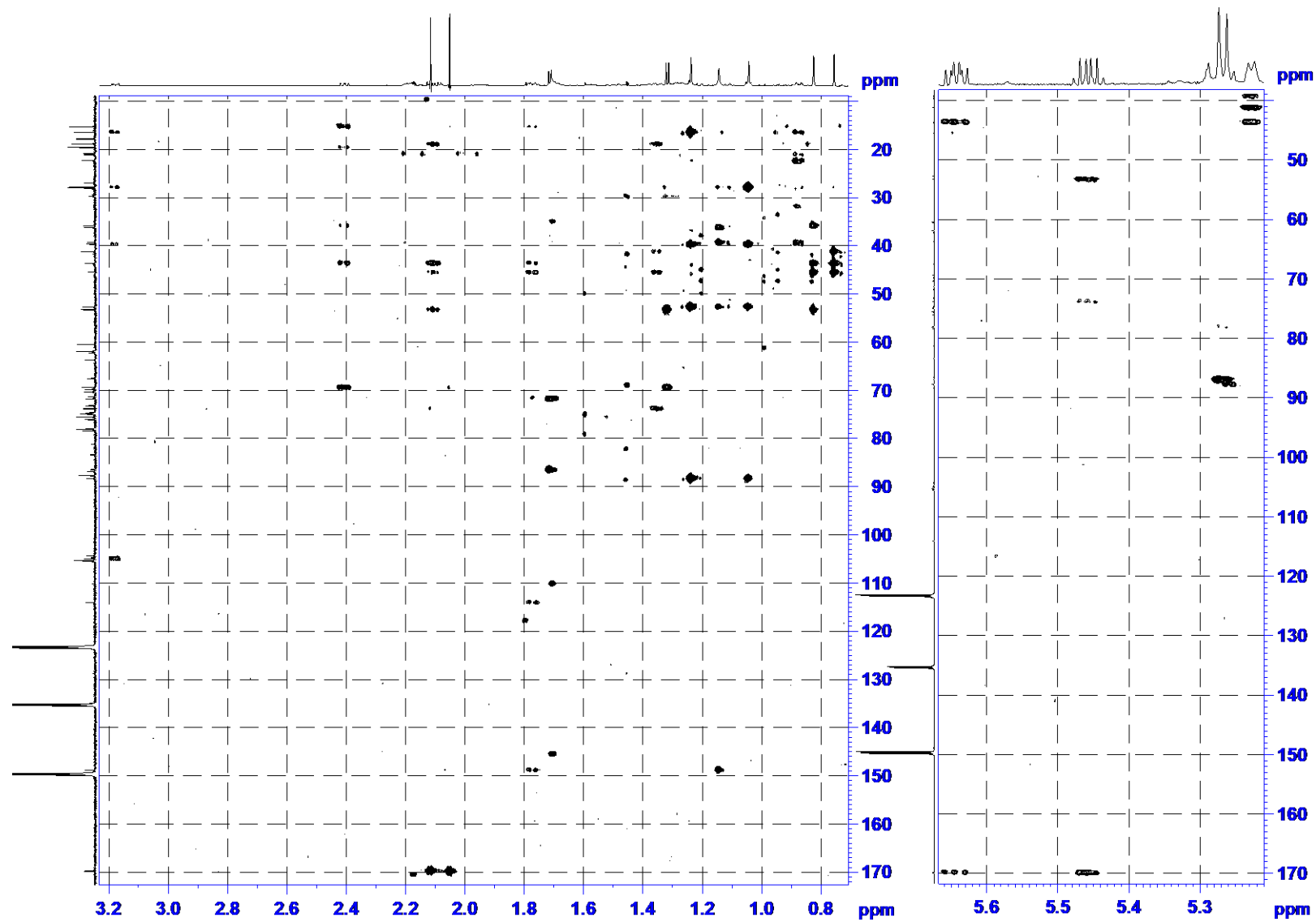


Figure S6. The HMBC (700.00 MHz) spectrum of the aglycone part of kuriloside A<sub>1</sub> (**1**) in C<sub>5</sub>D<sub>5</sub>N/D<sub>2</sub>O (4/1)

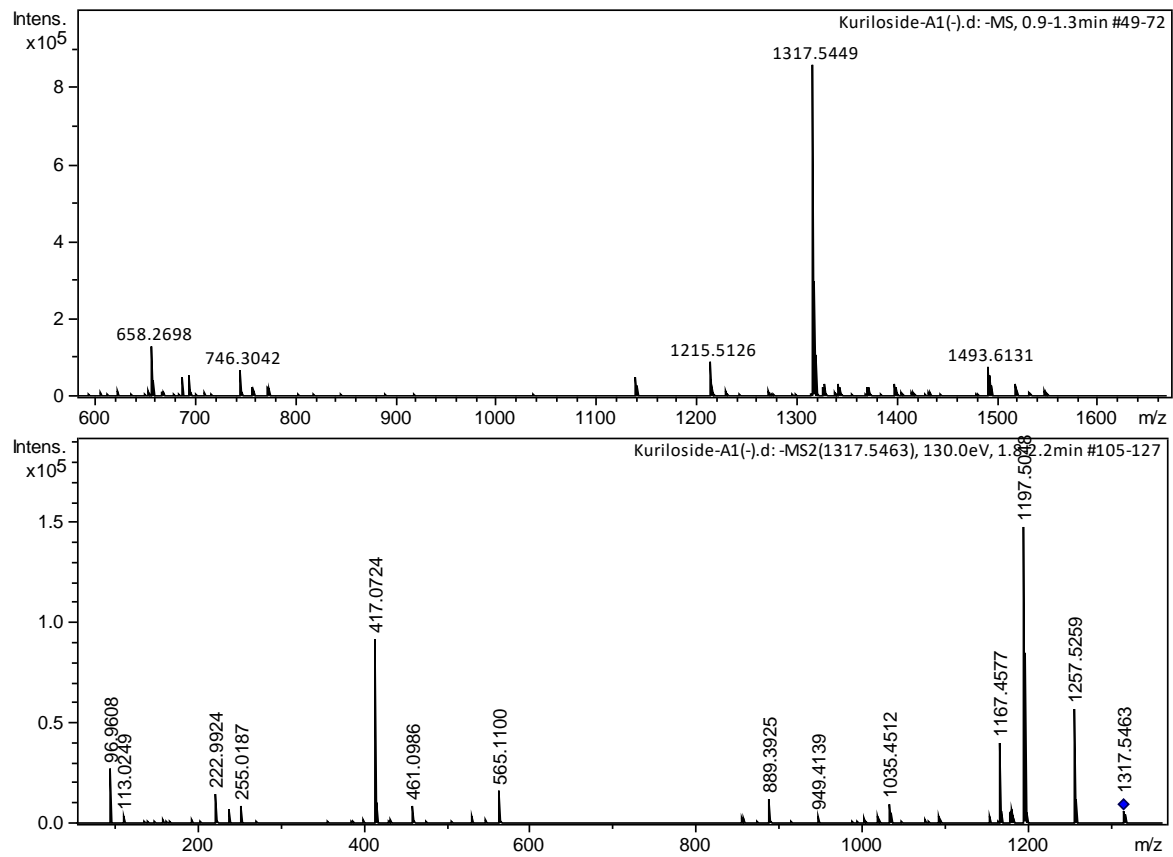


Figure S7. HR-ESI-MS and ESI-MS/MS spectra of kuriloside A<sub>1</sub> (**1**)

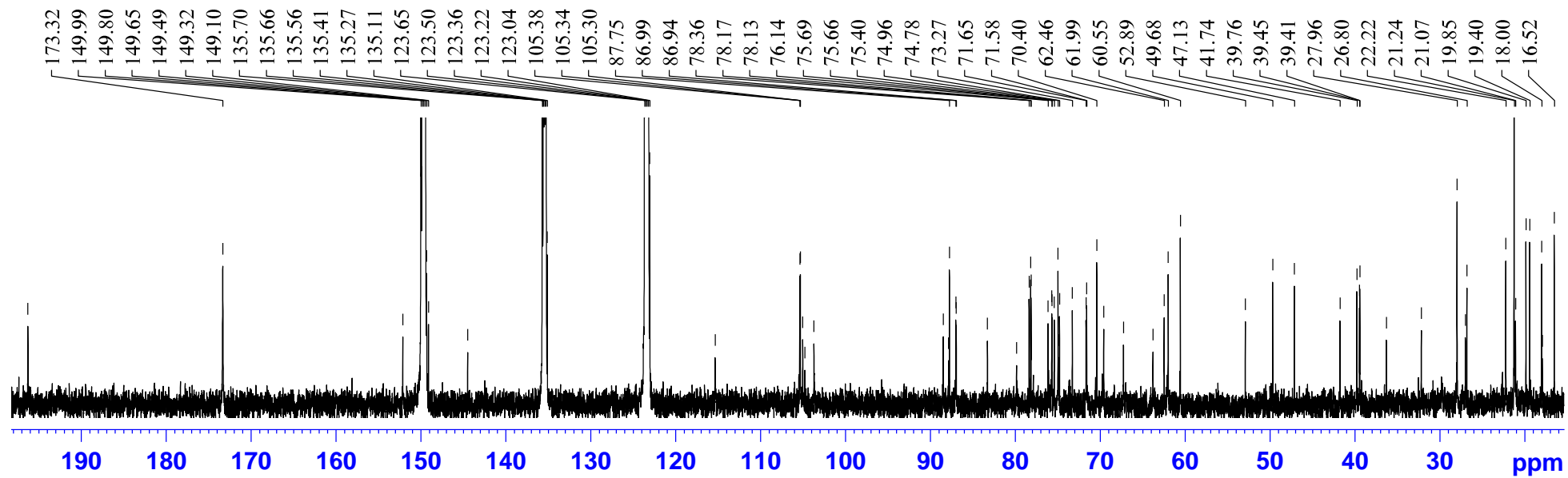


Figure S8. The  $^{13}\text{C}$  NMR (176.03 MHz) spectrum of kuriloside  $\text{A}_2$  (**2**) in  $\text{C}_5\text{D}_5\text{N}/\text{D}_2\text{O}$  (4/1)

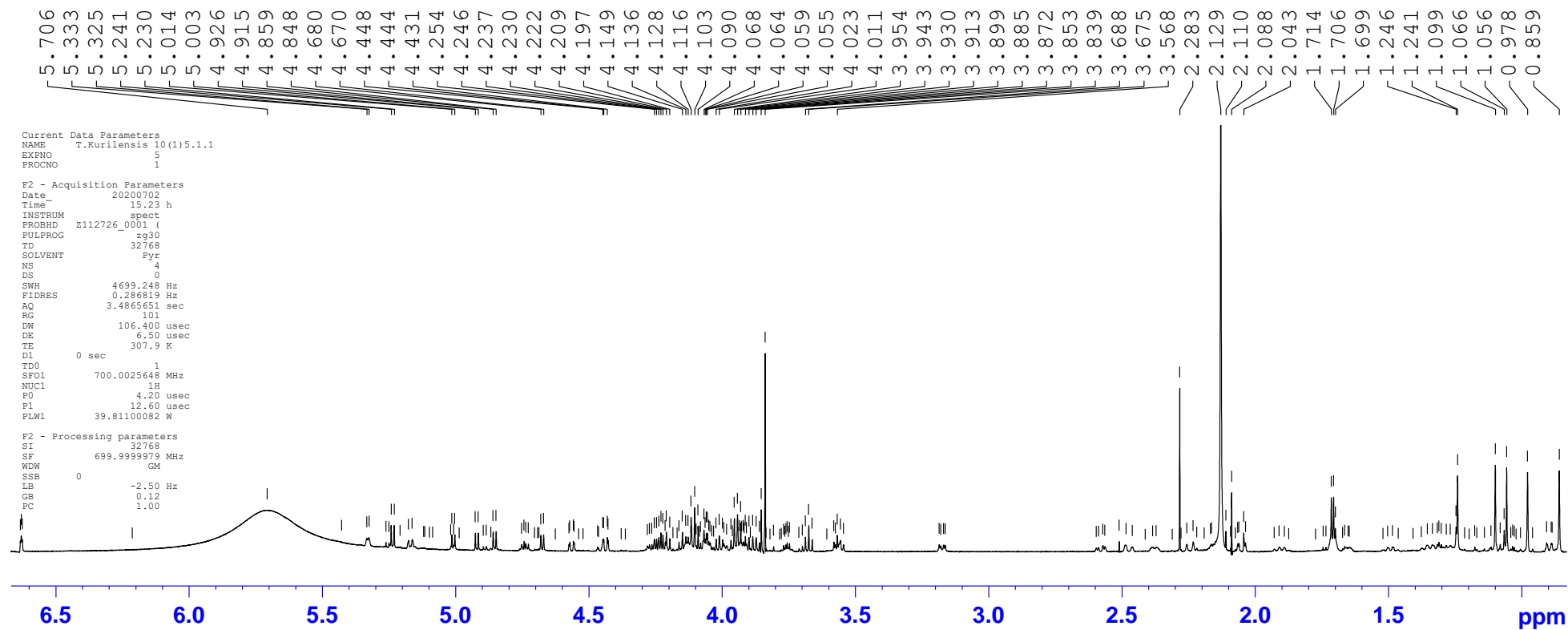


Figure S9. The  $^1\text{H}$  NMR (700.00 MHz) spectrum of kuriloside A<sub>2</sub> (2) in  $\text{C}_5\text{D}_5\text{N}/\text{D}_2\text{O}$  (4/1)

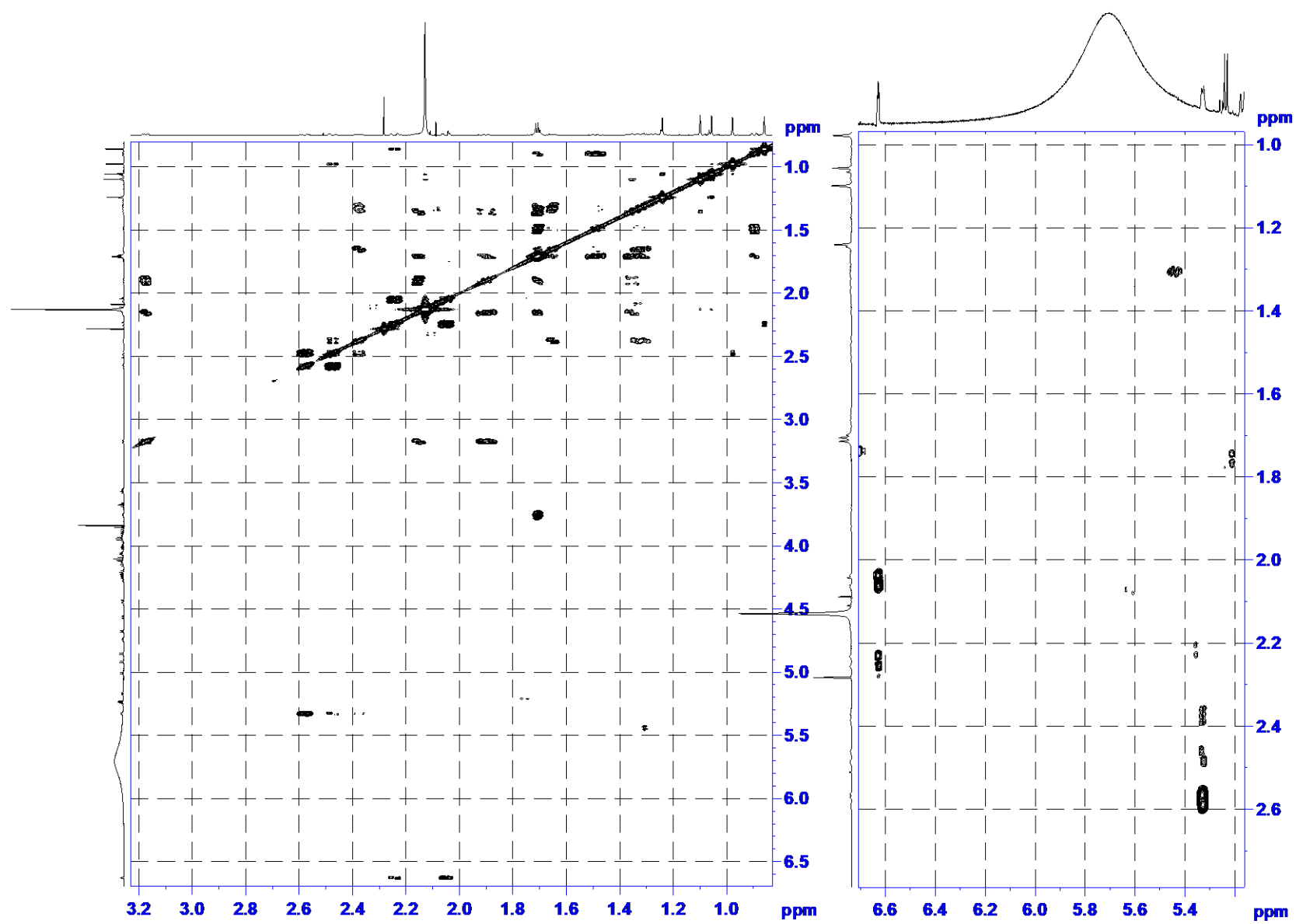


Figure S10. The COSY (700.00 MHz) spectrum of the aglycone part of kuriloside A<sub>2</sub> (**2**) in C<sub>5</sub>D<sub>5</sub>N/D<sub>2</sub>O (4/1)

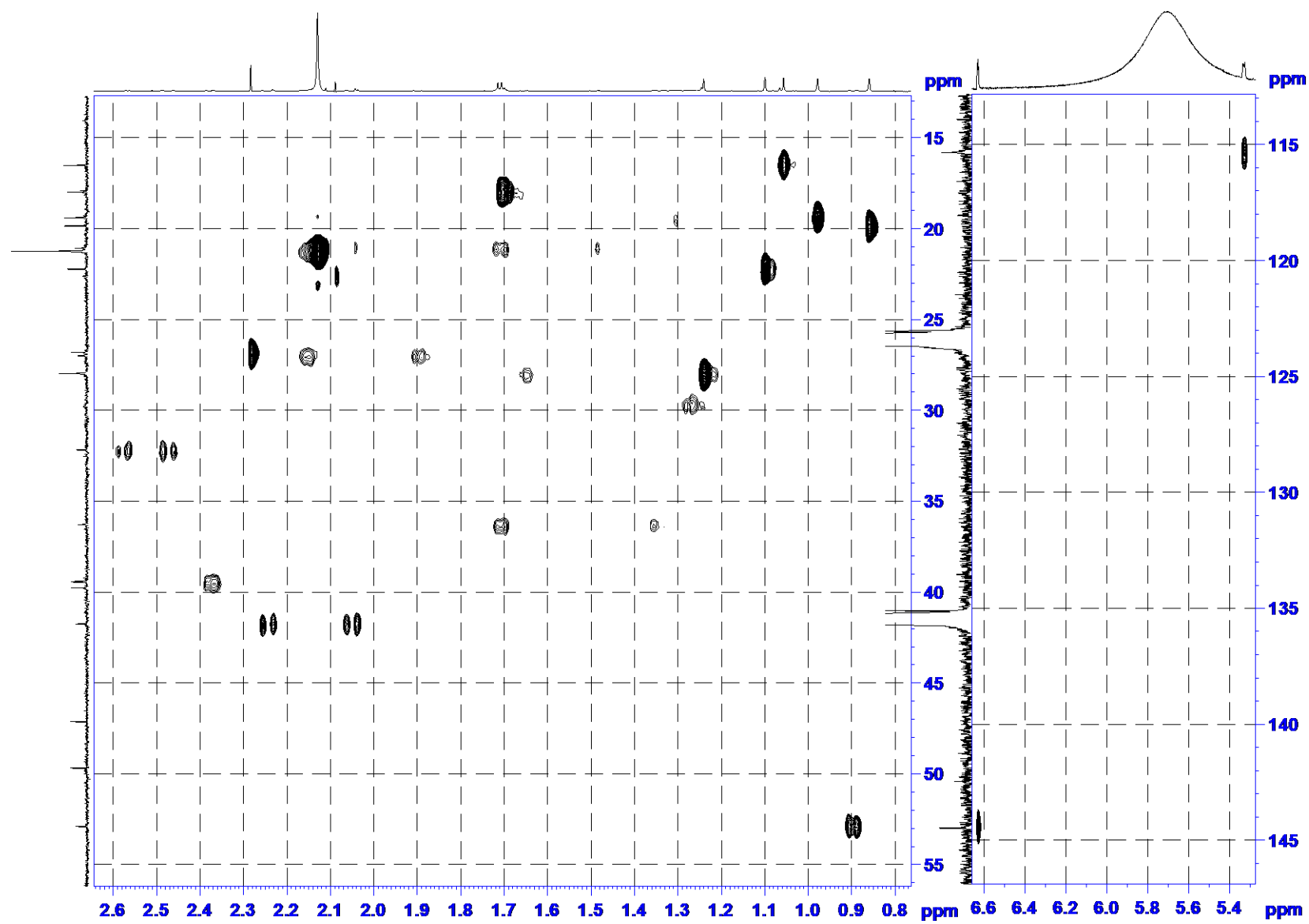


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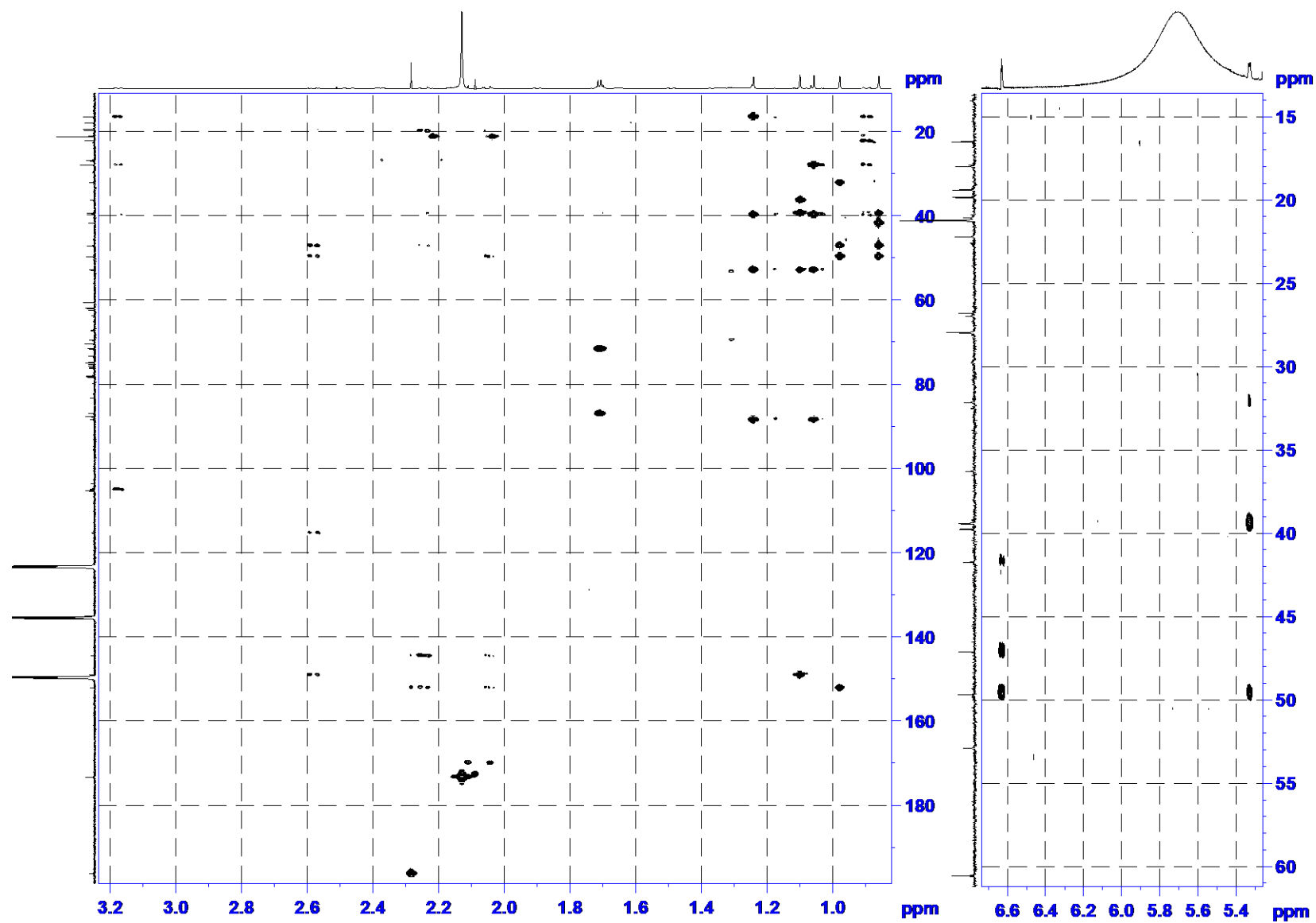


Figure S12. The HMBC (700.00 MHz) spectrum of the aglycone part of kuriloside A<sub>2</sub> (2) in C<sub>5</sub>D<sub>5</sub>N/D<sub>2</sub>O (4/1)



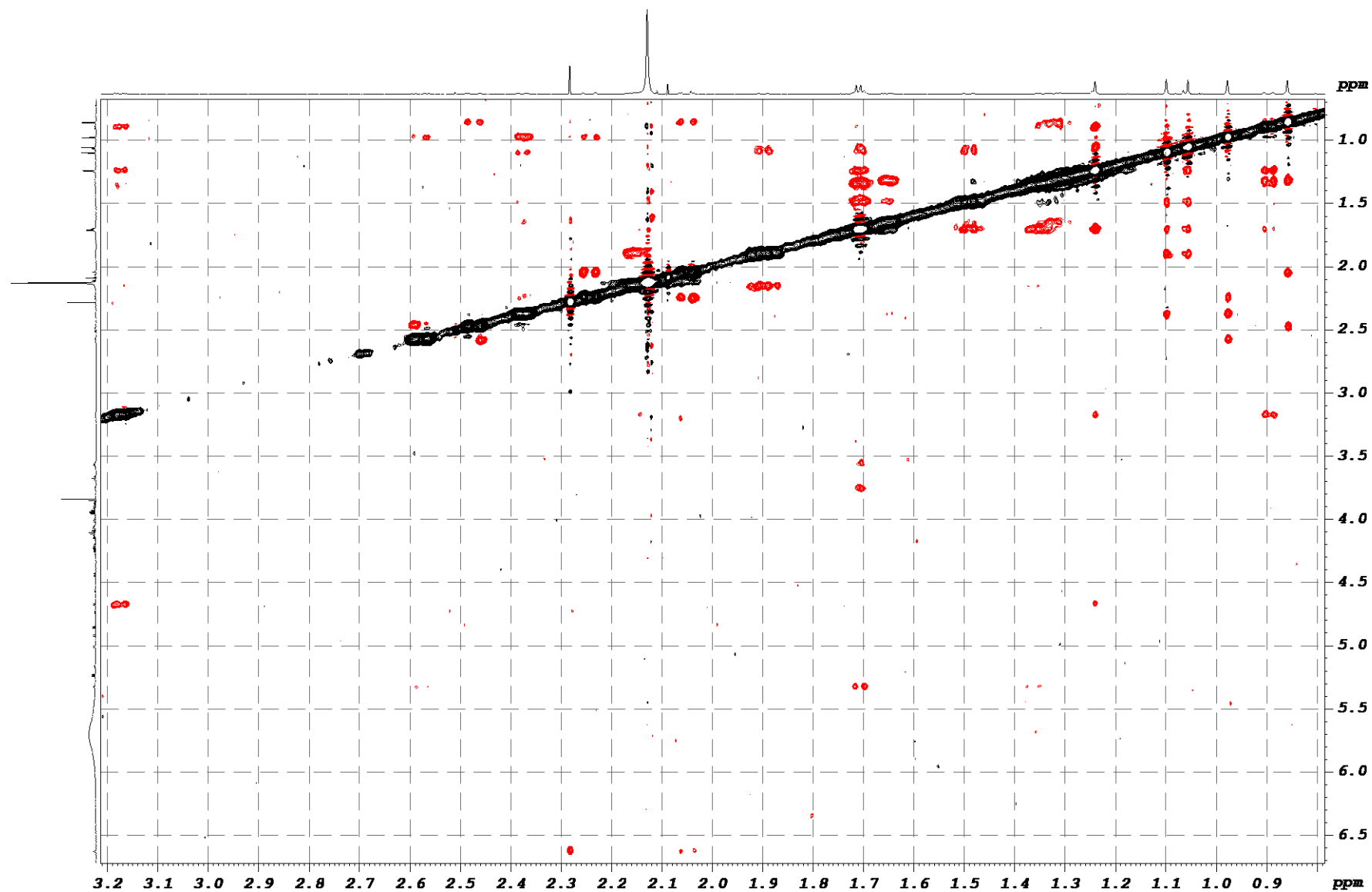


Figure S13. The ROESY (700.00 MHz) spectrum of the aglycone part of kuriloside A<sub>2</sub> (2) in C<sub>5</sub>D<sub>5</sub>N/D<sub>2</sub>O (4/1)

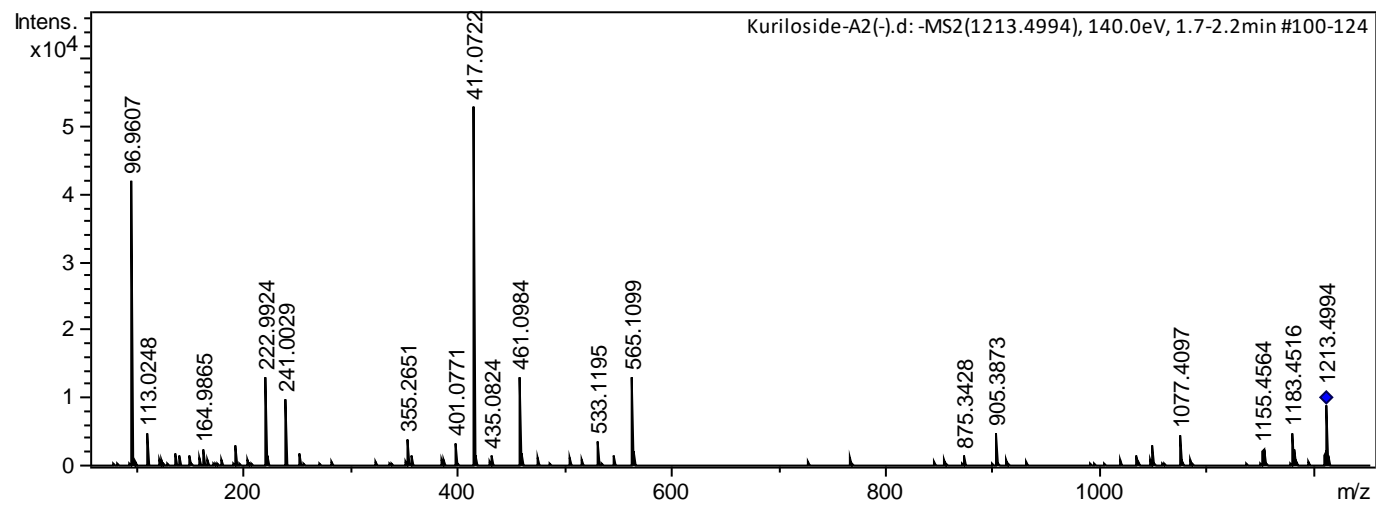
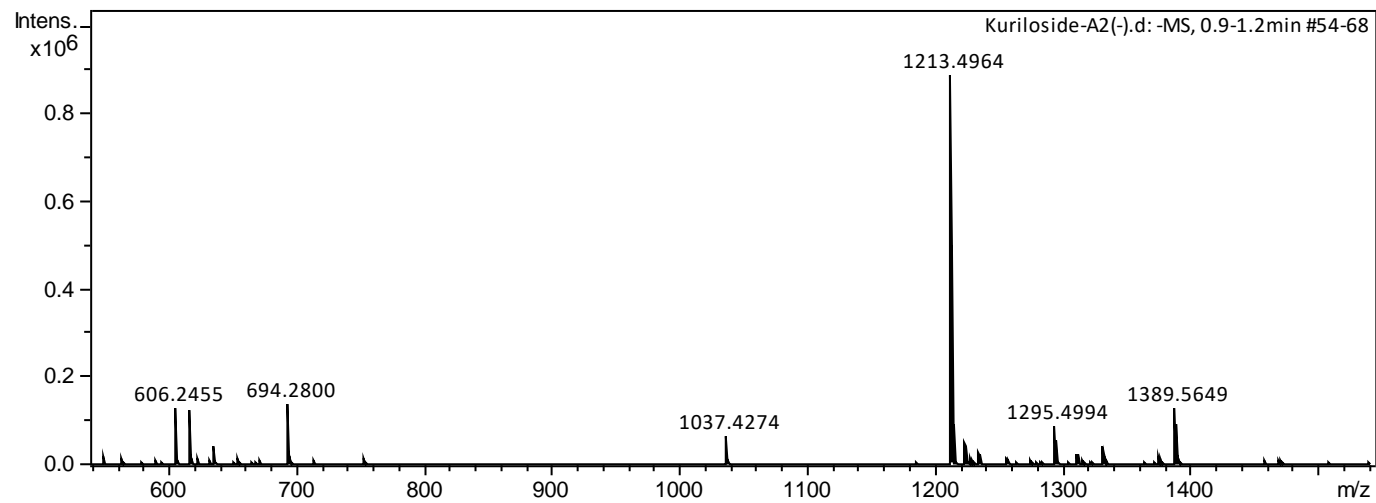


Figure S14. HR-ESI-MS and ESI-MS/MS spectra of kuriloside A<sub>2</sub> (2)

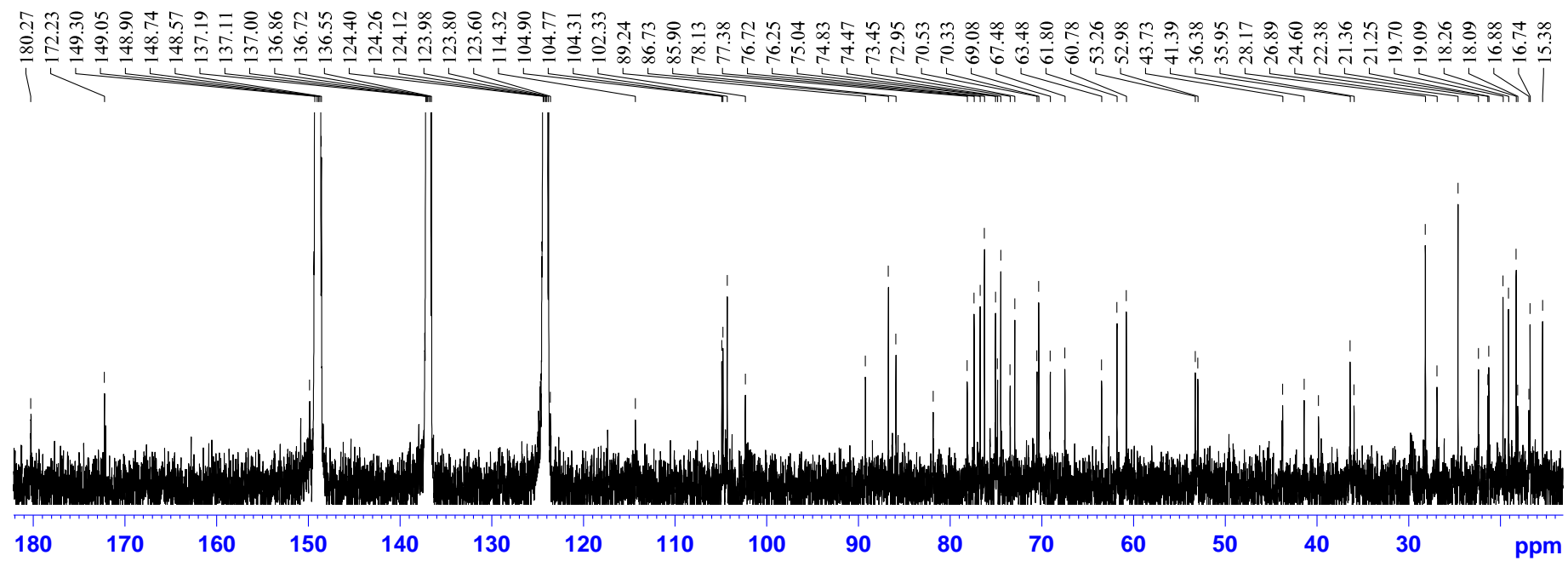


Figure S15. The  $^{13}\text{C}$  NMR (176.03 MHz) spectrum of kuriloside  $\text{C}_1$  (**3**) in  $\text{C}_5\text{D}_5\text{N}/\text{D}_2\text{O}$  (4/1)

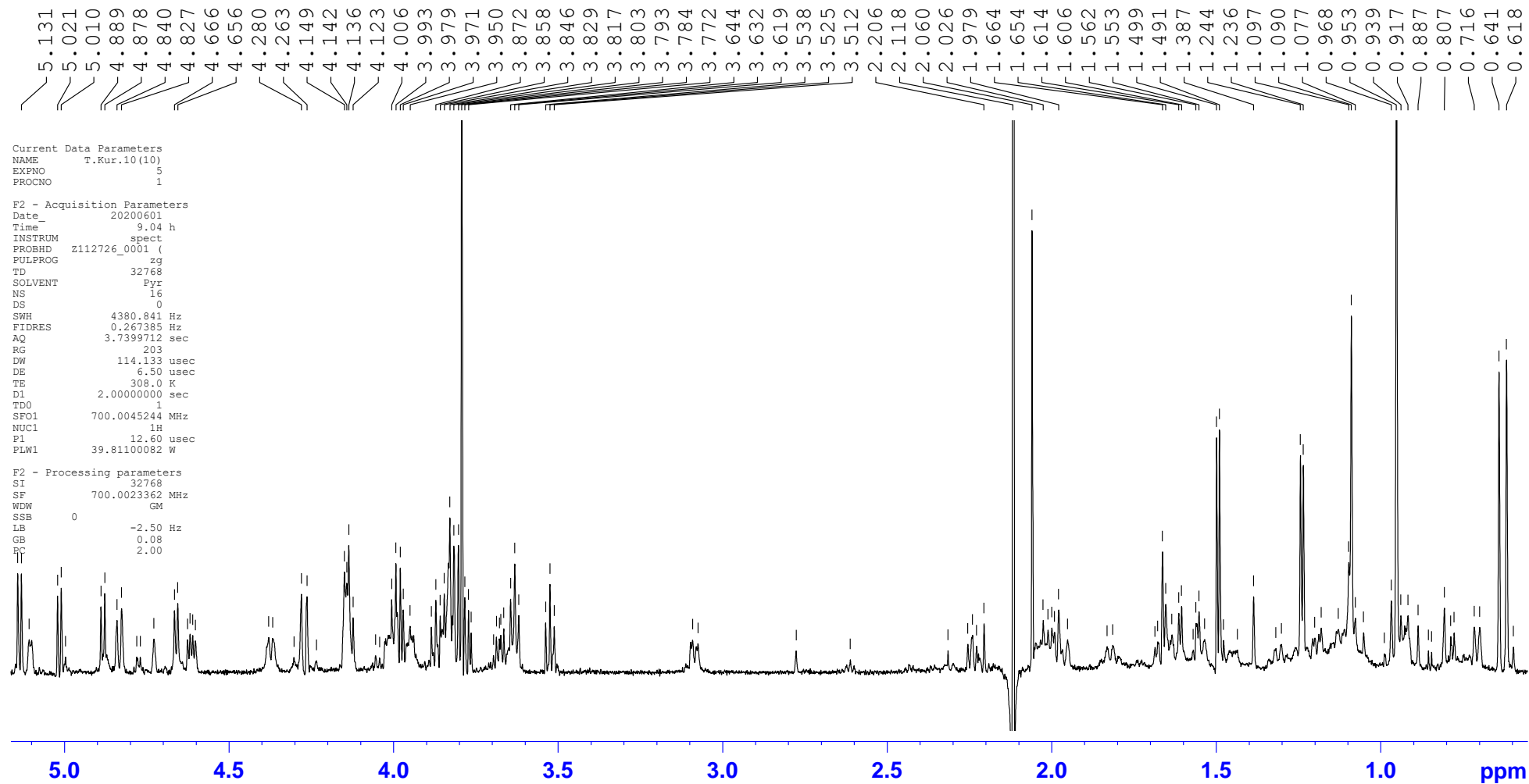


Figure S16. The  $^1\text{H}$  NMR (700.00 MHz) spectrum of kuriloside  $\text{C}_1$  (**3**) in  $\text{C}_5\text{D}_5\text{N}/\text{D}_2\text{O}$  (4/1)

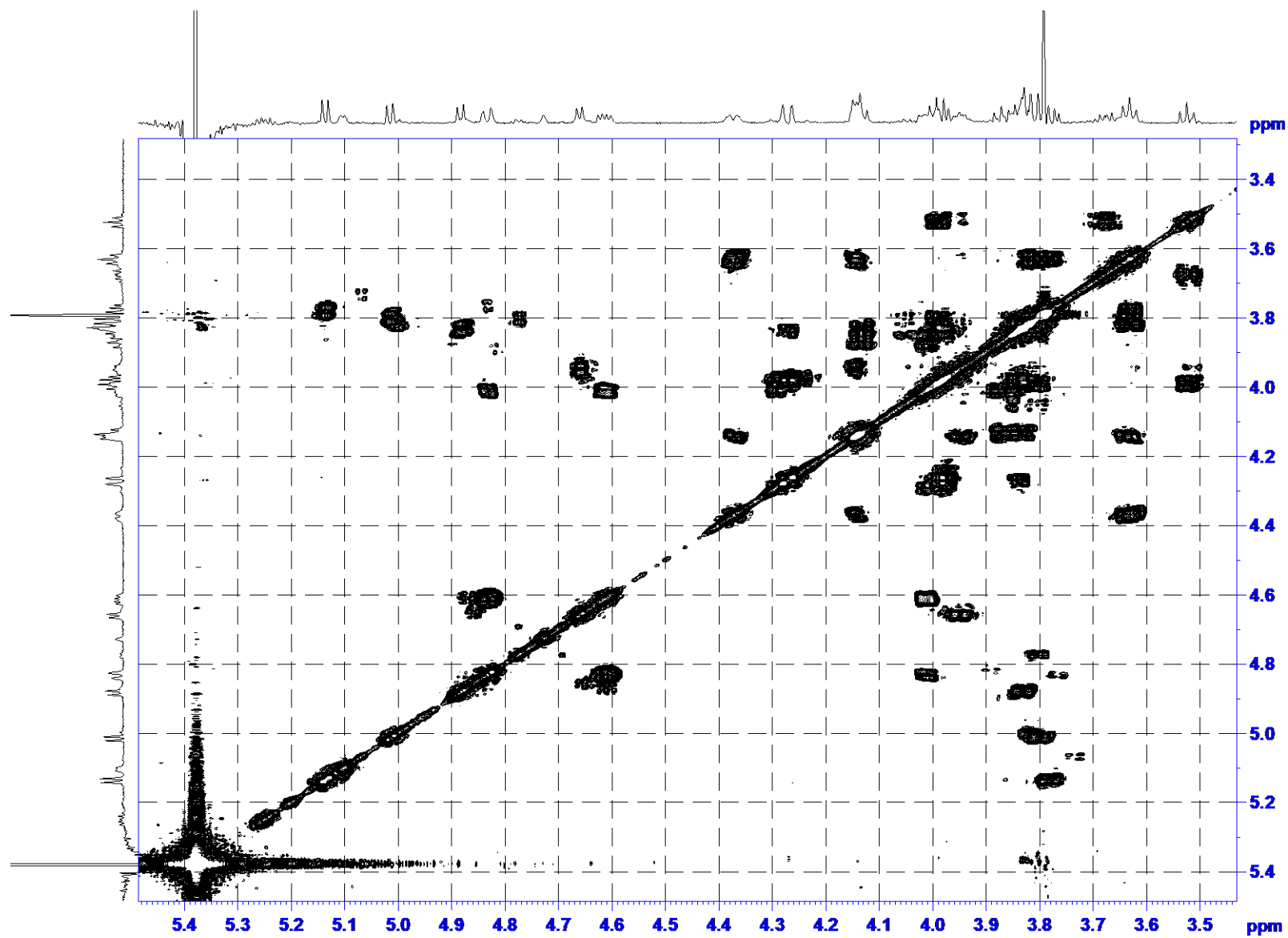


Figure S17. The COSY (700.00 MHz) spectrum of the carbohydrate part of kuriloside C<sub>1</sub> (**3**) in C<sub>5</sub>D<sub>5</sub>N/D<sub>2</sub>O (4/1)

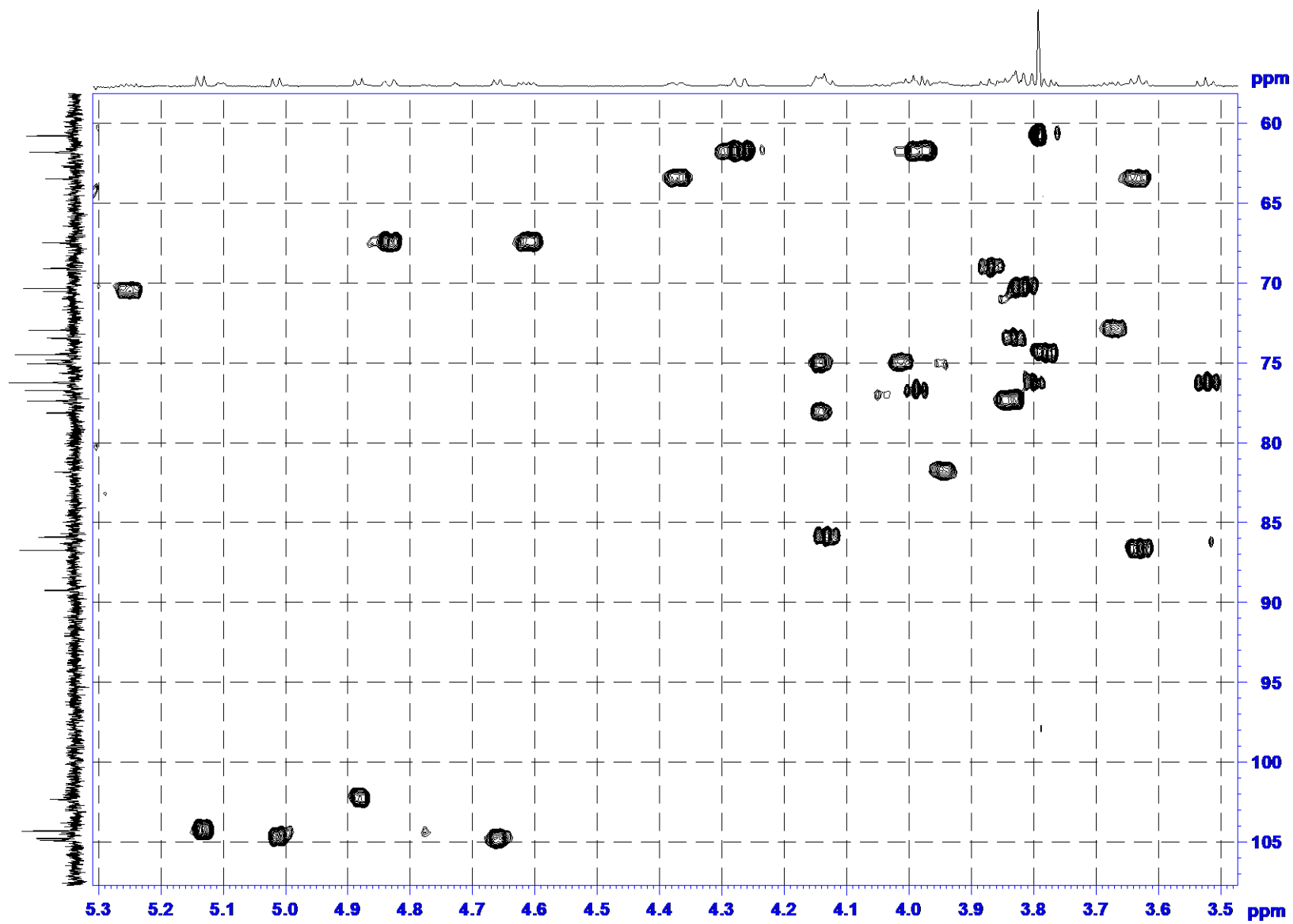


Figure S18. The HSQC (700.00 MHz) spectrum of the carbohydrate part of kuriloside  $\text{C}_1$  (3) in  $\text{C}_5\text{D}_5\text{N}/\text{D}_2\text{O}$  (4/1)

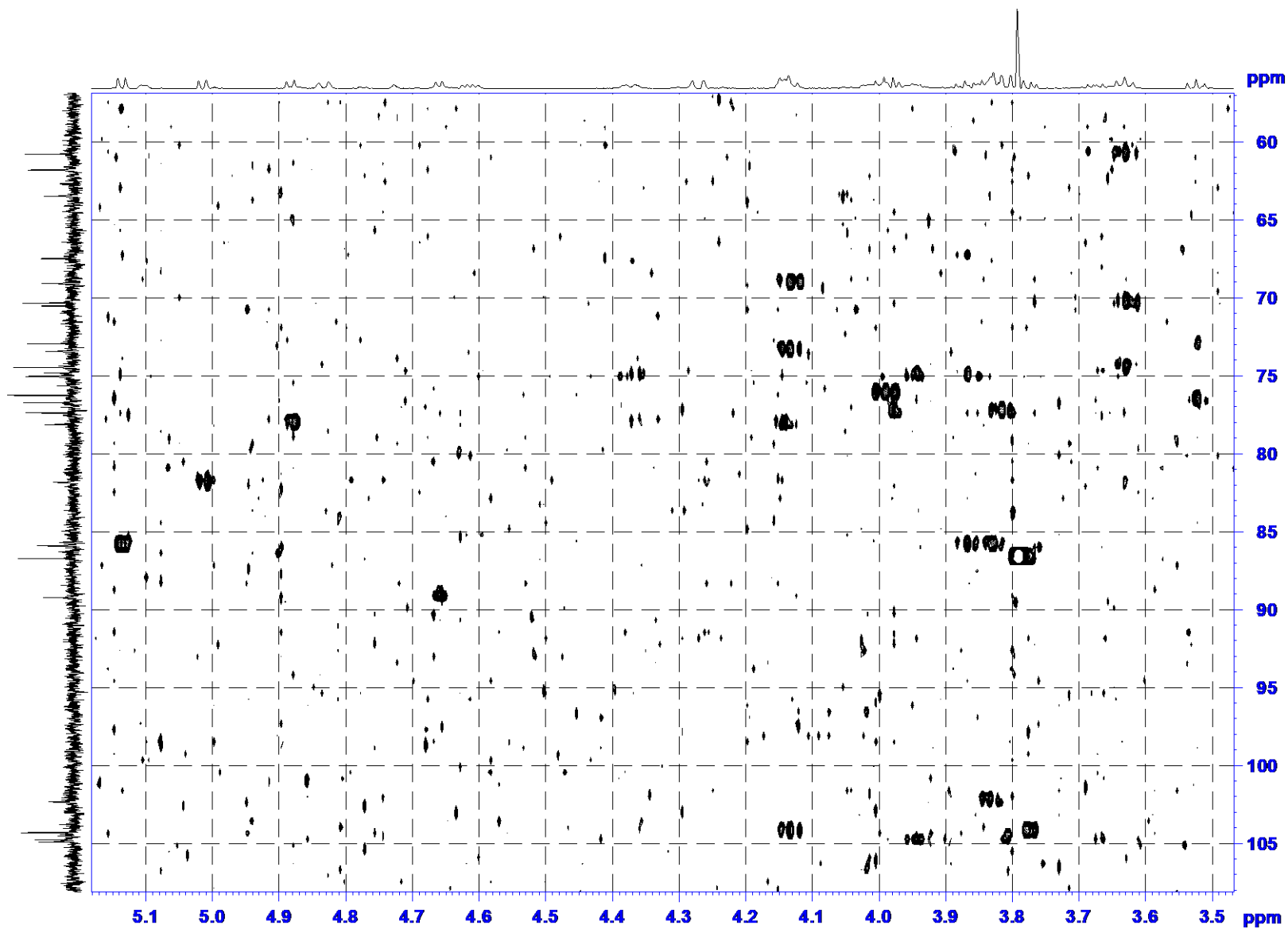


Figure S19. The HMBC (700.00 MHz) spectrum of the carbohydrate part of kuriloside C<sub>1</sub> (3) in C<sub>5</sub>D<sub>5</sub>N/D<sub>2</sub>O (4/1)

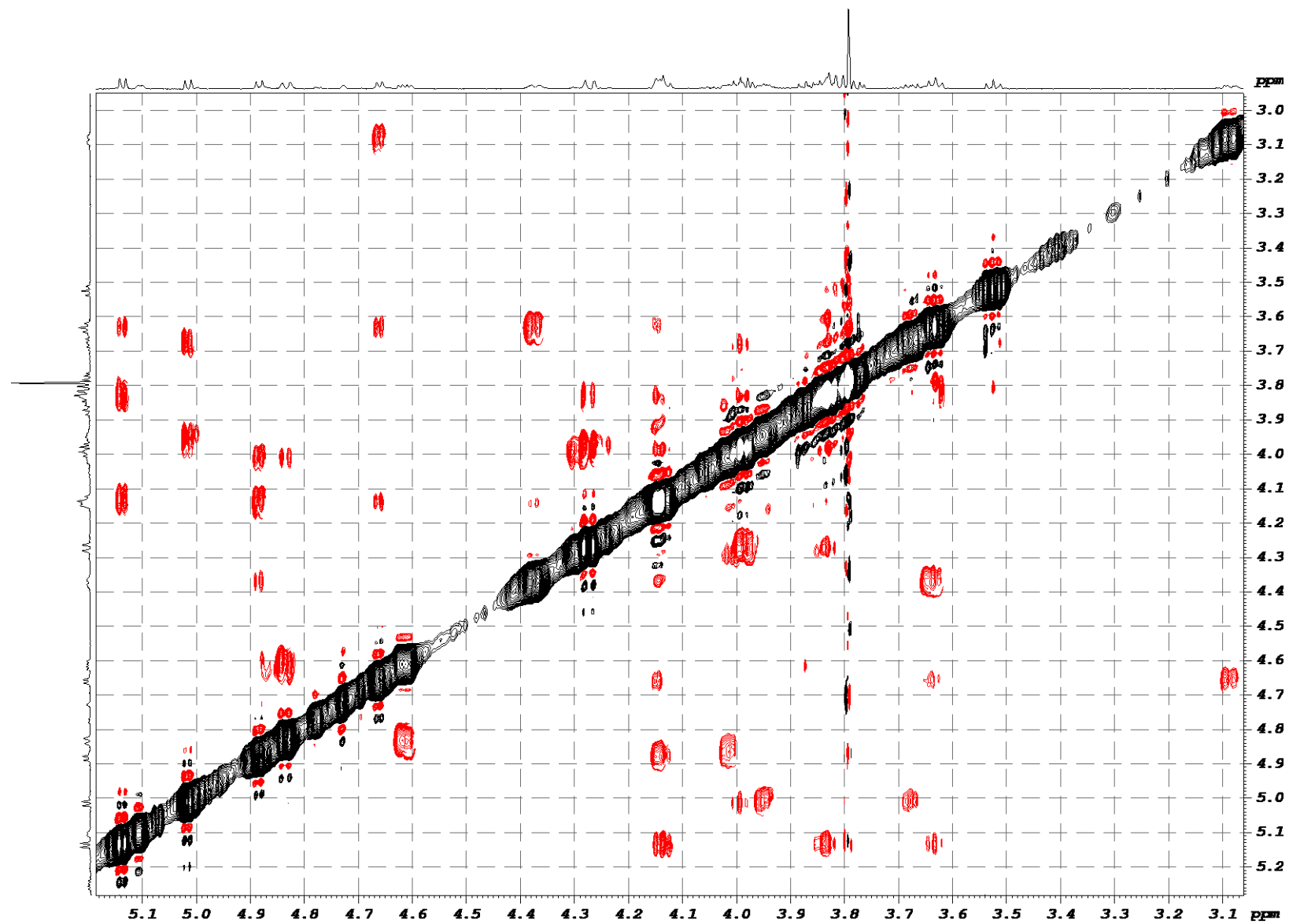


Figure S20. The ROESY (700.00 MHz) spectrum of the carbohydrate part of kuriloside C<sub>1</sub> (3) in C<sub>5</sub>D<sub>5</sub>N/D<sub>2</sub>O (4/1)



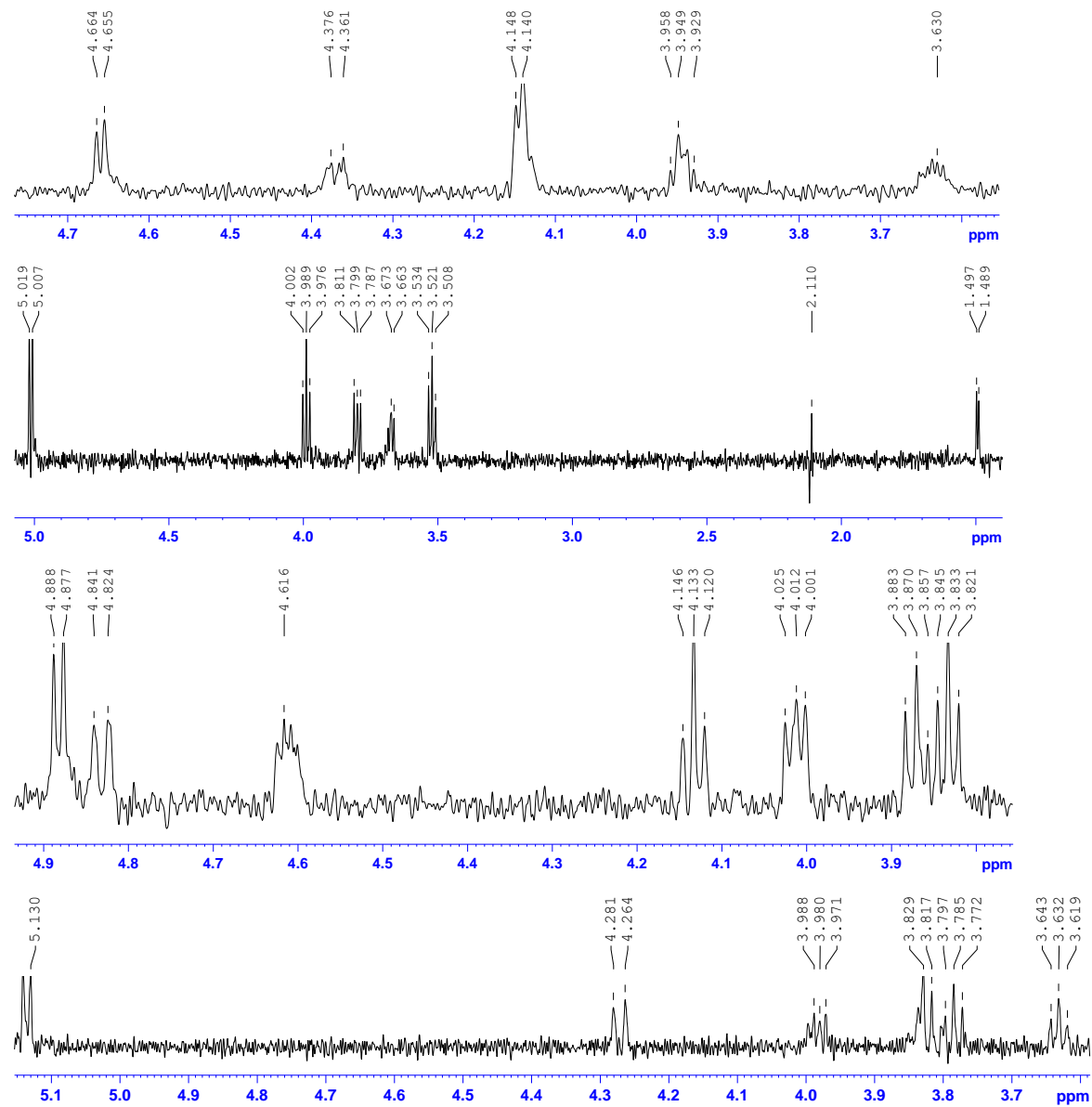


Figure S21. 1 D TOCSY (700.00 MHz) spectra of the carbohydrate part of kuriloside C<sub>1</sub> (**3**) in C<sub>5</sub>D<sub>5</sub>N/D<sub>2</sub>O (4/1)

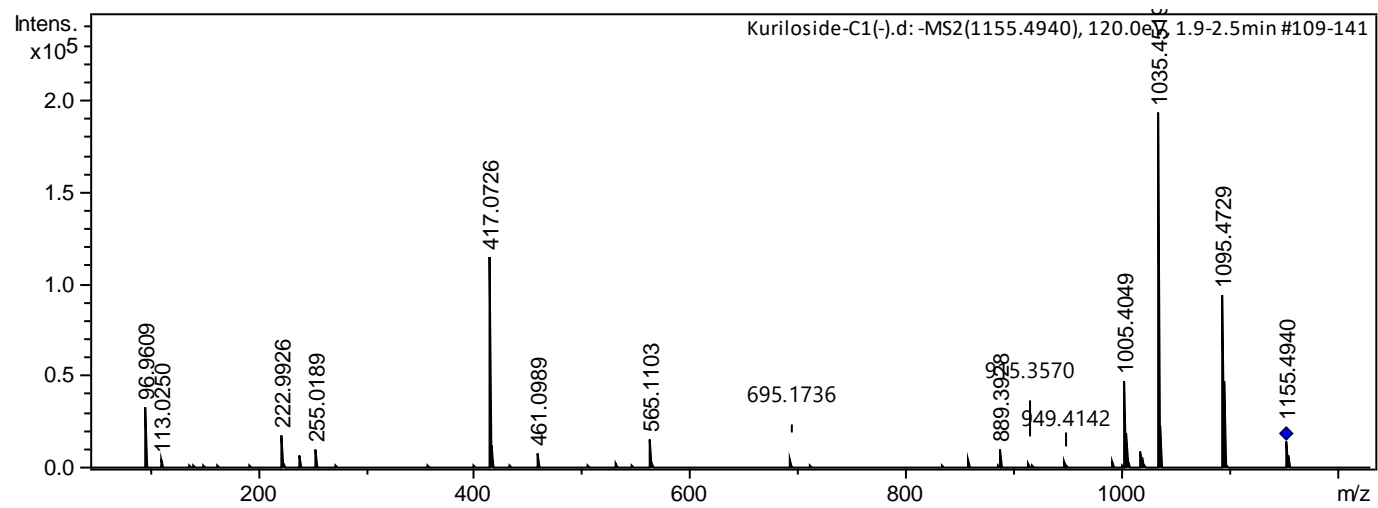
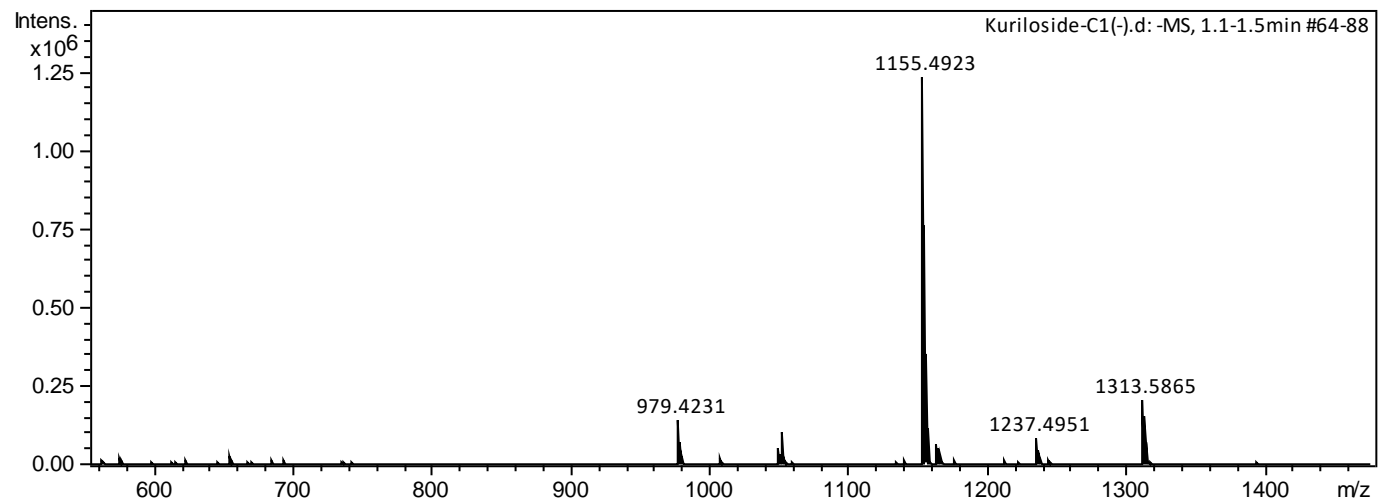


Figure S22. HR-ESI-MS and ESI-MS/MS spectra of kurilosiide C<sub>1</sub> (3)

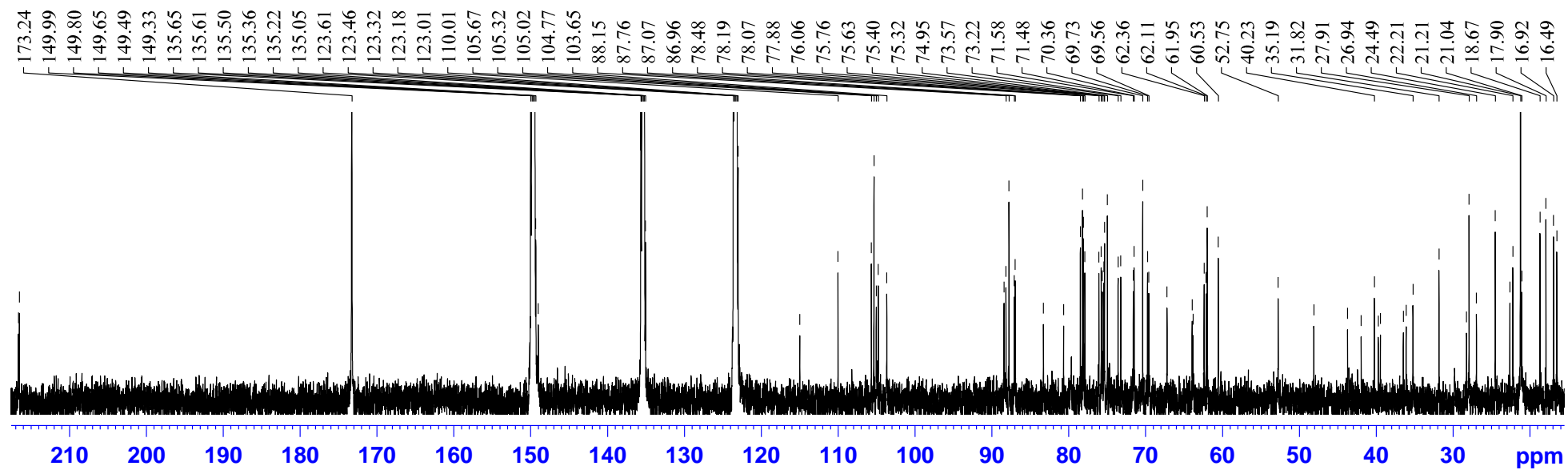


Figure S23. The  $^{13}\text{C}$  NMR (176.03 MHz) spectrum of kuriloside D (4) in  $\text{C}_5\text{D}_5\text{N}/\text{D}_2\text{O}$  (4/1)

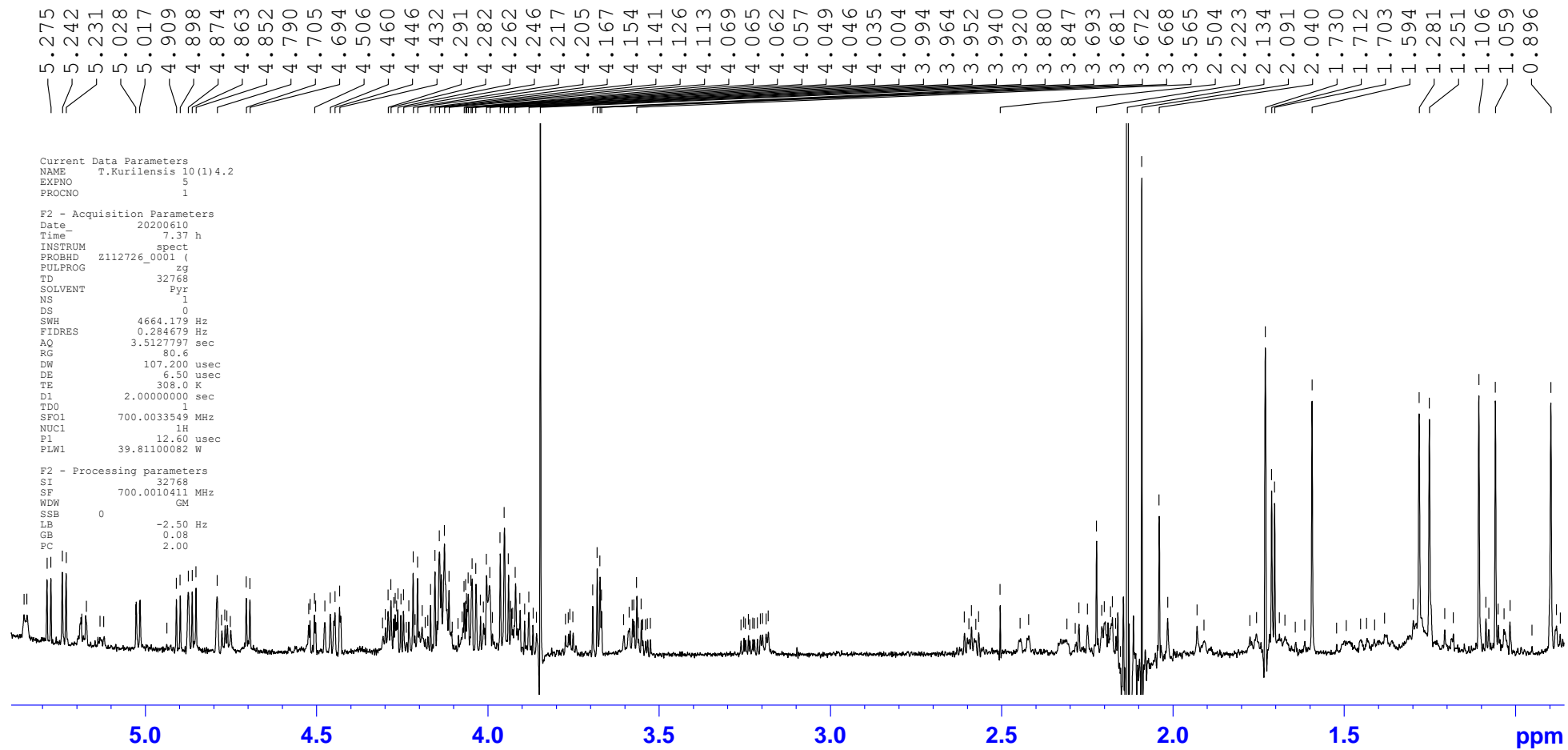


Figure S24. The  $^1\text{H}$  NMR (700.00 MHz) spectrum of kurilaside D (**4**) in  $\text{C}_5\text{D}_5\text{N}/\text{D}_2\text{O}$  (4/1)

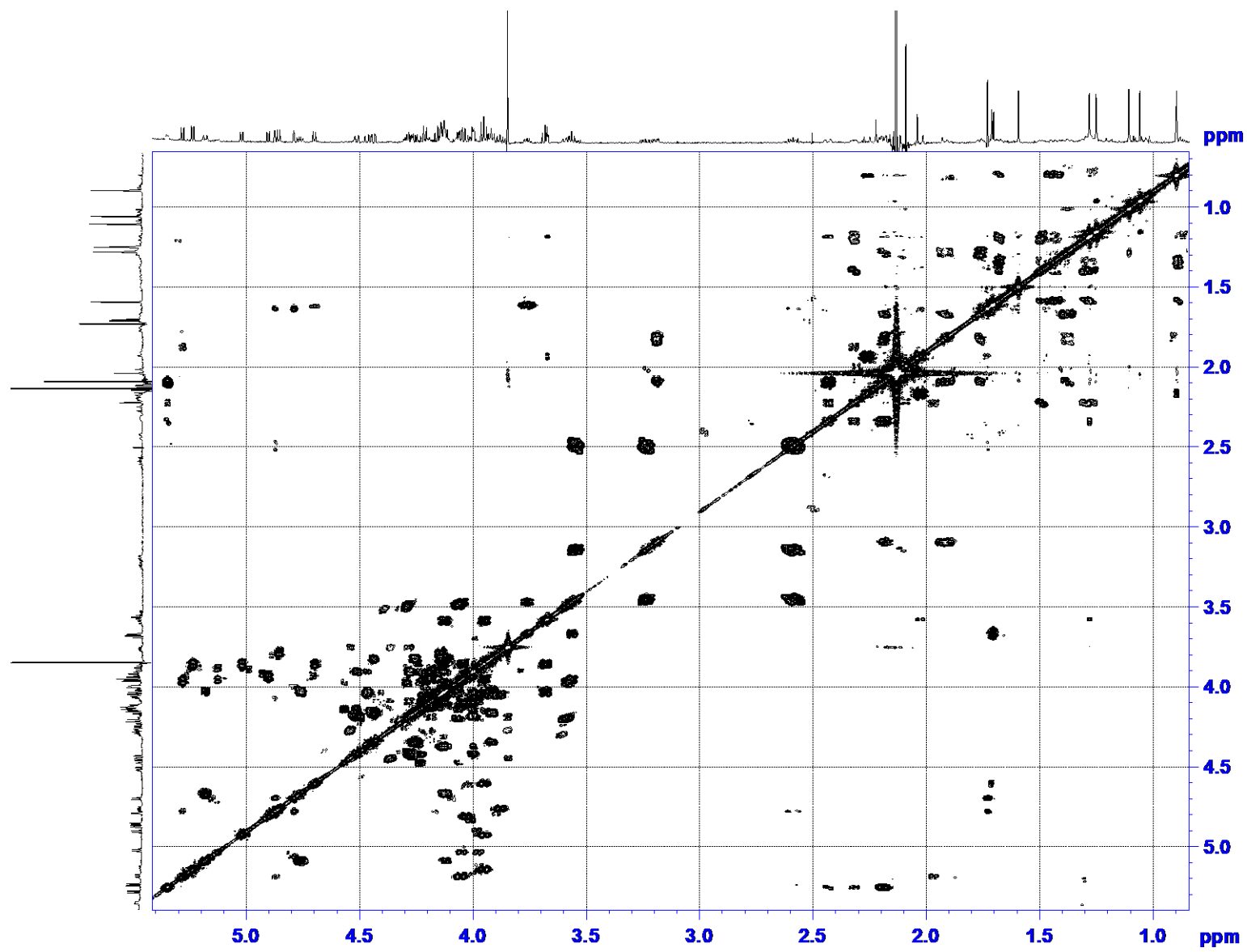


Figure S25. The COSY (700.00 MHz) spectrum of kuriloside D (**4**) in  $C_5D_5N/D_2O$  (4/1)

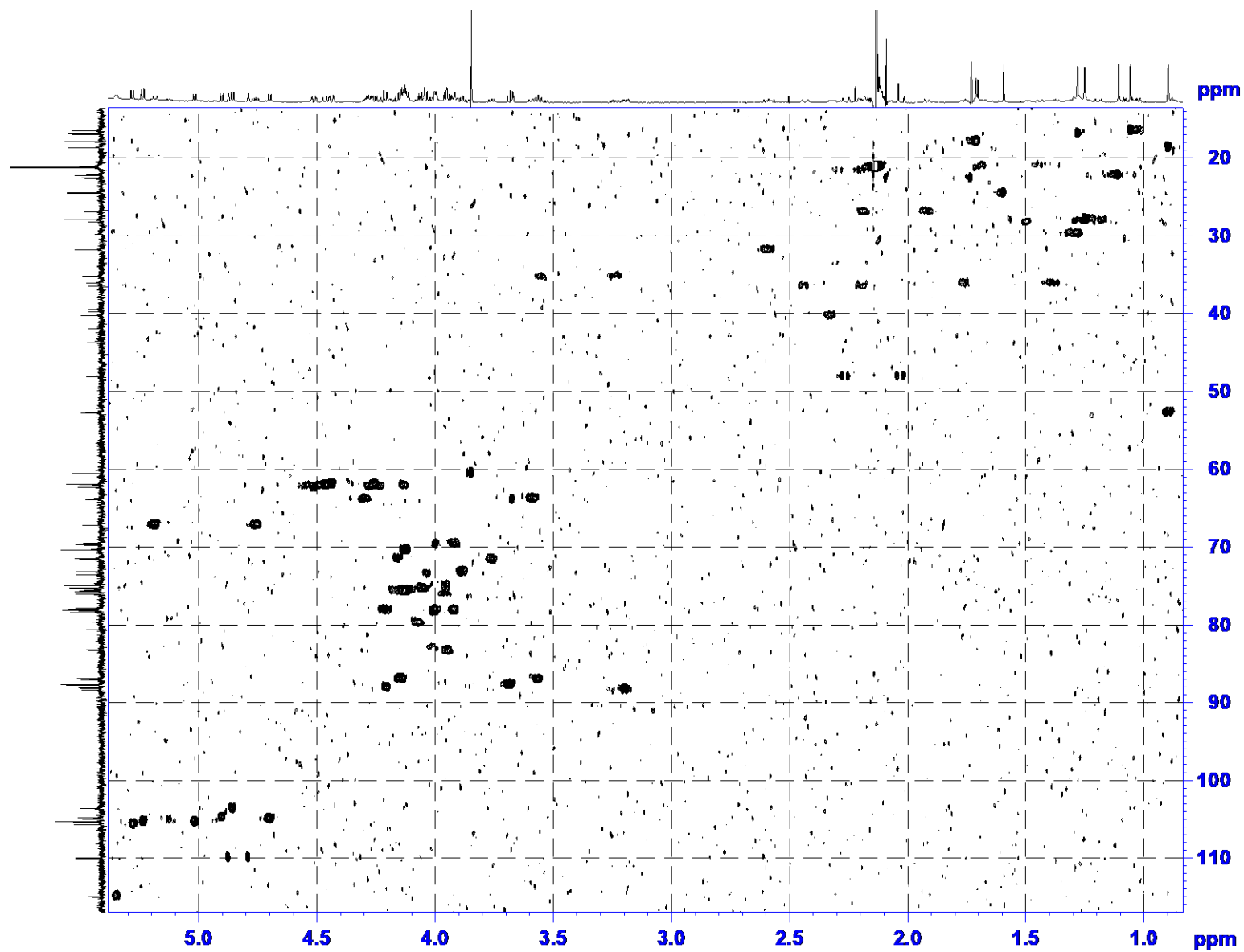


Figure S26. The HSQC (700.00 MHz) spectrum of kuriloside D (4) in C<sub>5</sub>D<sub>5</sub>N/D<sub>2</sub>O (4/1)

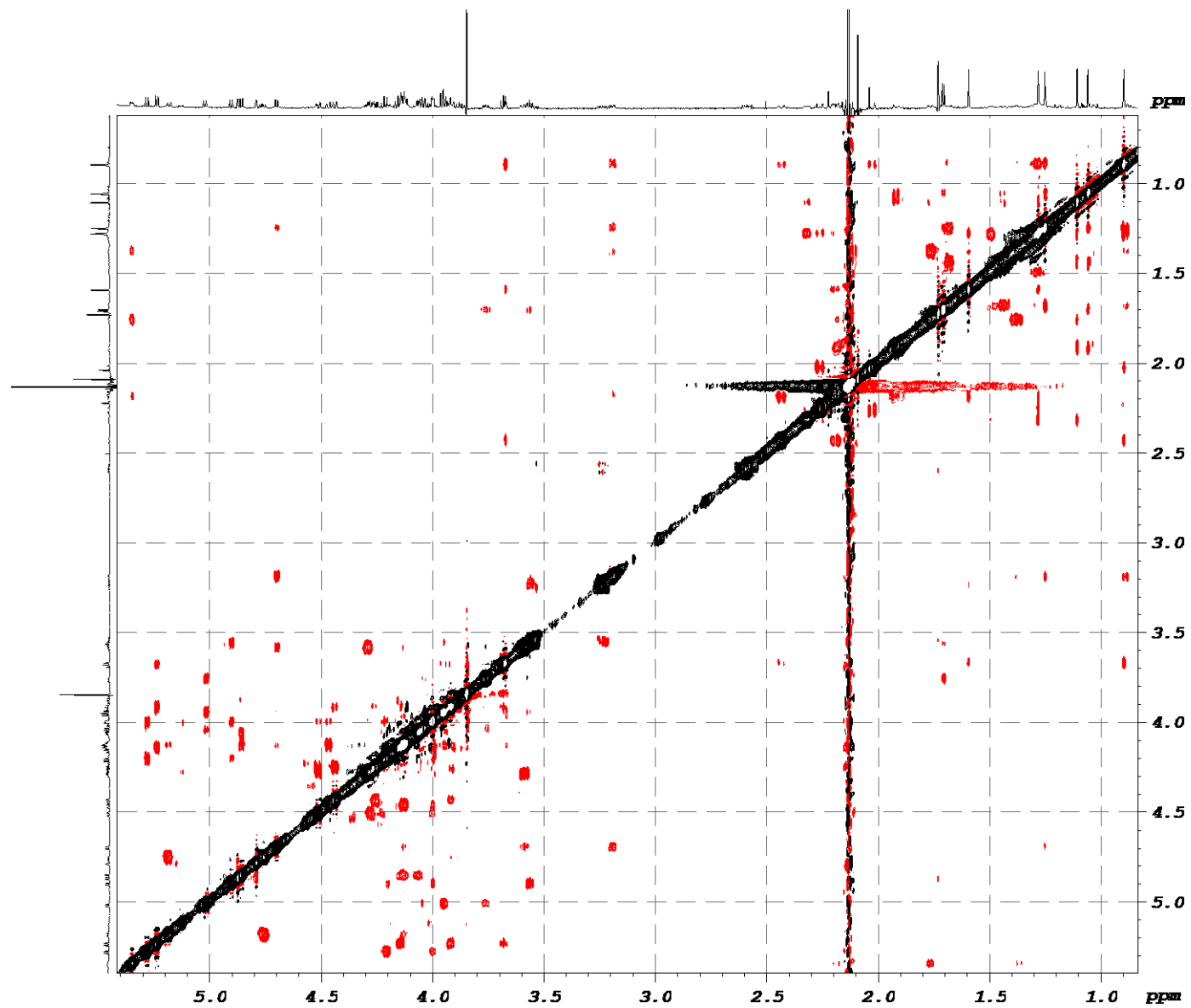


Figure S27. The ROESY (700.00 MHz) spectrum of kuriloside D (**4**) in C<sub>5</sub>D<sub>5</sub>N/D<sub>2</sub>O (4/1)

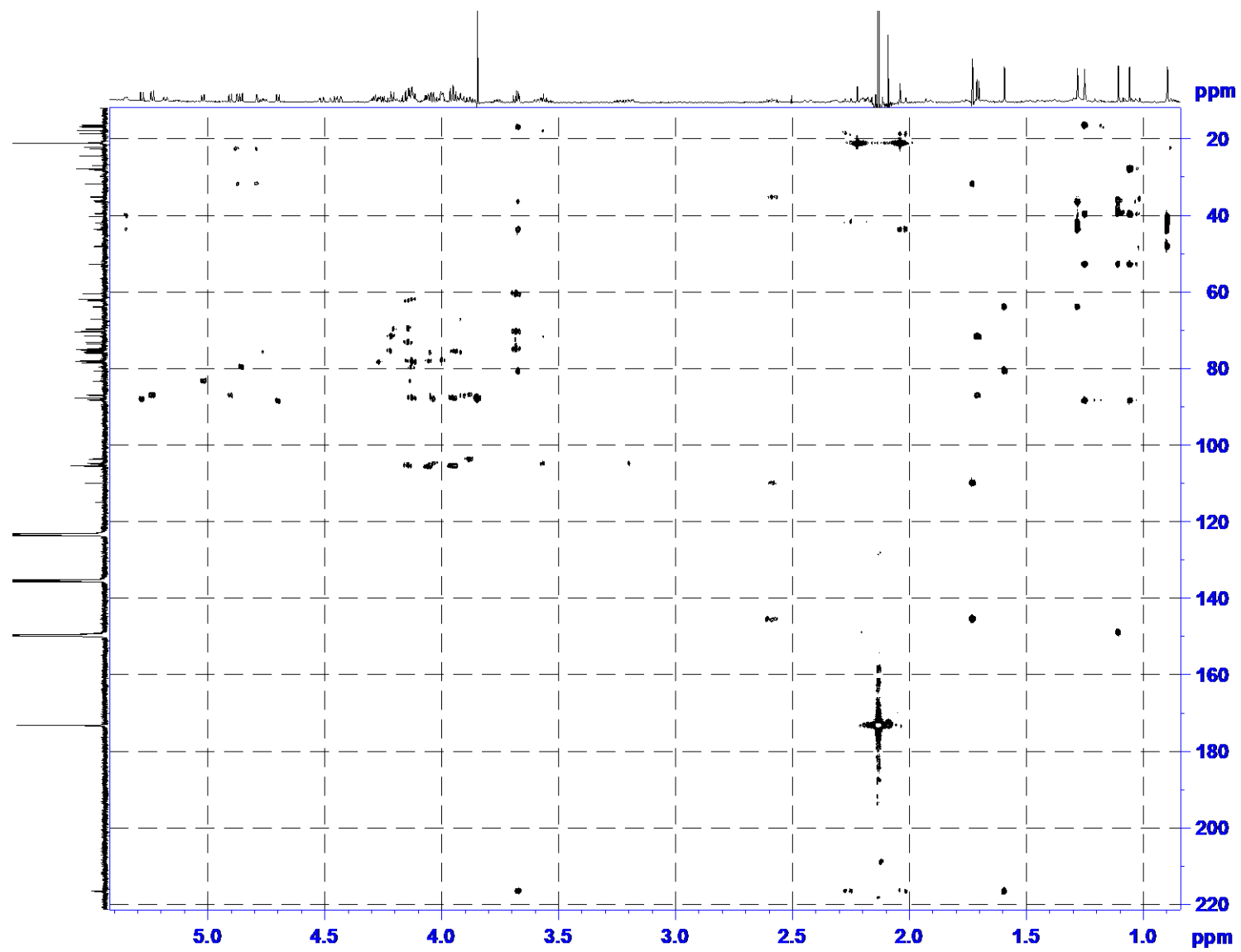


Figure S28. The HMBC (700.00 MHz) spectrum of kuriloside D (4) in  $\text{C}_5\text{D}_5\text{N}/\text{D}_2\text{O}$  (4/1)



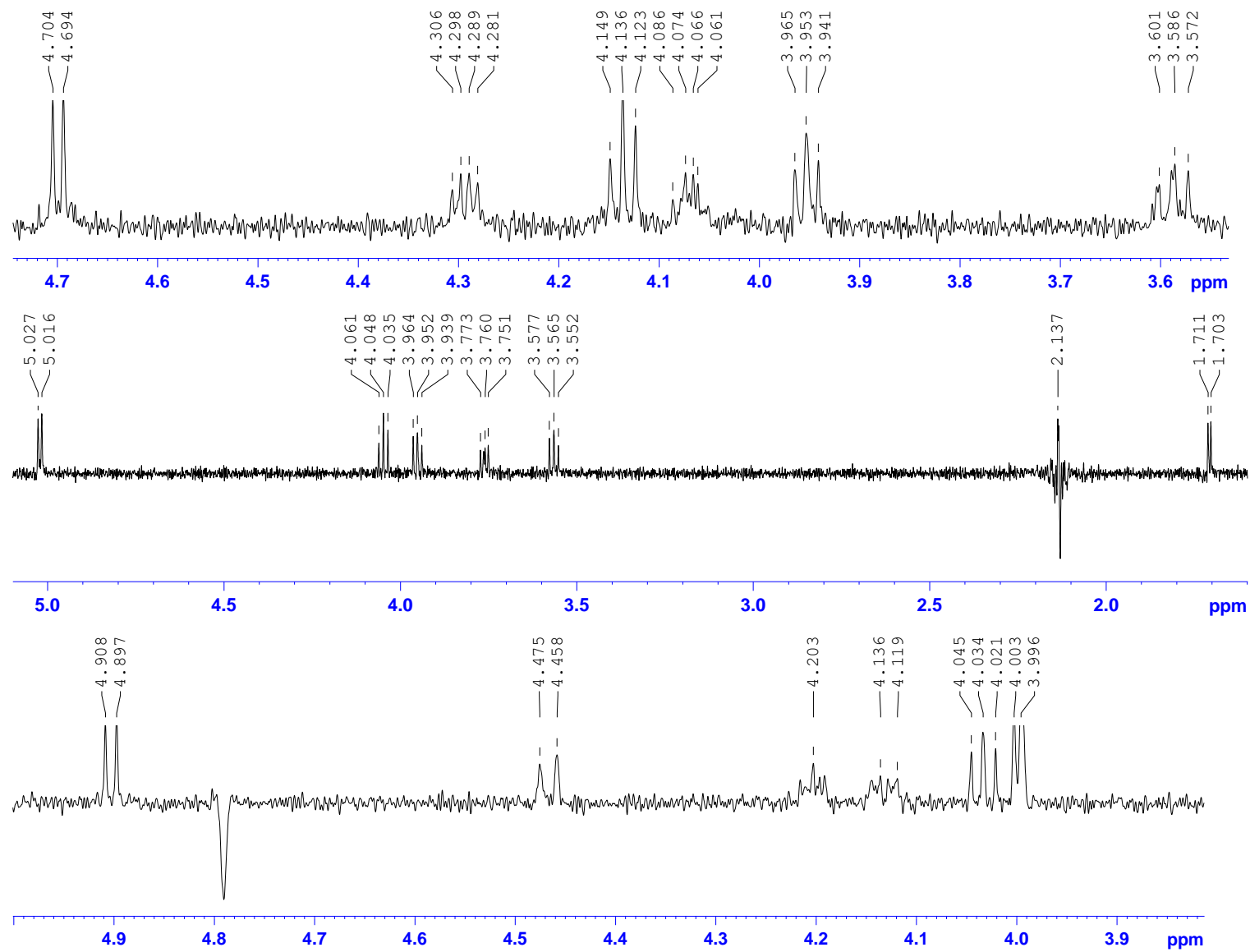


Figure S29. 1 D TOCSY (700.00 MHz) spectra of kuriloside D (**4**) in C<sub>5</sub>D<sub>5</sub>N/D<sub>2</sub>O (4/1)

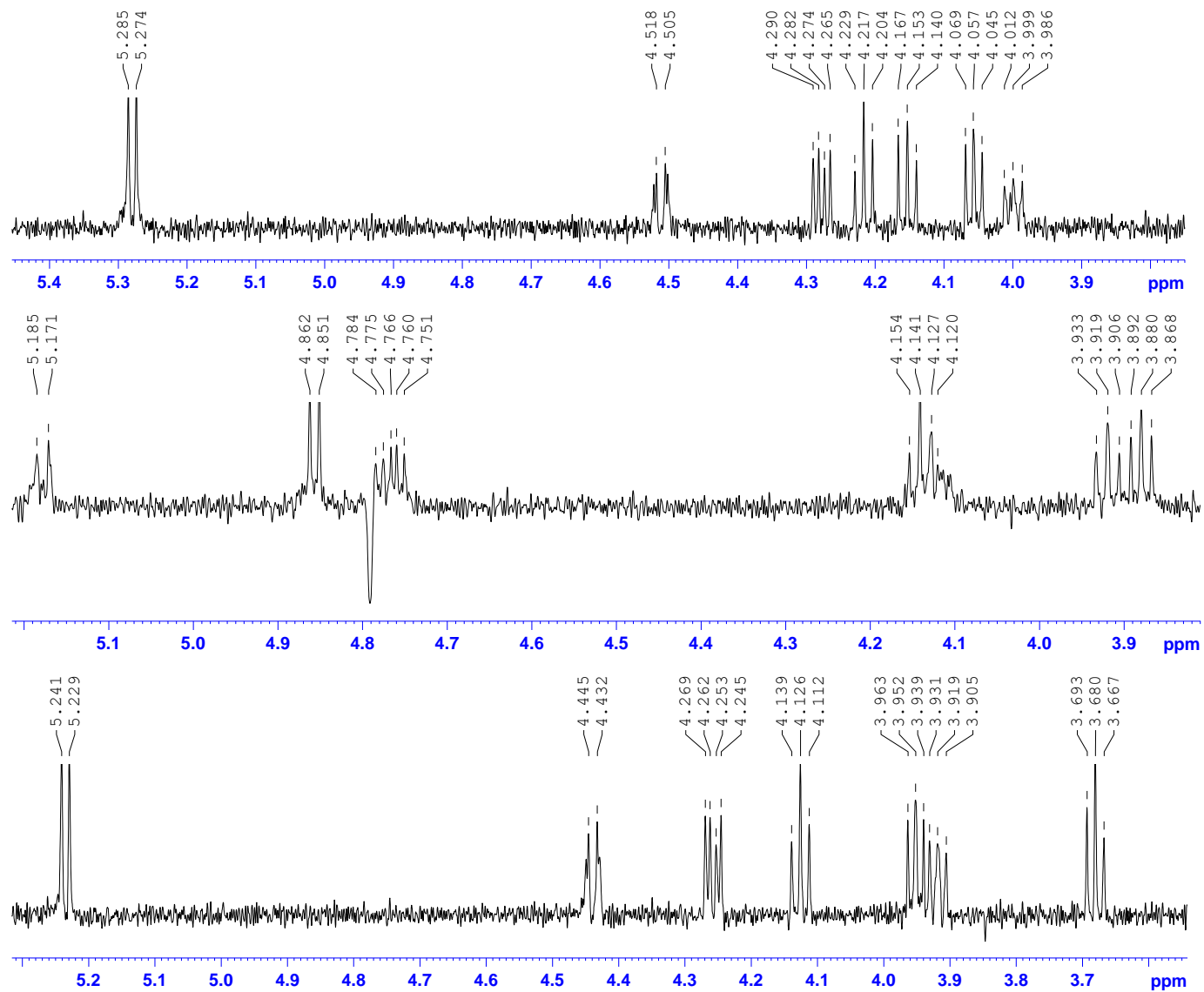


Figure S30. 1 D TOCSY (700.00 MHz) spectra of kuriloside D (**4**) in C<sub>5</sub>D<sub>5</sub>N/D<sub>2</sub>O (4/1)

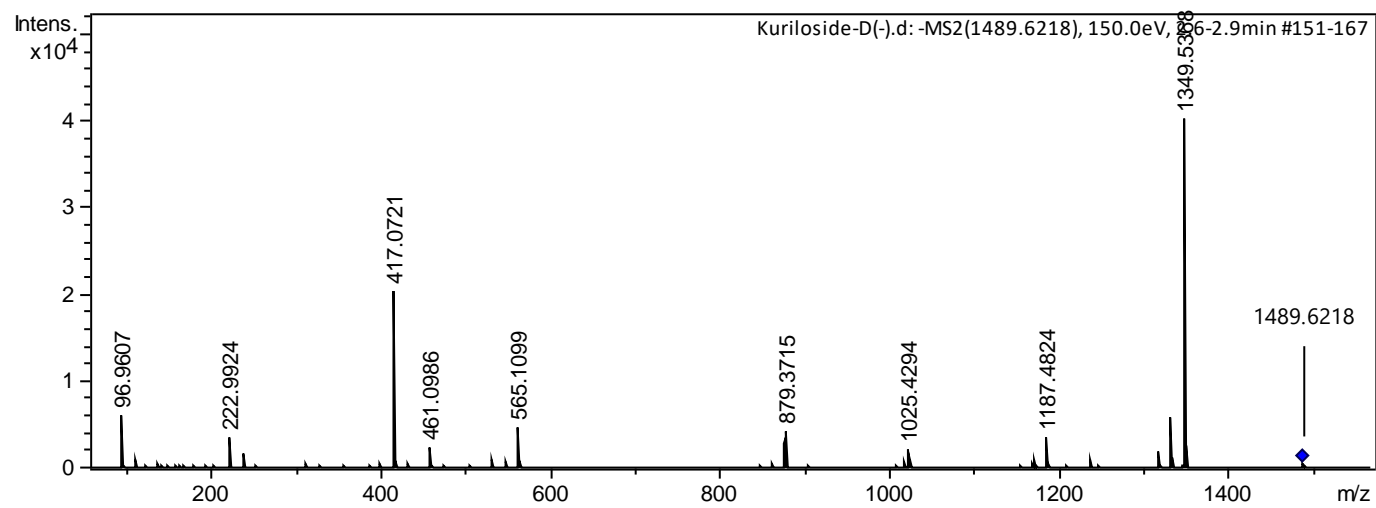
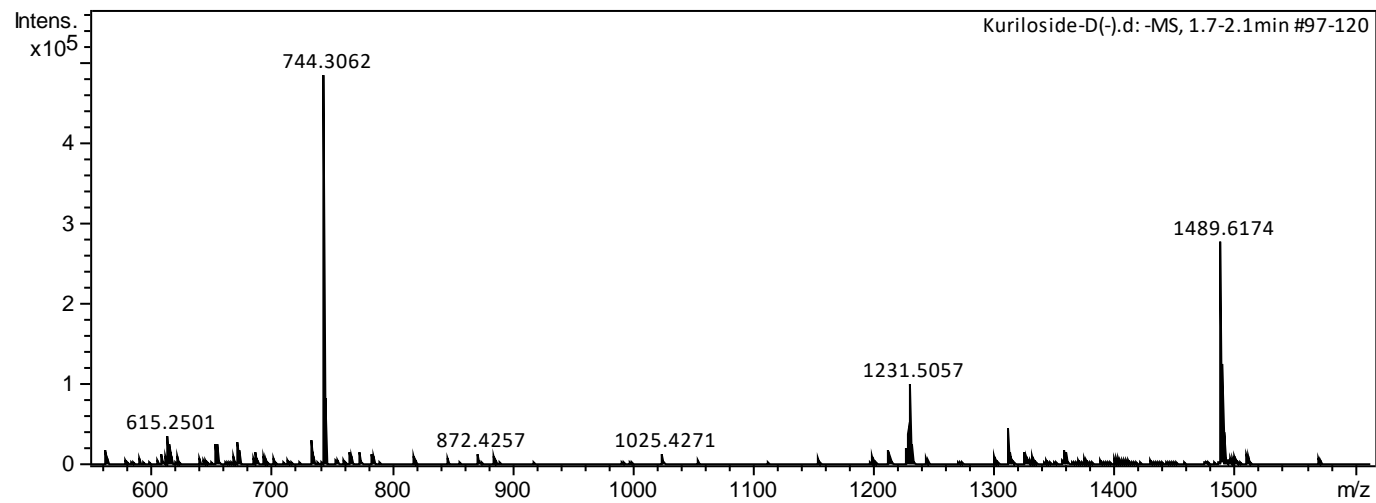


Figure S31. HR-ESI-MS and ESI-MS/MS spectra of kuriloside D (4)

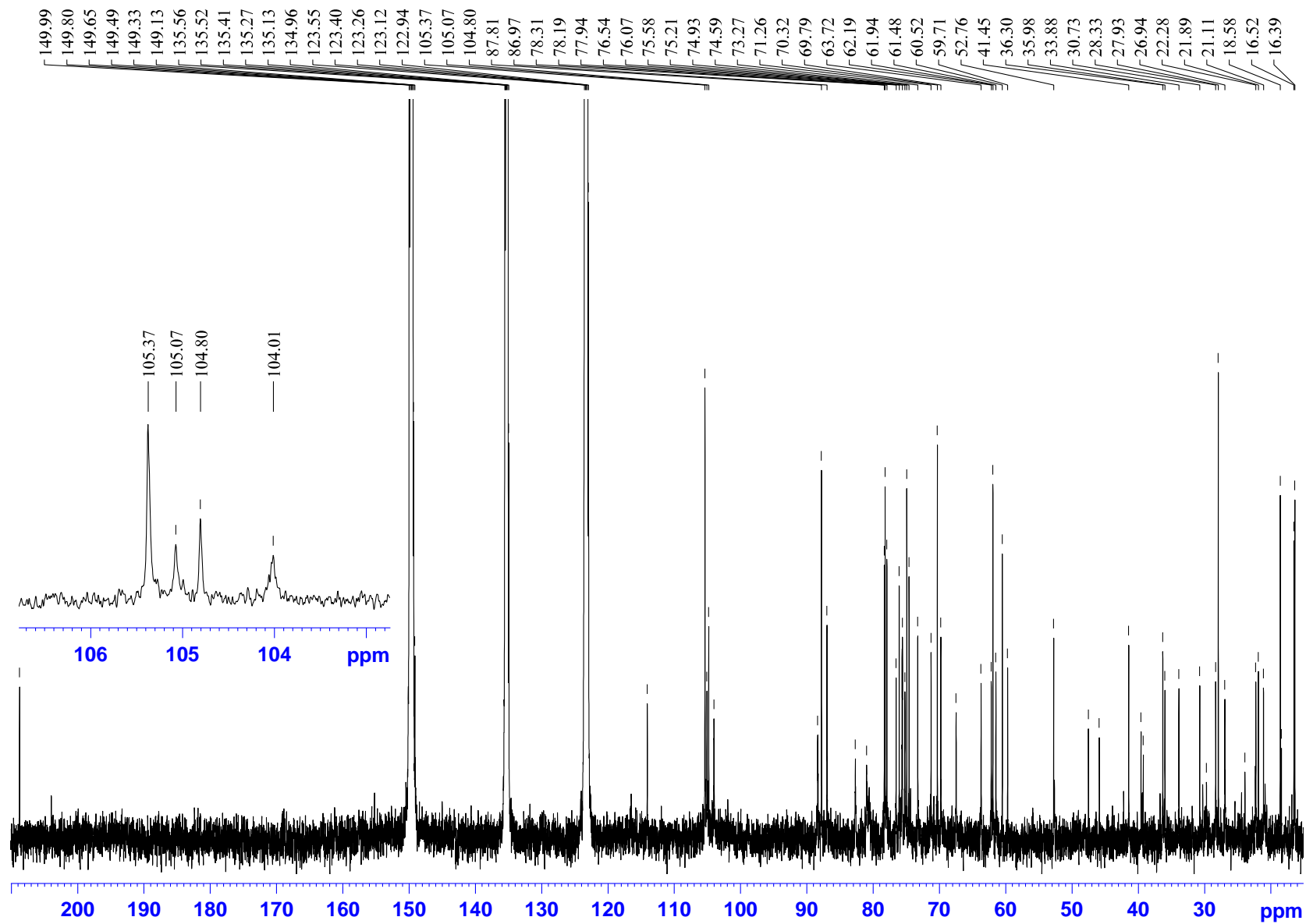


Figure S32. The  $^{13}\text{C}$  NMR (176.03 MHz) spectrum of kuriloside E (5) in  $\text{C}_5\text{D}_5\text{N}/\text{D}_2\text{O}$  (4/1)

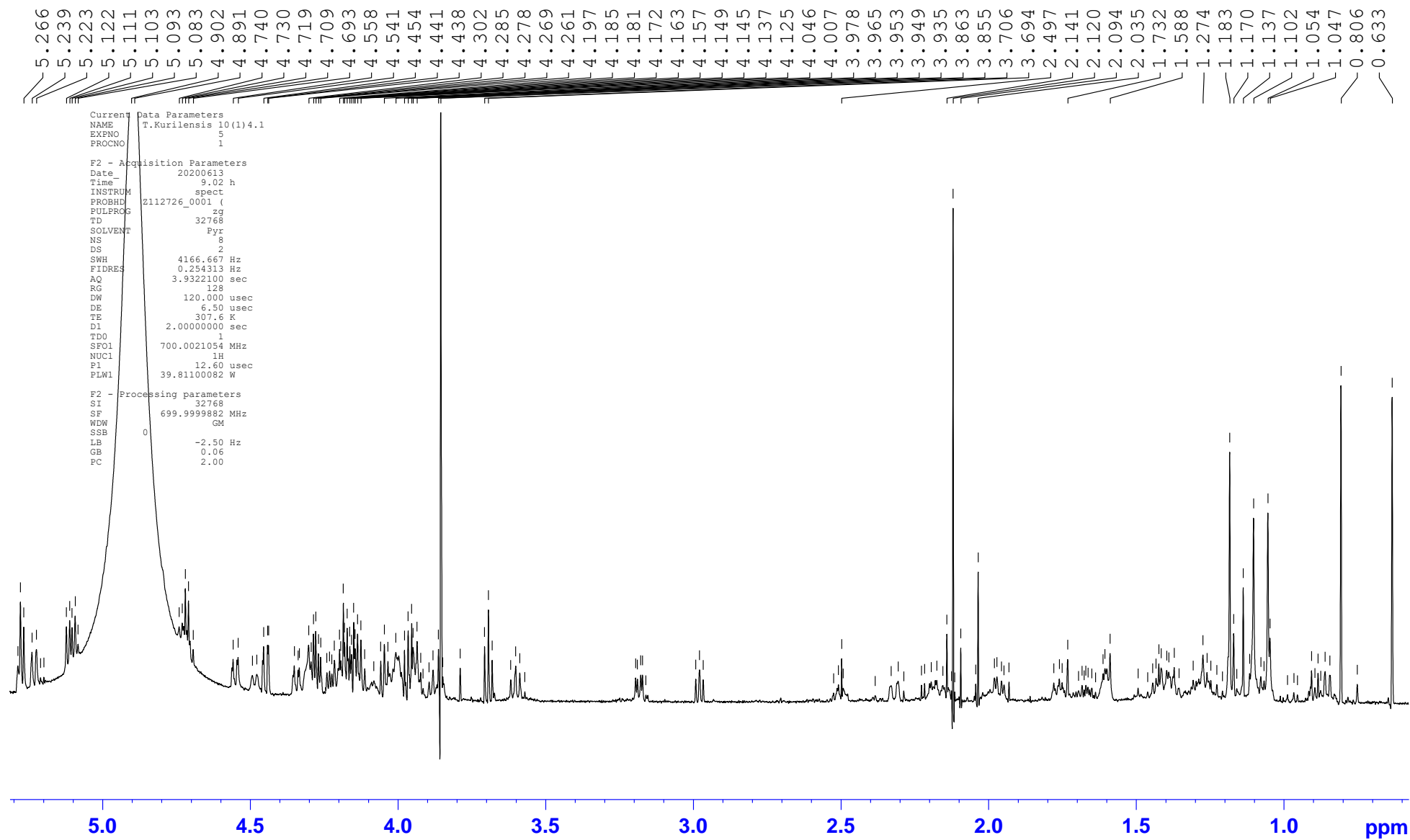


Figure S33. The  $^1\text{H}$  NMR (700.00 MHz) spectrum of kuriloside E (5) in  $\text{C}_5\text{D}_5\text{N}/\text{D}_2\text{O}$  (4/1)

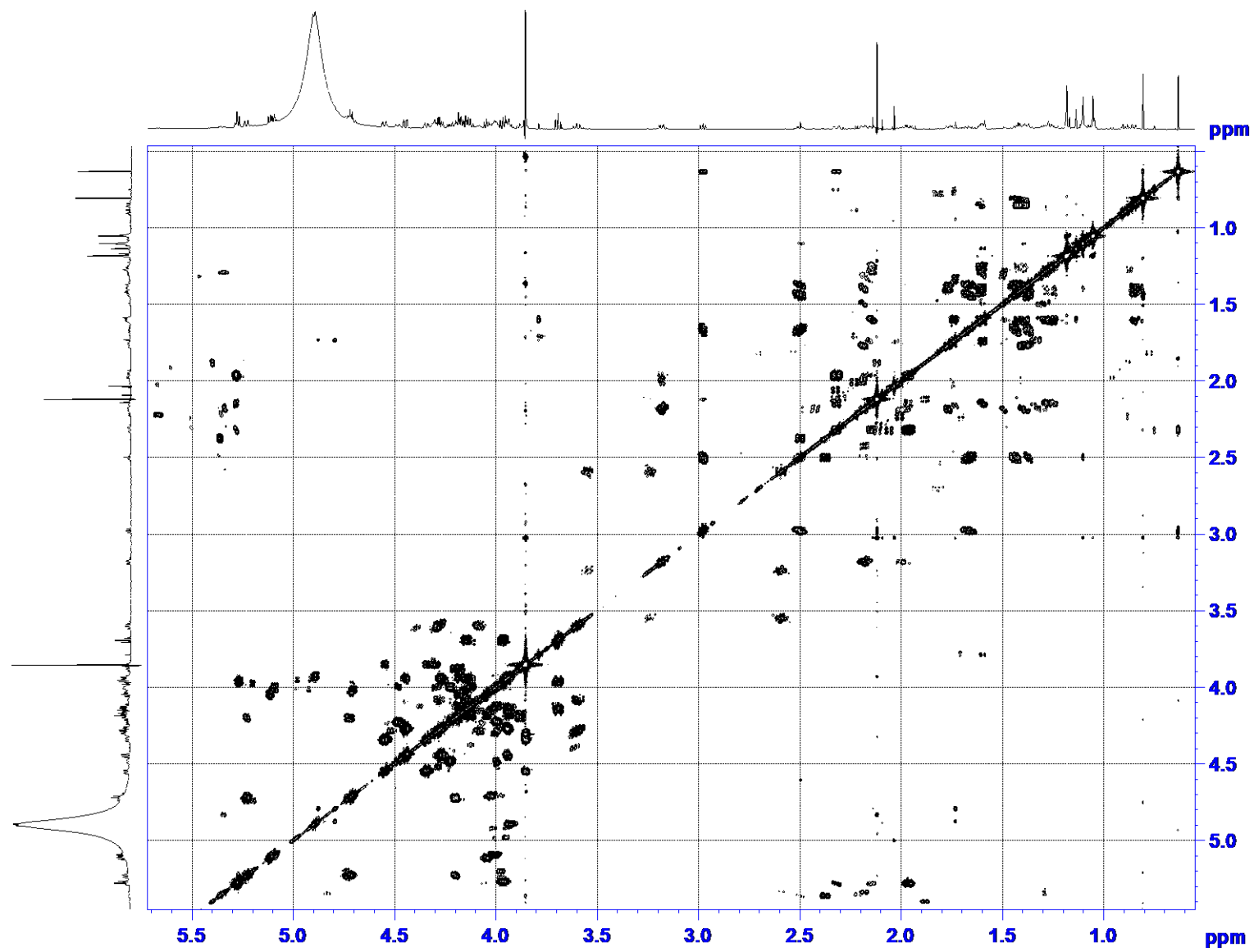


Figure S34. The COSY (700.00 MHz) spectrum of kuriloside E (5) in C<sub>5</sub>D<sub>5</sub>N/D<sub>2</sub>O (4/1)

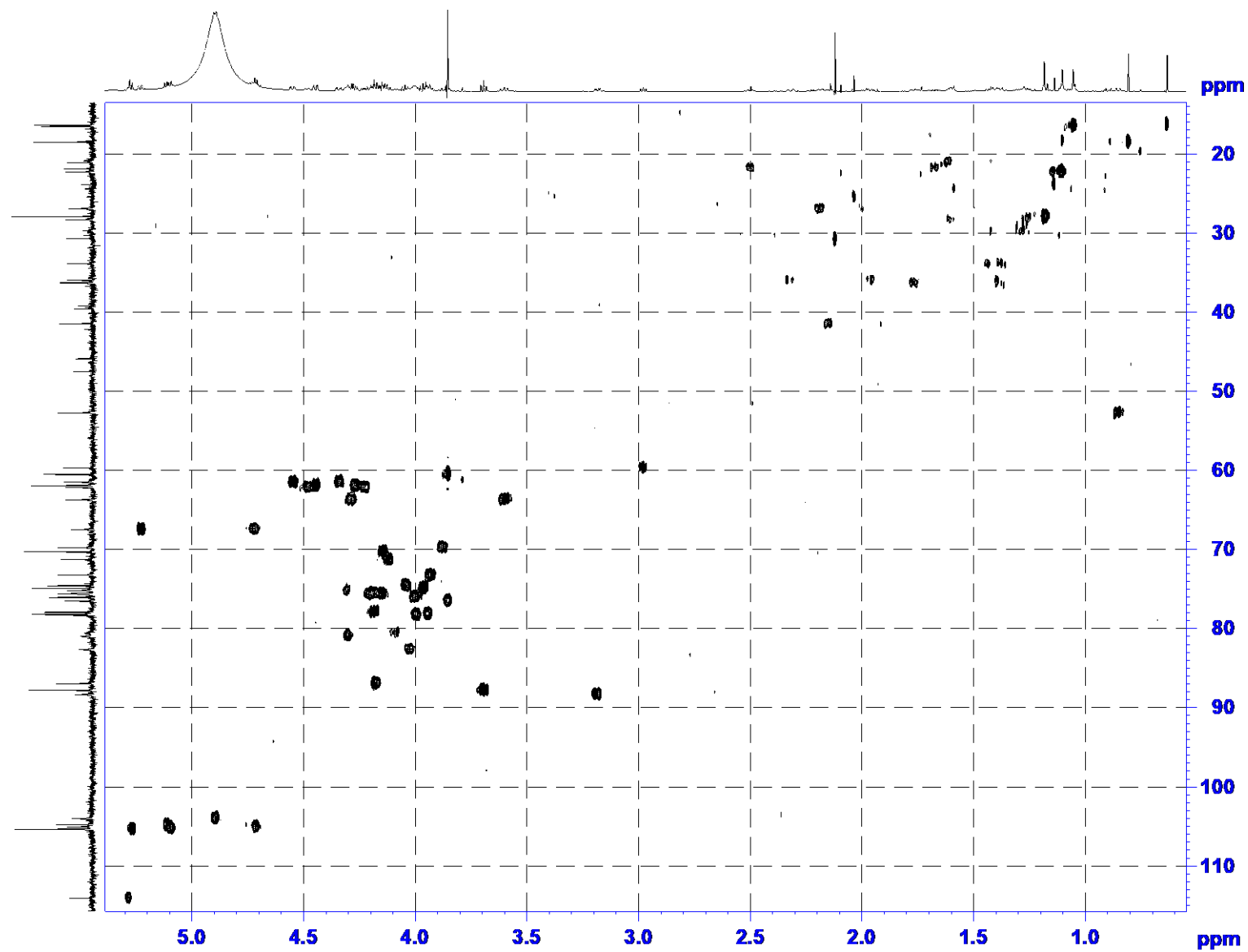


Figure S35. The HSQC (700.00 MHz) spectrum of kuriloside E (5) in  $\text{C}_5\text{D}_5\text{N}/\text{D}_2\text{O}$  (4/1)

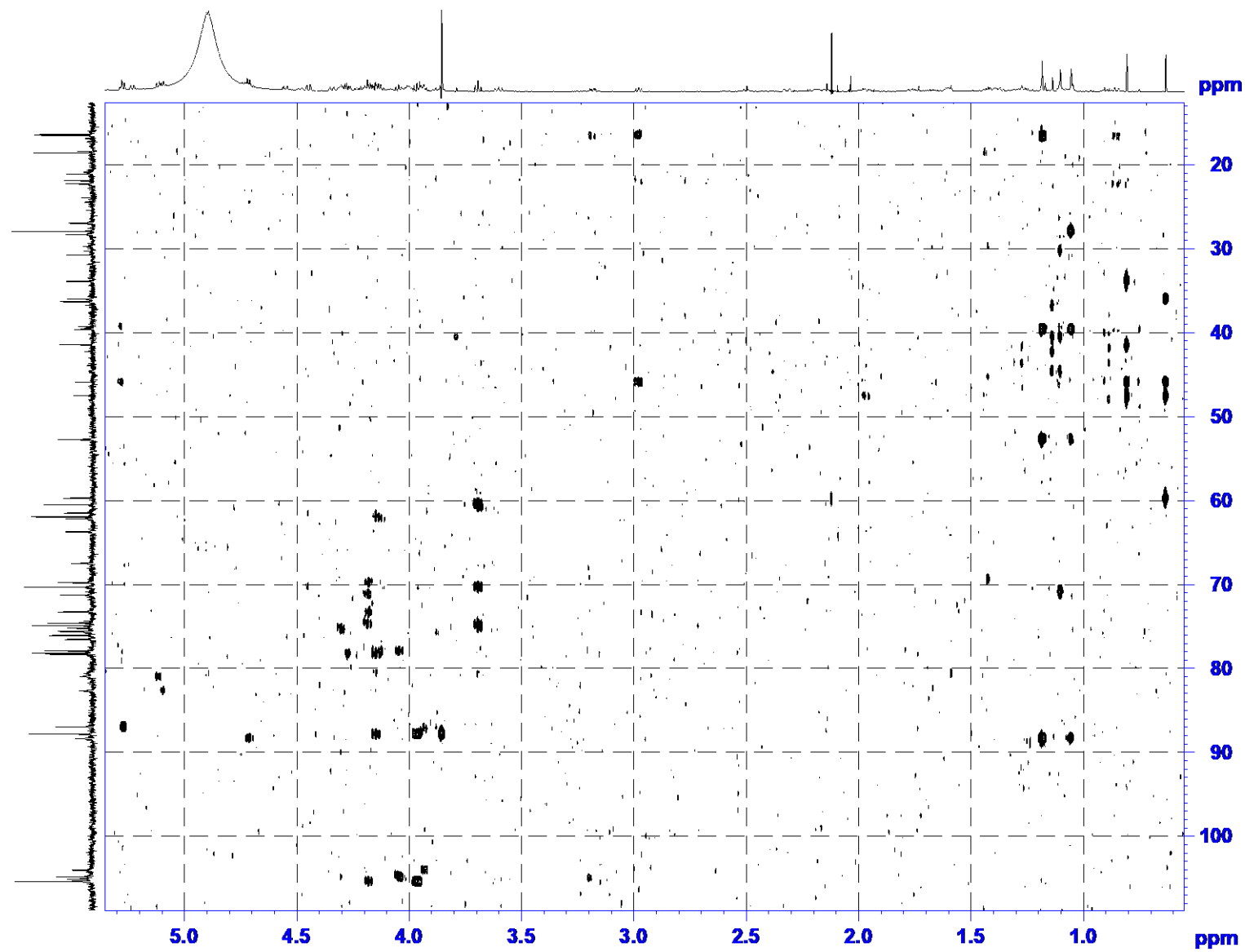


Figure S36. The HMBC (700.00 MHz) spectrum of kuriloside E (5) in  $C_5D_5N/D_2O$  (4/1)



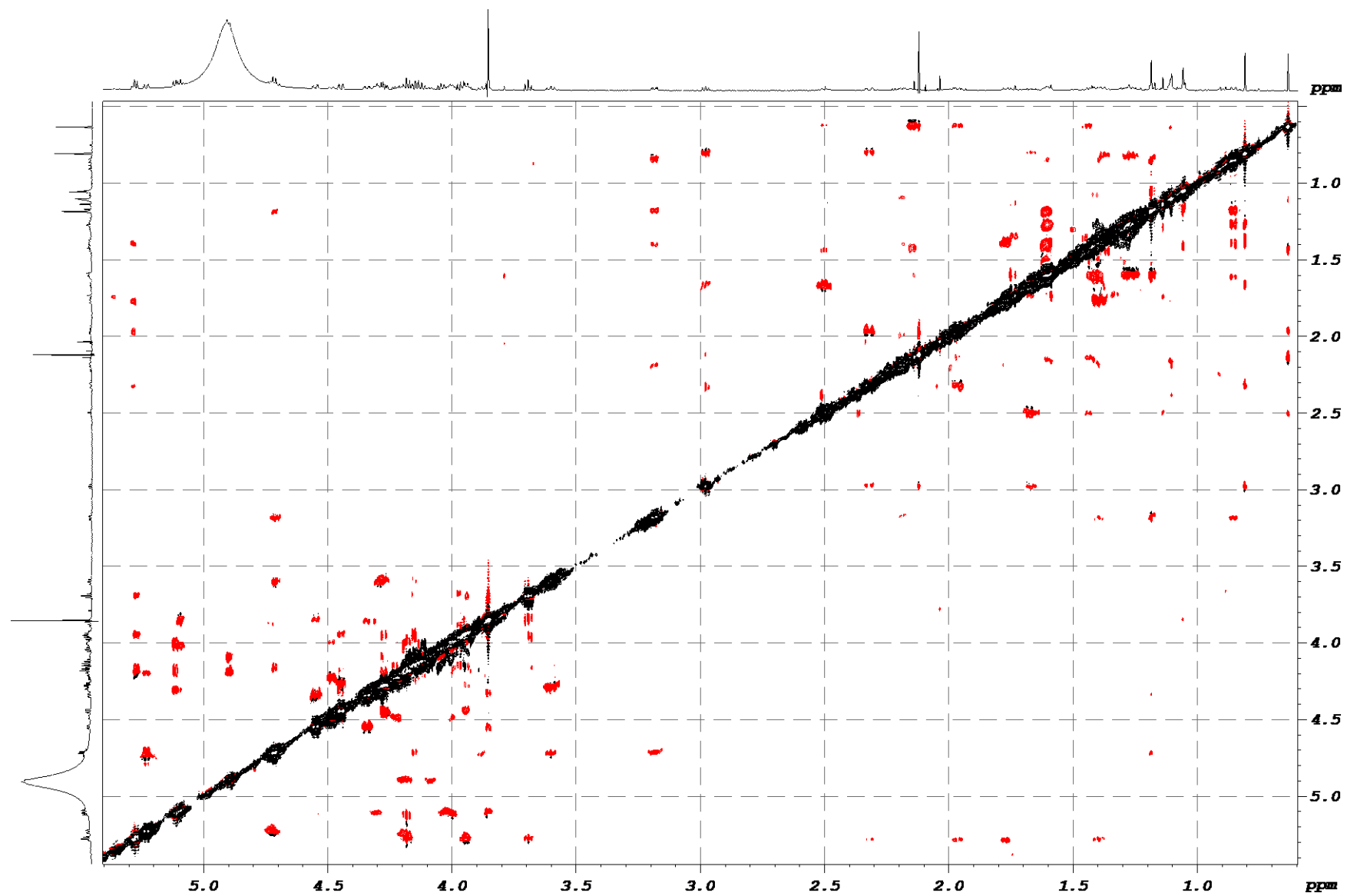


Figure S37. The ROESY (700.00 MHz) spectrum of kuriloside E (5) in C<sub>5</sub>D<sub>5</sub>N/D<sub>2</sub>O (4/1)

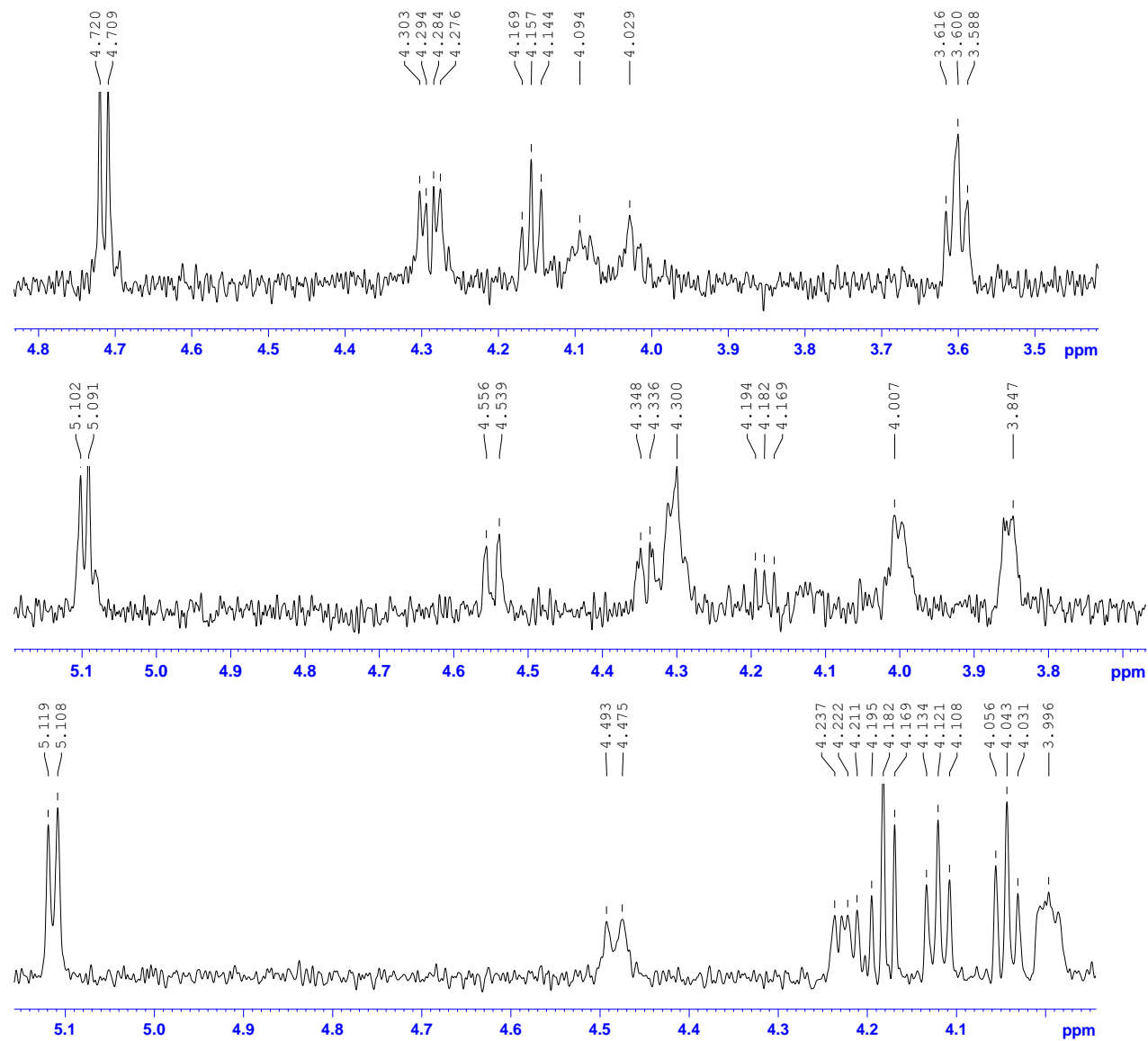


Figure S38. 1D TOCSY (700.00 MHz) spectra of kurilose E (5) in C<sub>5</sub>D<sub>5</sub>N/D<sub>2</sub>O (4/1)

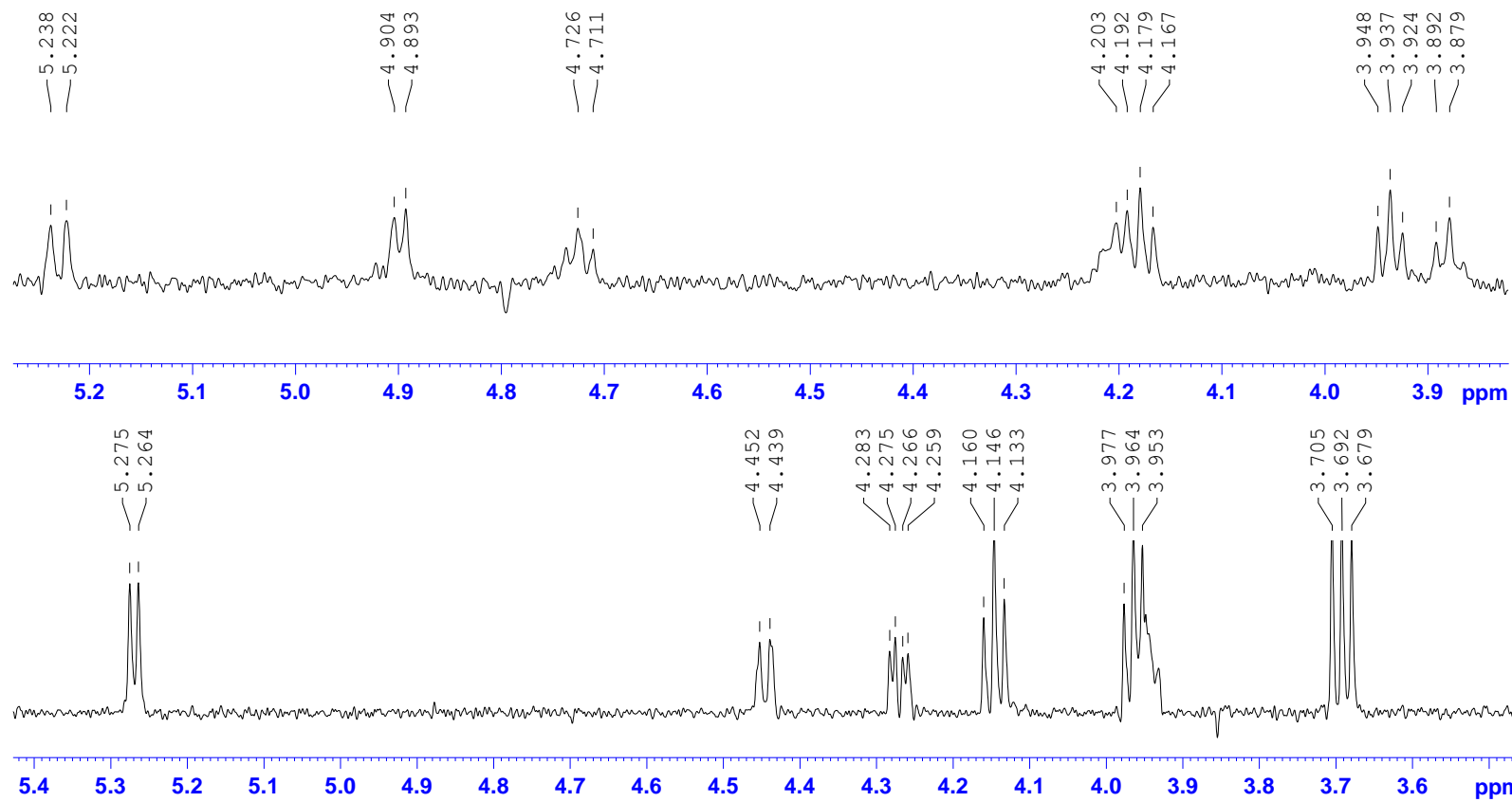


Figure S39. 1D TOCSY (700.00 MHz) spectra of kuriloside E (5) in C<sub>5</sub>D<sub>5</sub>N/D<sub>2</sub>O (4/1)

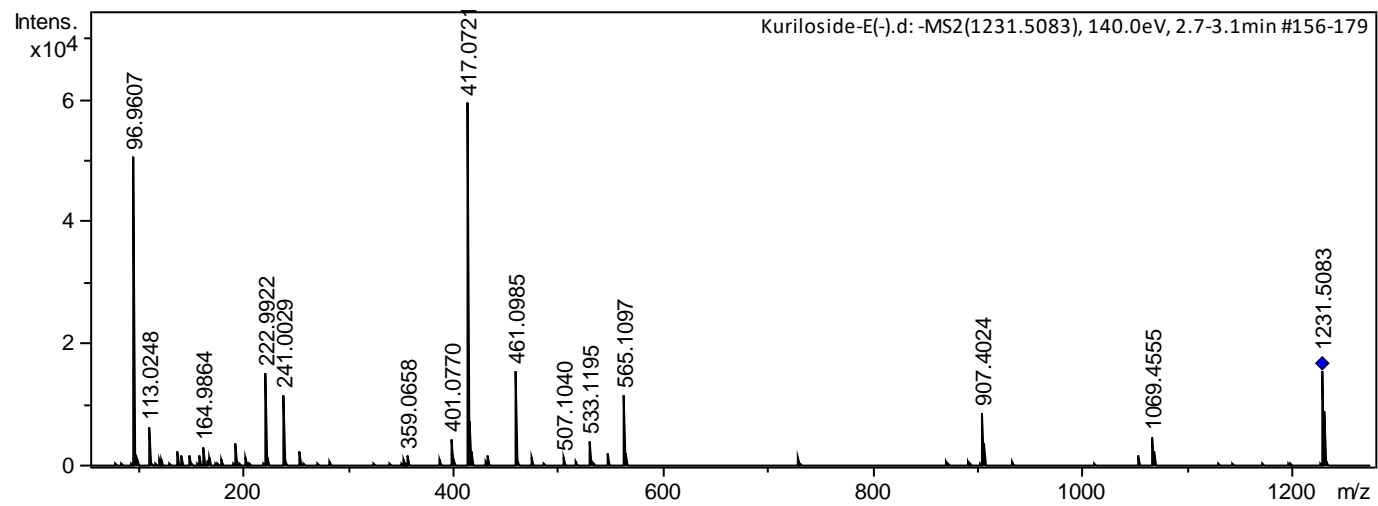
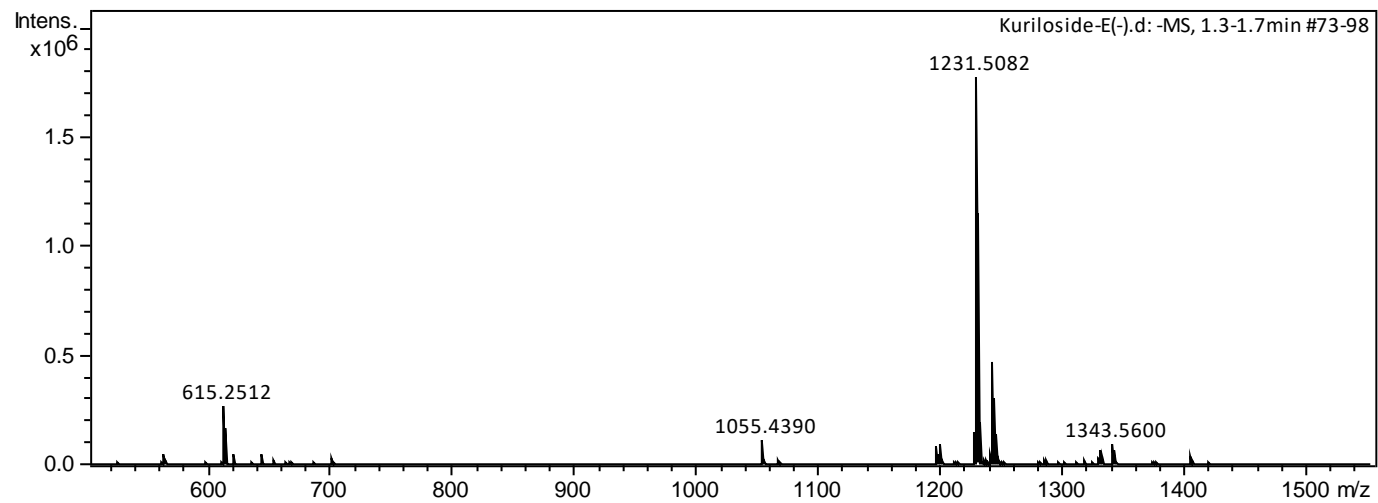


Figure S40. HR-ESI-MS and ESI-MS/MS spectra of kurilosite E (5)

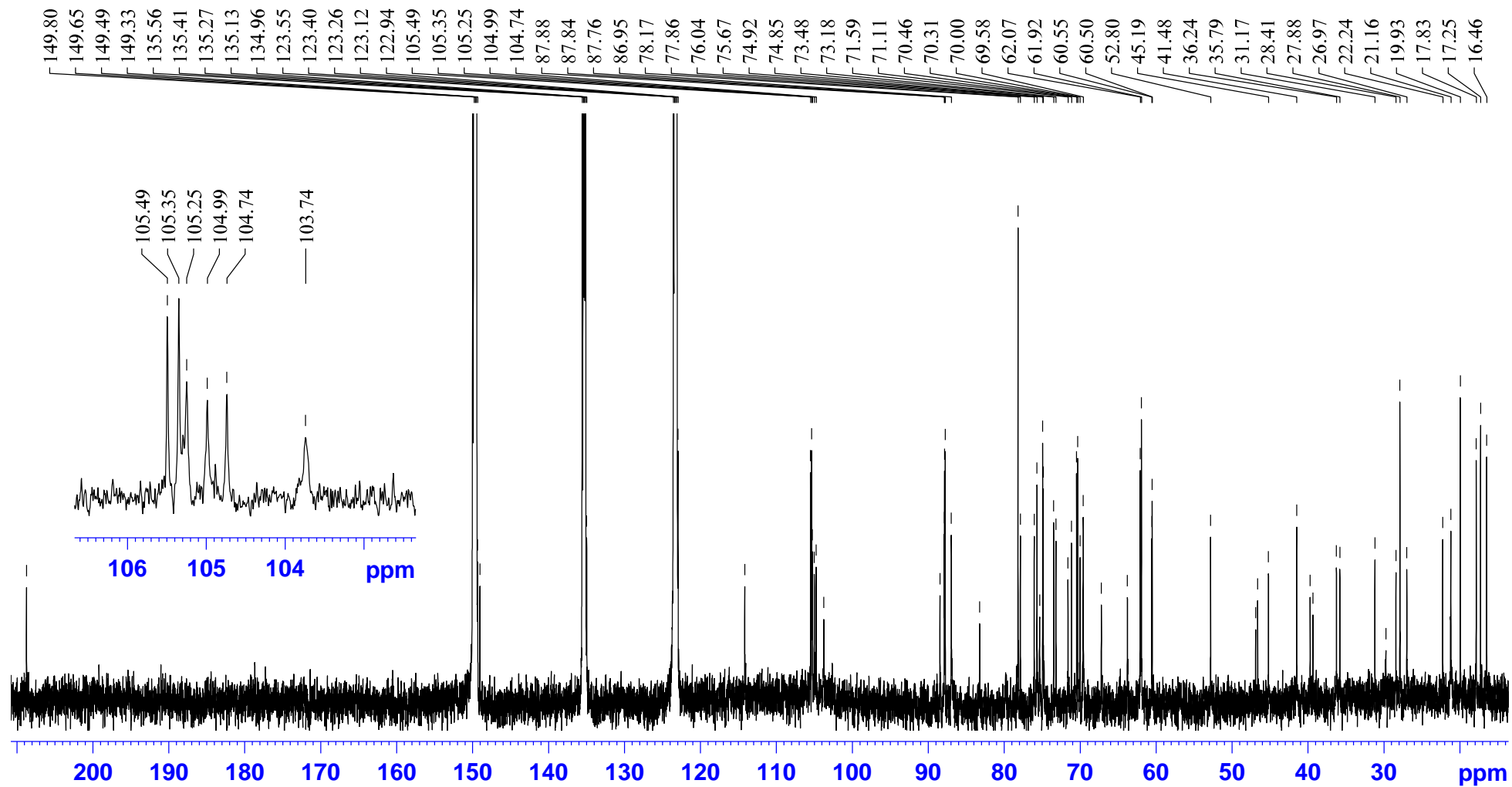


Figure S41. The  $^{13}\text{C}$  NMR (176.03 MHz) spectrum of kuriloside F (**6**) in  $\text{C}_5\text{D}_5\text{N}/\text{D}_2\text{O}$  (4/1)

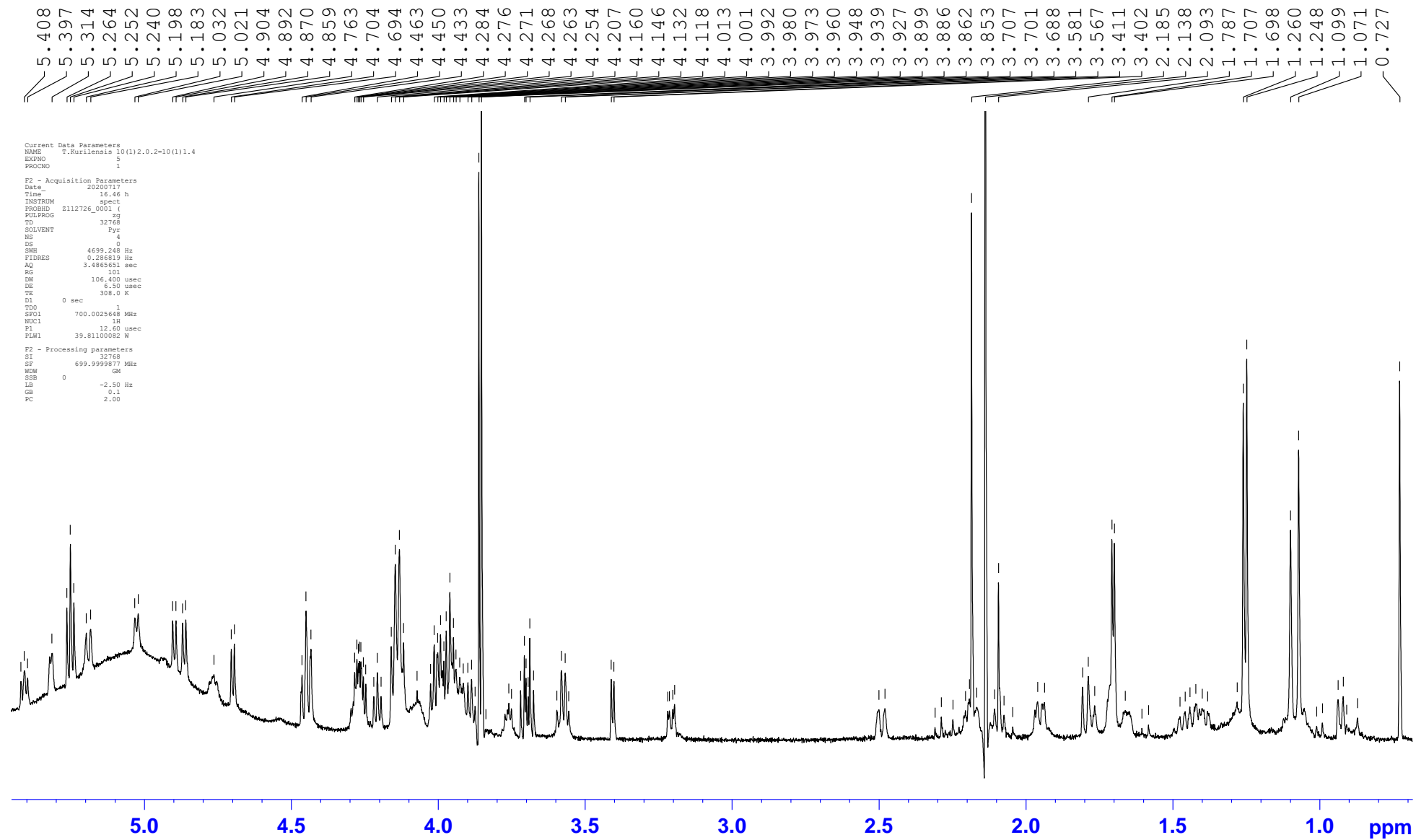


Figure S42. The  $^1\text{H}$  NMR (700.00 MHz) spectrum of kurilaside F (6) in  $\text{C}_5\text{D}_5\text{N}/\text{D}_2\text{O}$  (4/1)

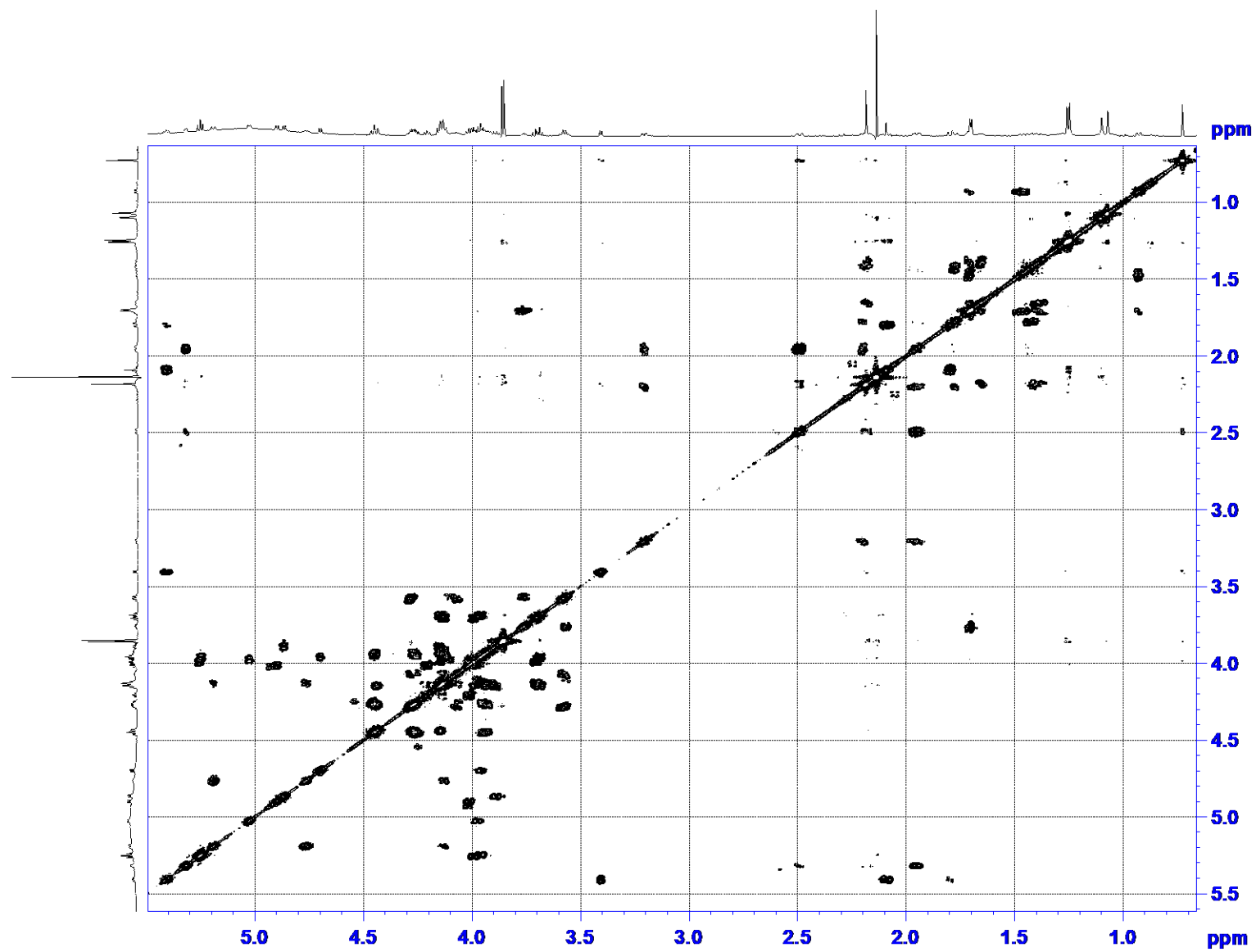


Figure S43. The COSY (700.00 MHz) spectrum of kuriloside F (6) in  $C_5D_5N/D_2O$  (4/1)

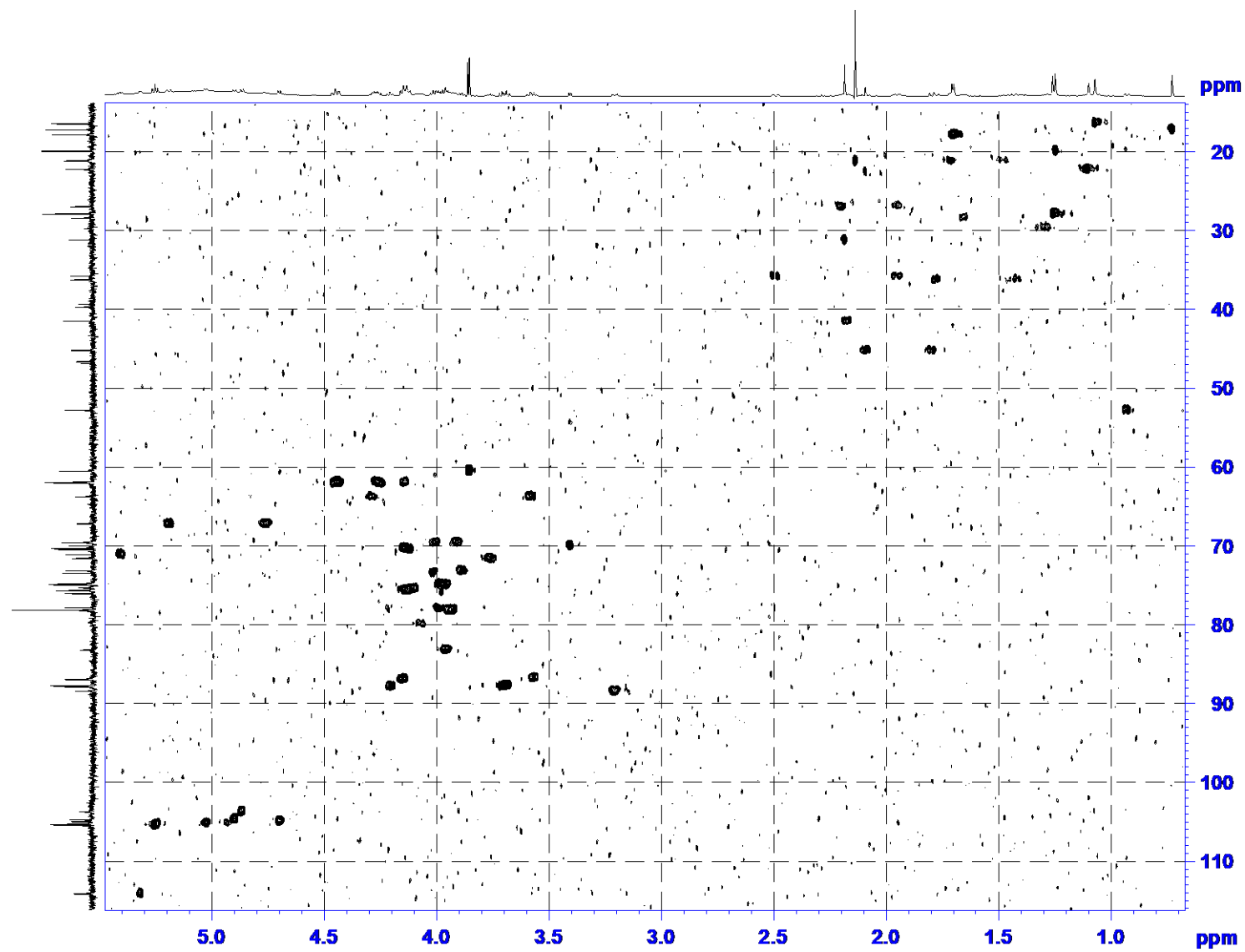


Figure S44. The HSQC (700.00 MHz) spectrum of kuriloside F (6) in  $\text{C}_5\text{D}_5\text{N}/\text{D}_2\text{O}$  (4/1)



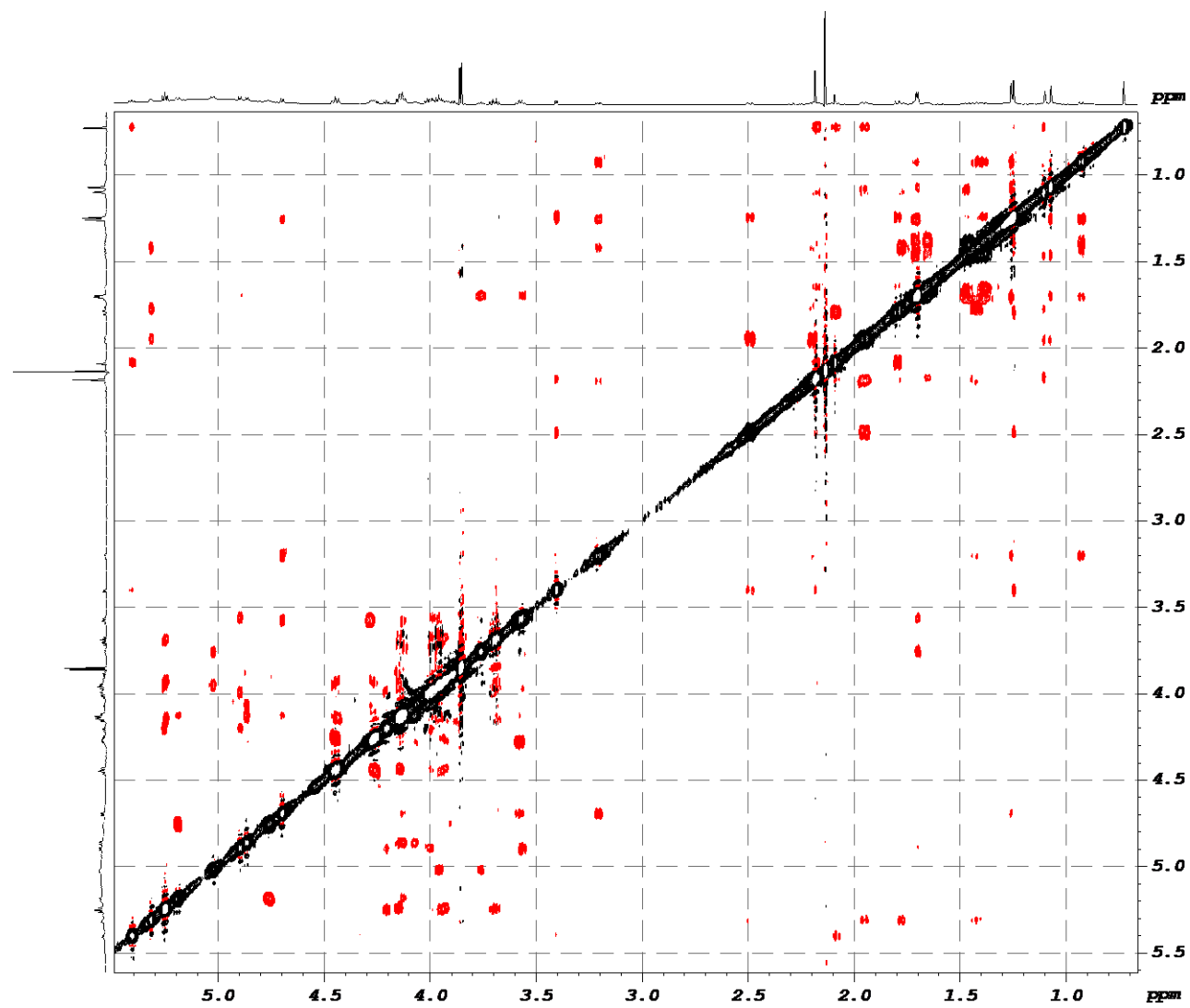


Figure S45. The ROESY (700.00 MHz) spectrum of kuriloside F (**6**) in C<sub>5</sub>D<sub>5</sub>N/D<sub>2</sub>O (4/1)

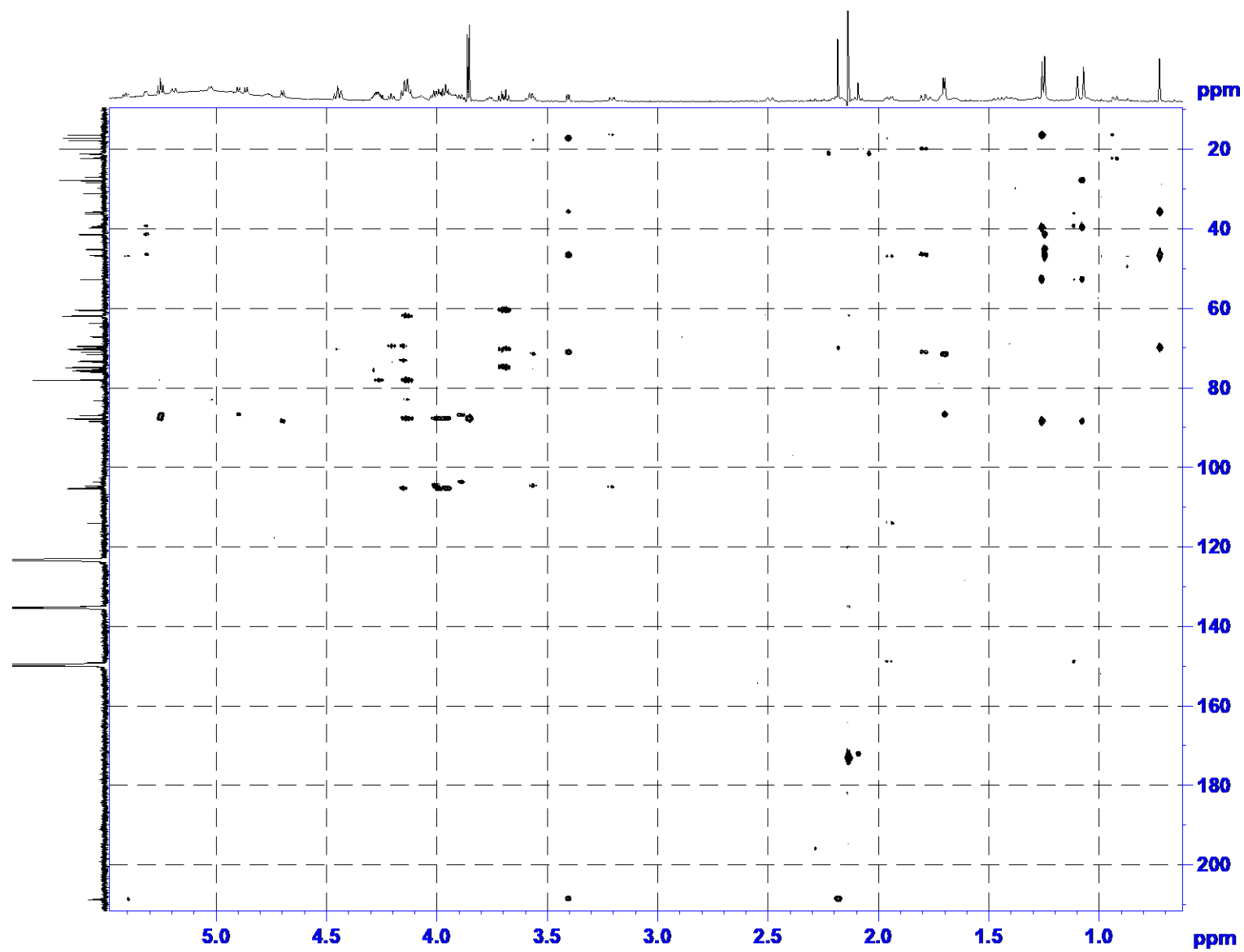


Figure S46. The HMBC (700.00 MHz) spectrum of kuriloside F (6) in  $C_5D_5N/D_2O$  (4/1)

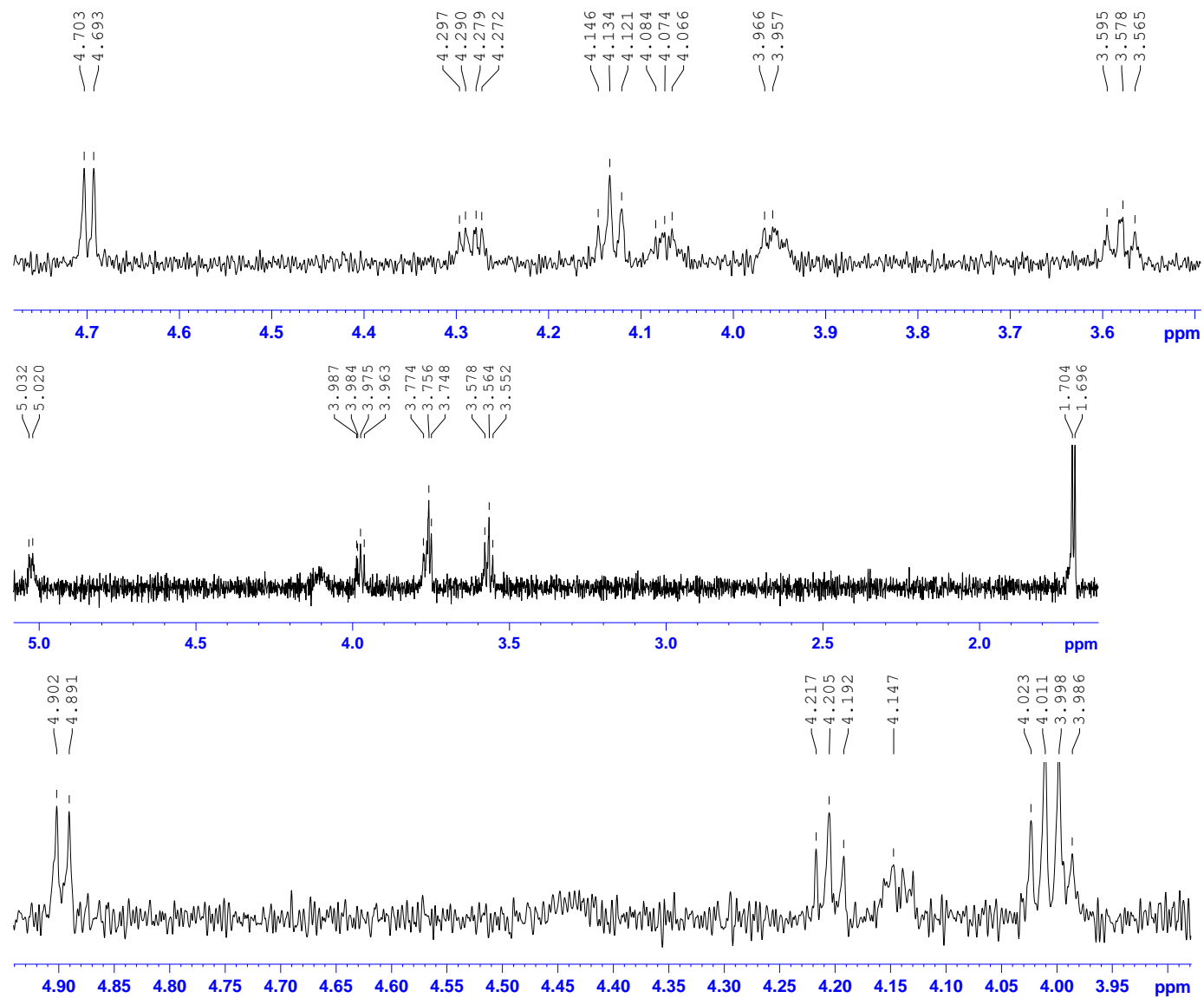


Figure S47. 1D TOCSY (700.00 MHz) spectra of the carbohydrate part of kuriloside F (6) in C<sub>5</sub>D<sub>5</sub>N/D<sub>2</sub>O (4/1)

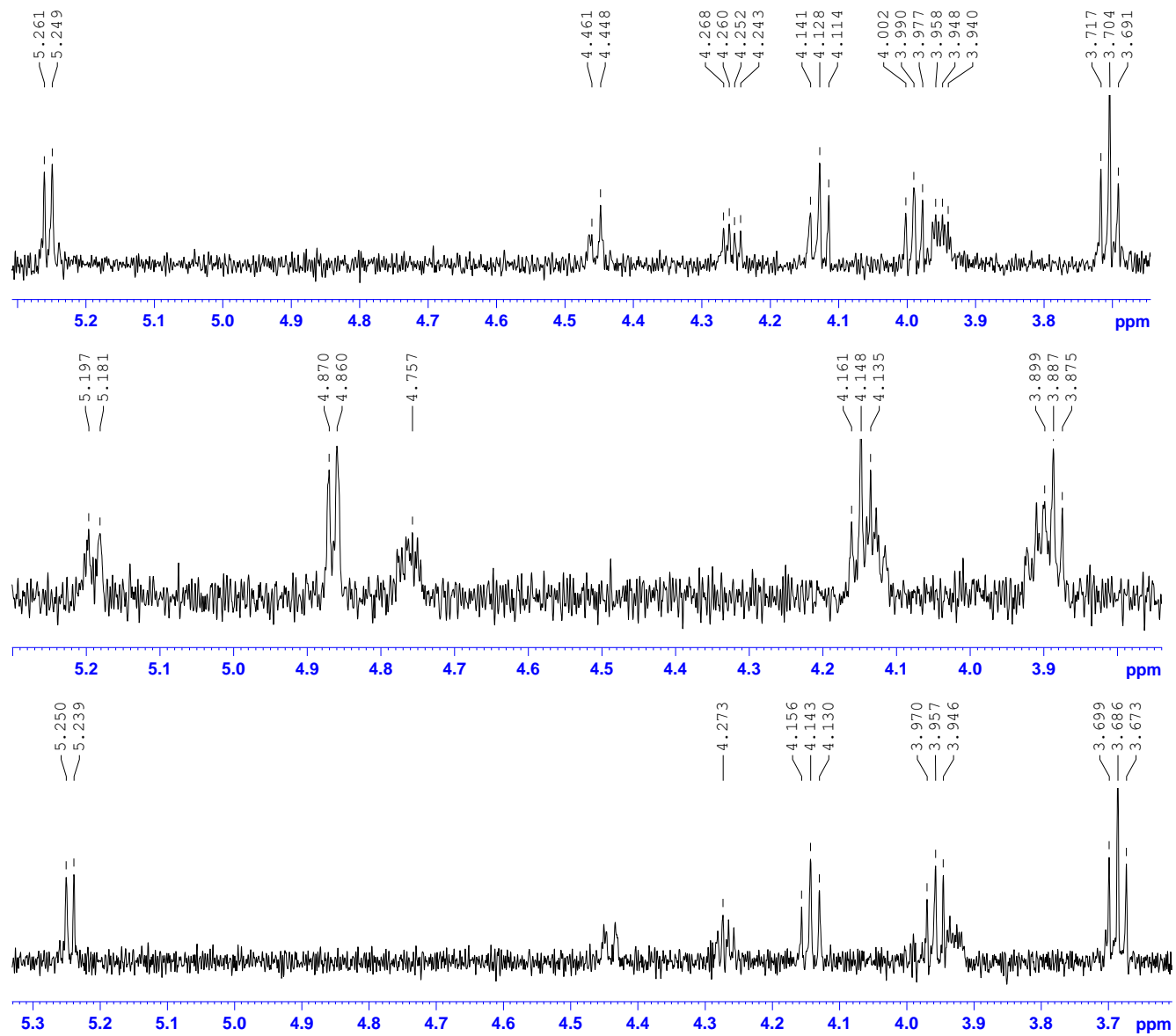


Figure S48. 1D TOCSY (700.00 MHz) spectra of the carbohydrate part of kuriloside F (**6**) in  $C_5D_5N/D_2O$  (4/1)

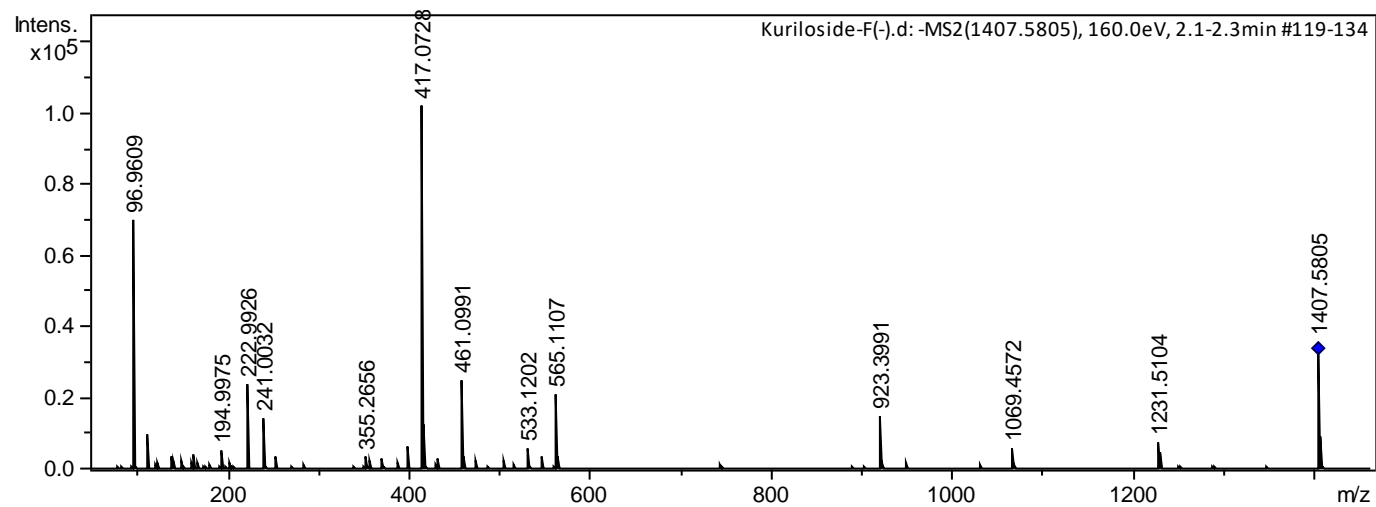
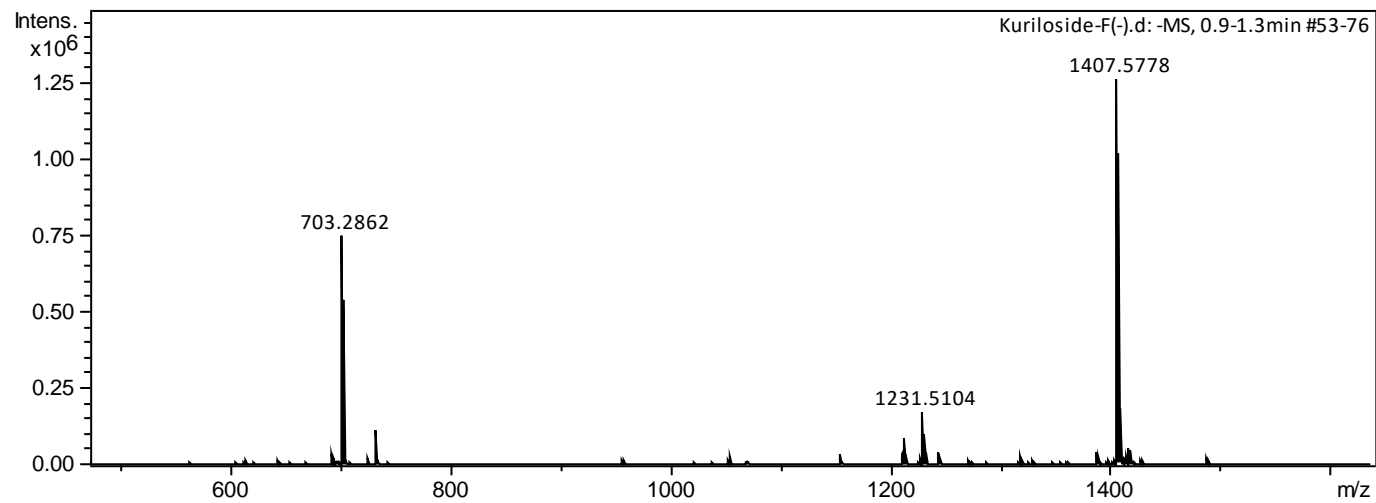


Figure S49. HR-ESI-MS and ESI-MS/MS spectra of kurilosite F (6)

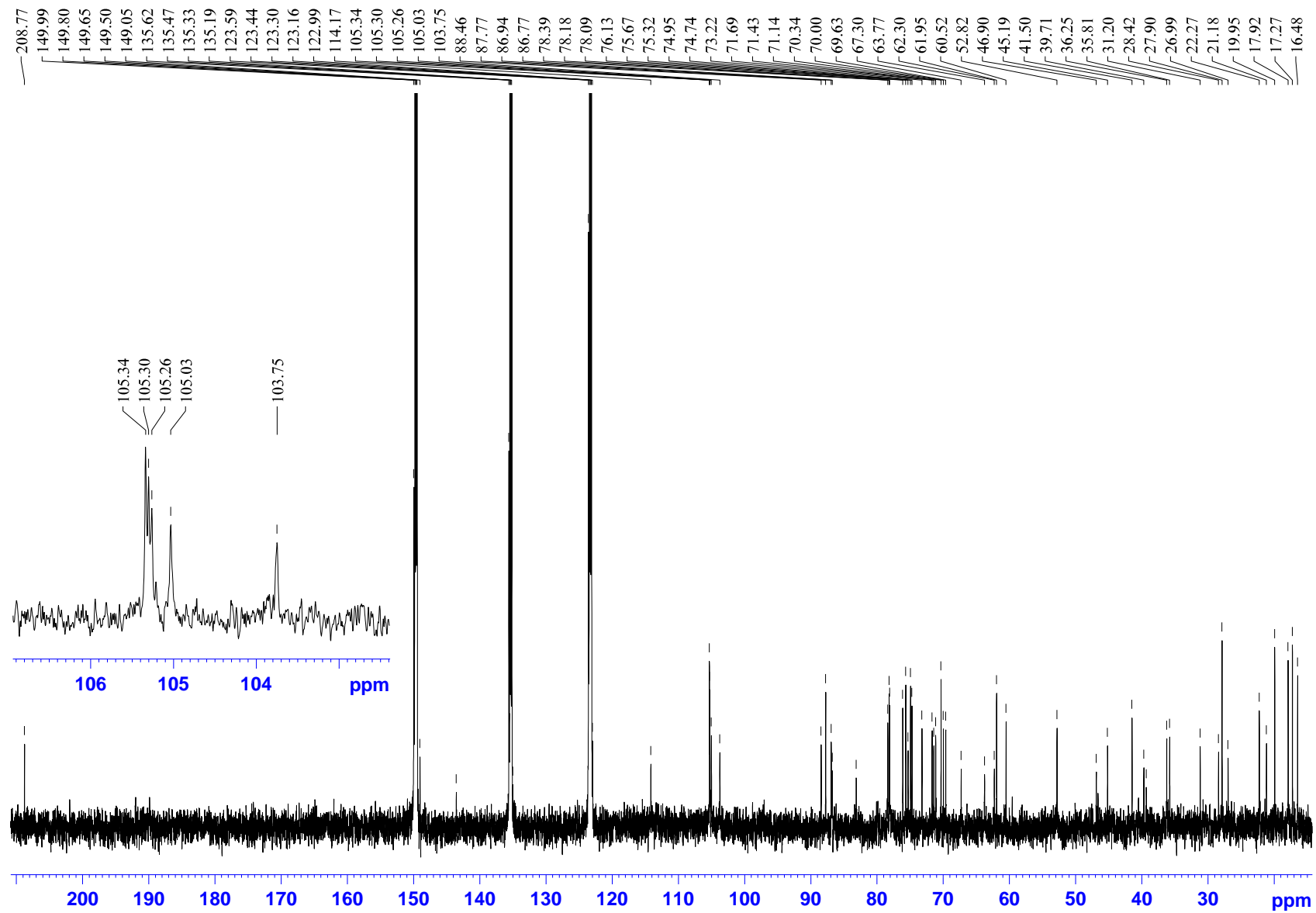


Figure S50. The  $^{13}\text{C}$  NMR (176.03 MHz) spectrum of kuriloside A (7) in  $\text{C}_5\text{D}_5\text{N}/\text{D}_2\text{O}$  (4/1)

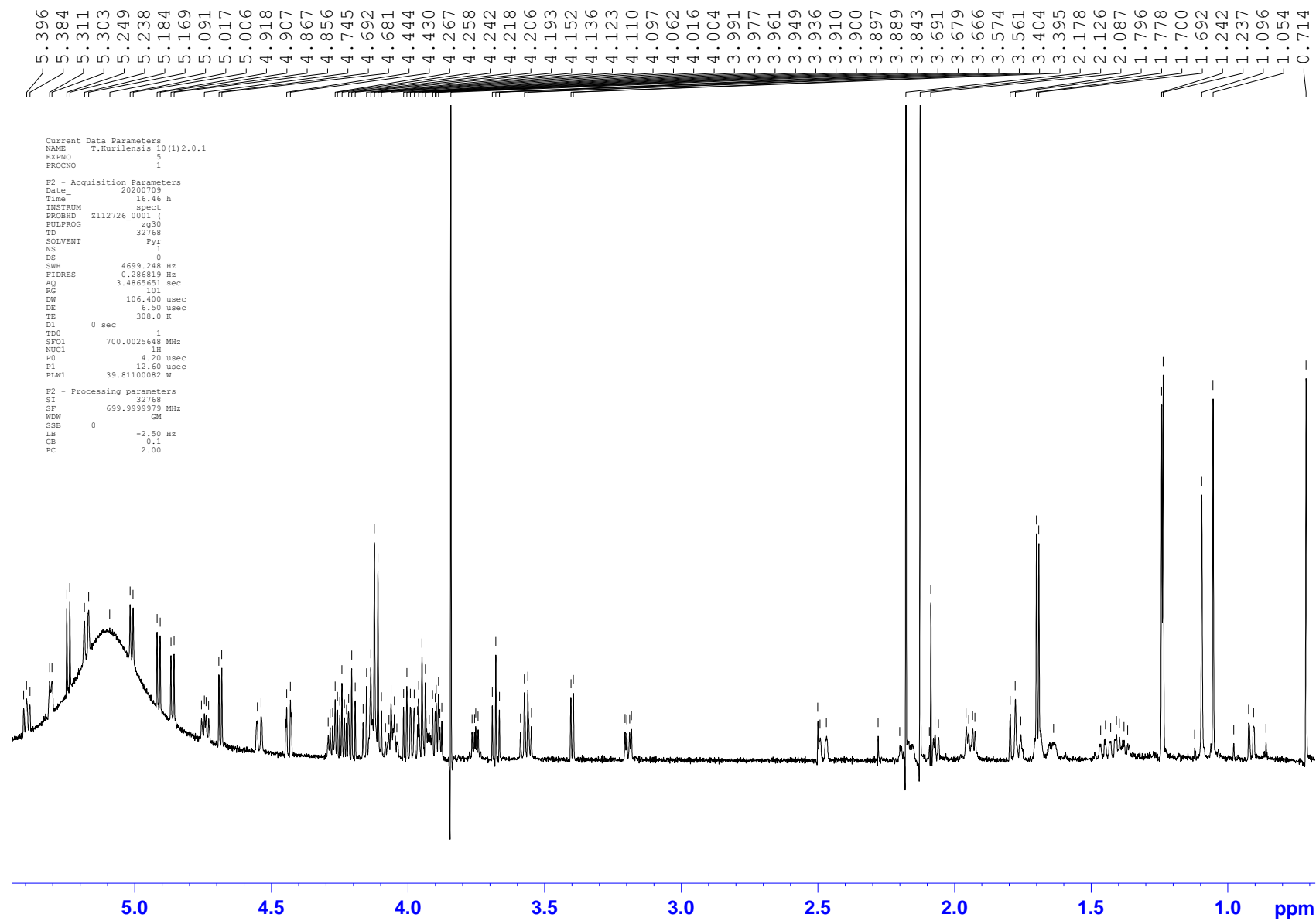


Figure S51. The  $^1\text{H}$  NMR (700.00 MHz) spectrum of kuriloside A (**7**) in  $\text{C}_5\text{D}_5\text{N}/\text{D}_2\text{O}$  (4/1)

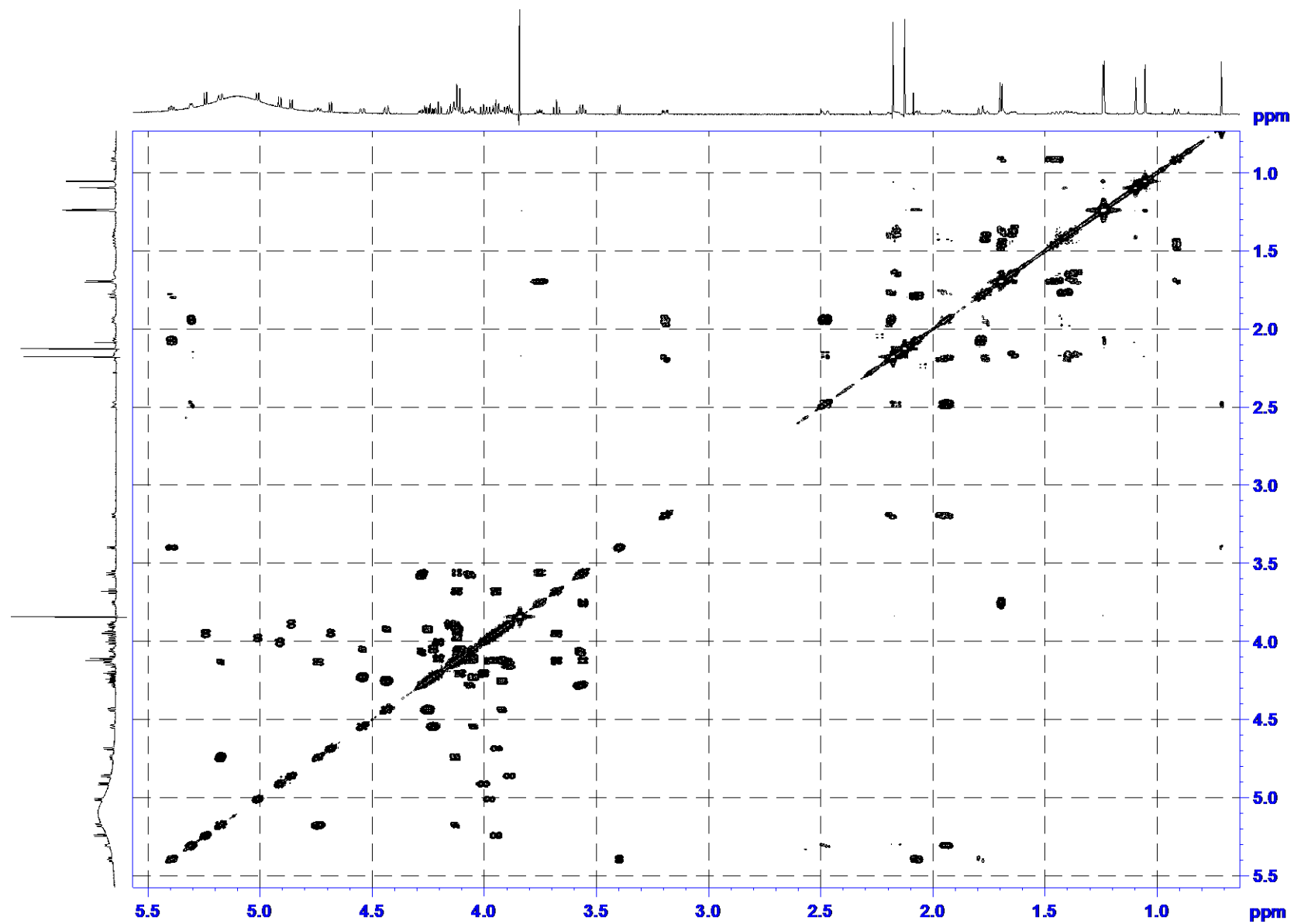


Figure S52. The COSY (700.00 MHz) spectrum of kuriloside A (7) in  $C_5D_5N/D_2O$  (4/1)



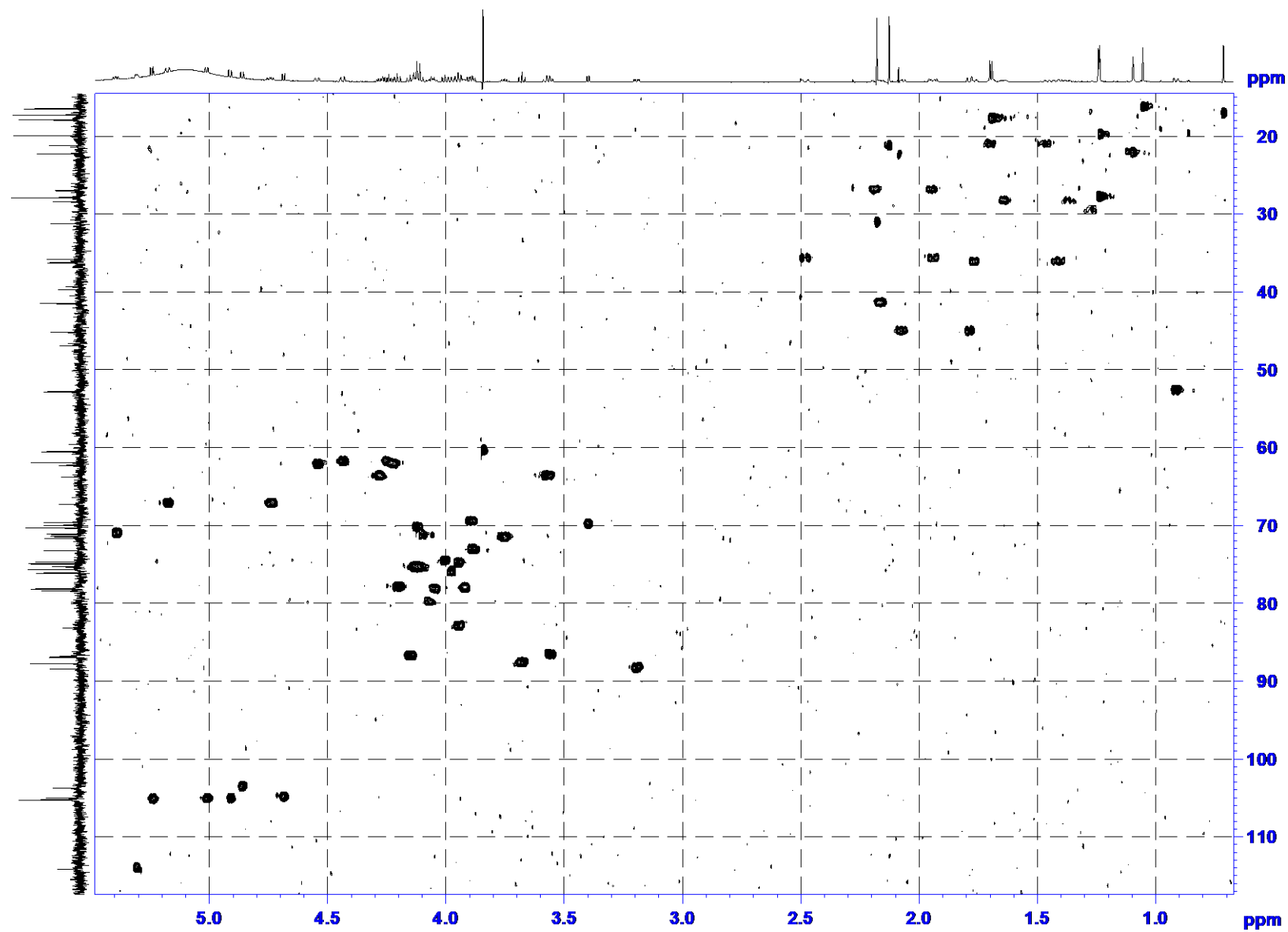


Figure S53. The HSQC (700.00 MHz) spectrum of kuriloside A (7) in  $\text{C}_5\text{D}_5\text{N}/\text{D}_2\text{O}$  (4/1)

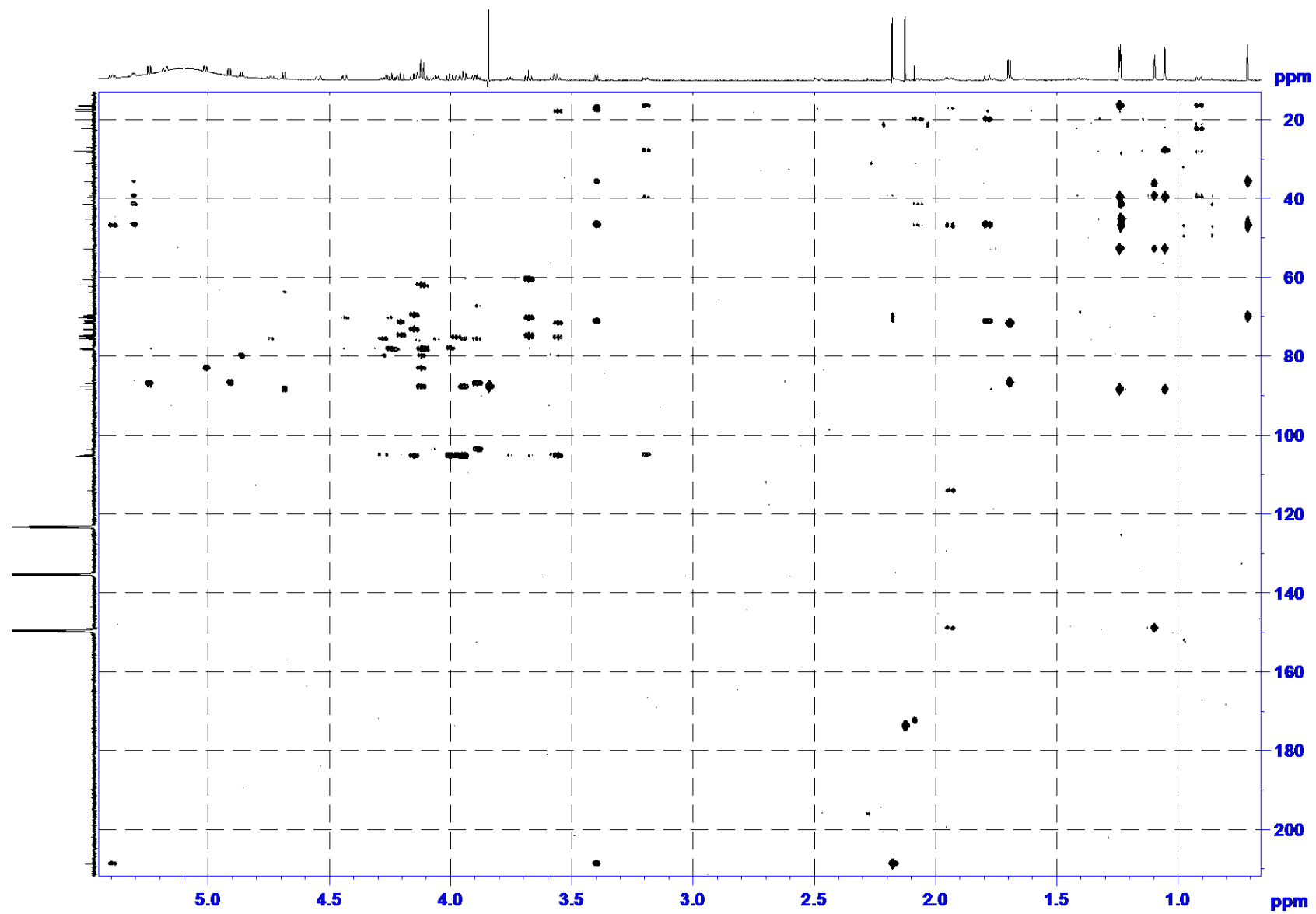


Figure S54. The HMBC (700.00 MHz) spectrum of kuriloside A (7) in  $\text{C}_5\text{D}_5\text{N}/\text{D}_2\text{O}$  (4/1)

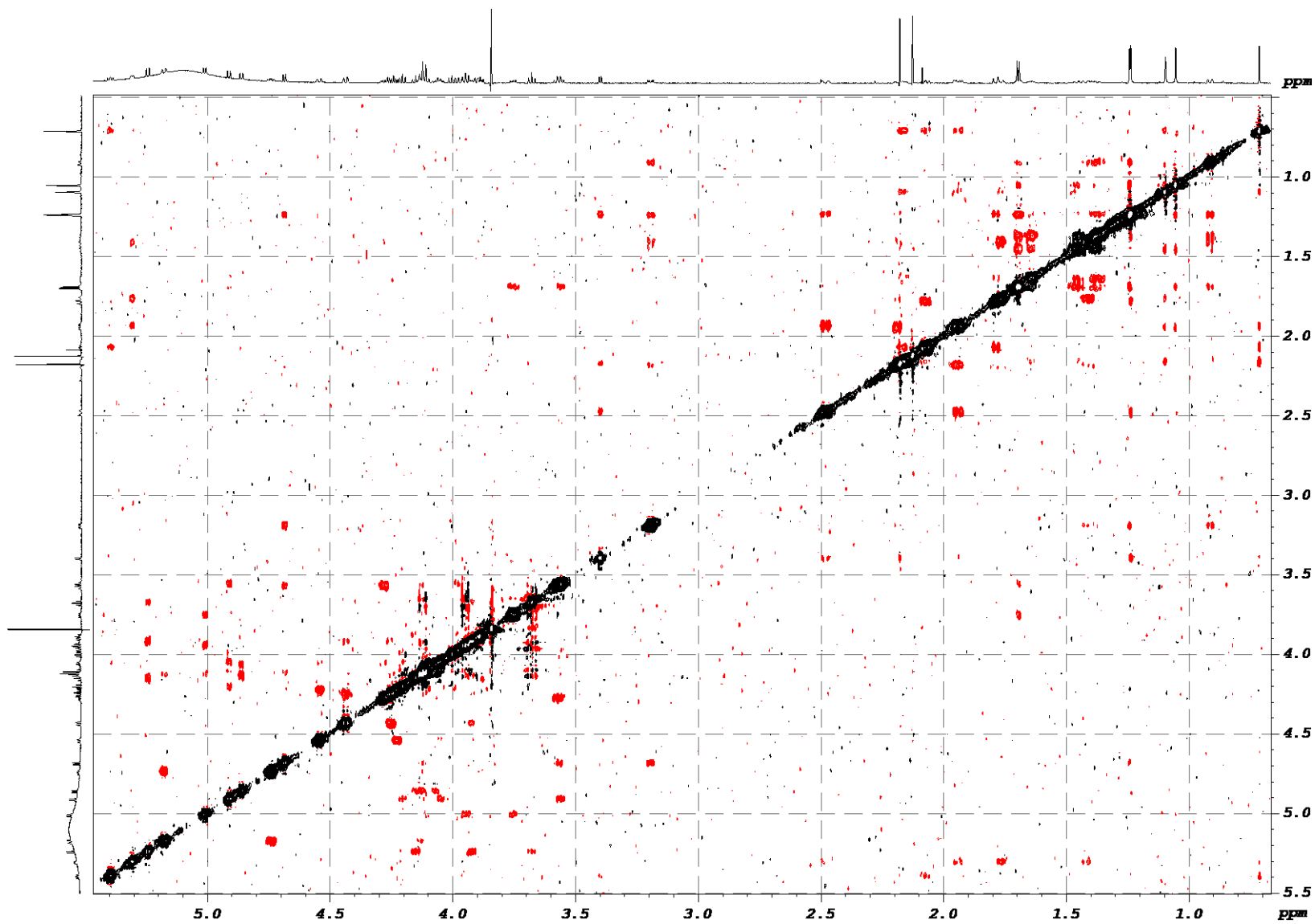


Figure S55. The ROESY (700.00 MHz) spectrum of kuriloside A (7) in C<sub>5</sub>D<sub>5</sub>N/D<sub>2</sub>O (4/1)

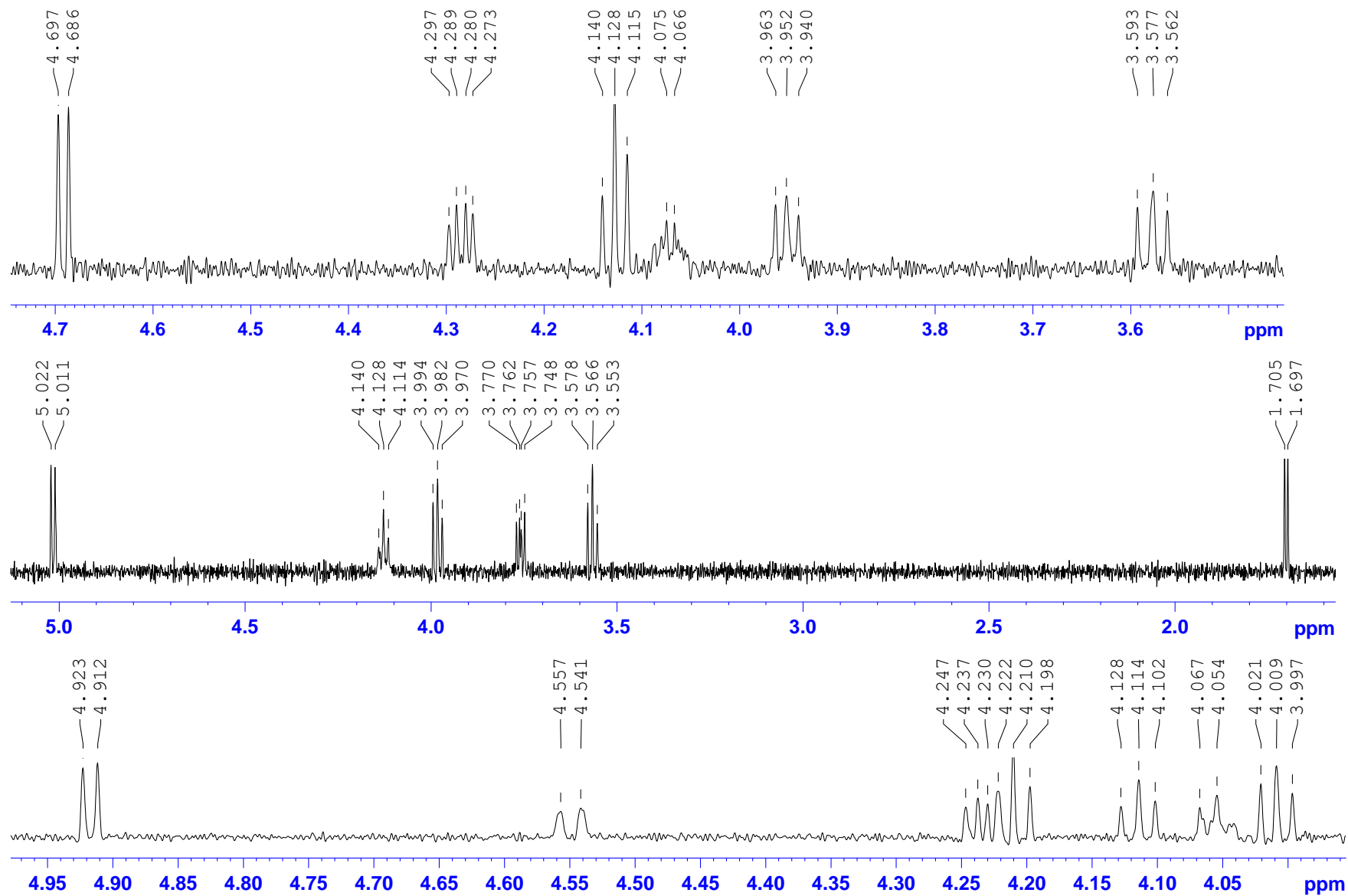


Figure S56. 1 D TOCSY (700.00 MHz) spectra of kuriloside A (7) in C<sub>5</sub>D<sub>5</sub>N/D<sub>2</sub>O (4/1)

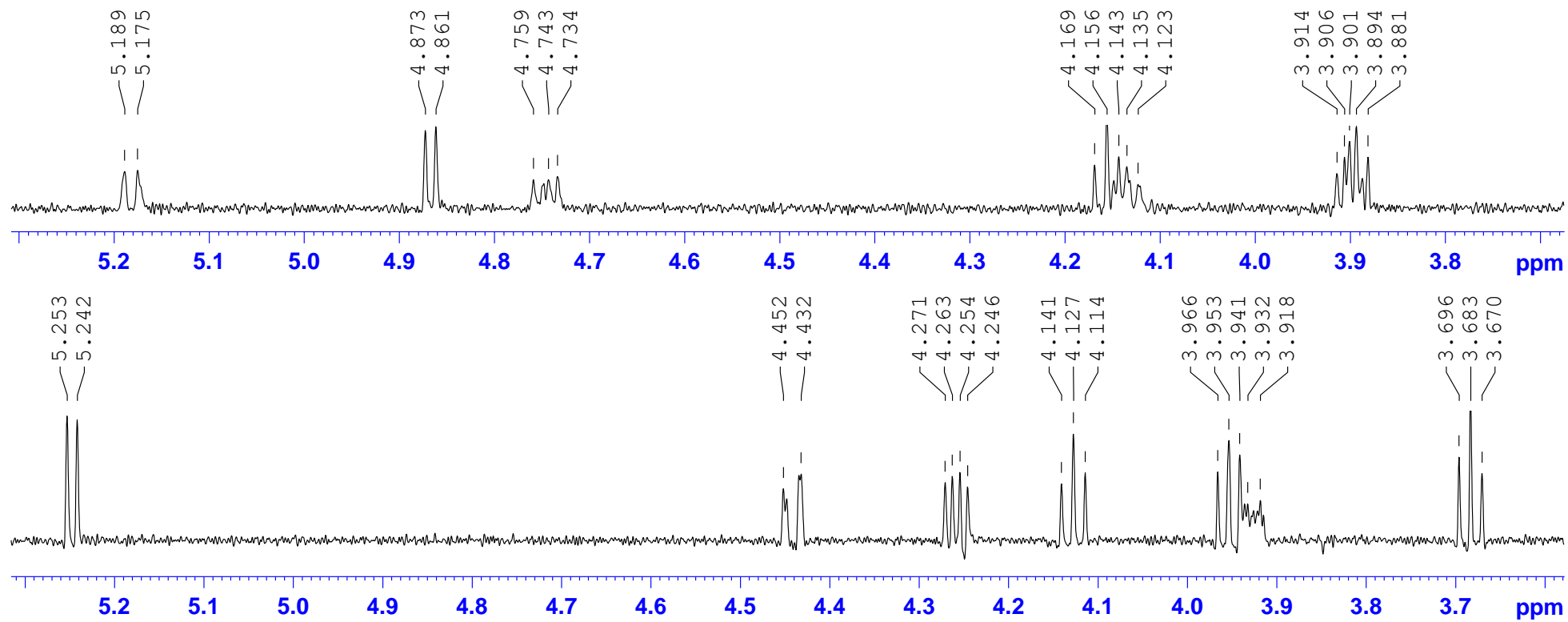


Figure S57. 1 D TOCSY (700.00 MHz) spectra of kuriloside A (7) in C<sub>5</sub>D<sub>5</sub>N/D<sub>2</sub>O (4/1)

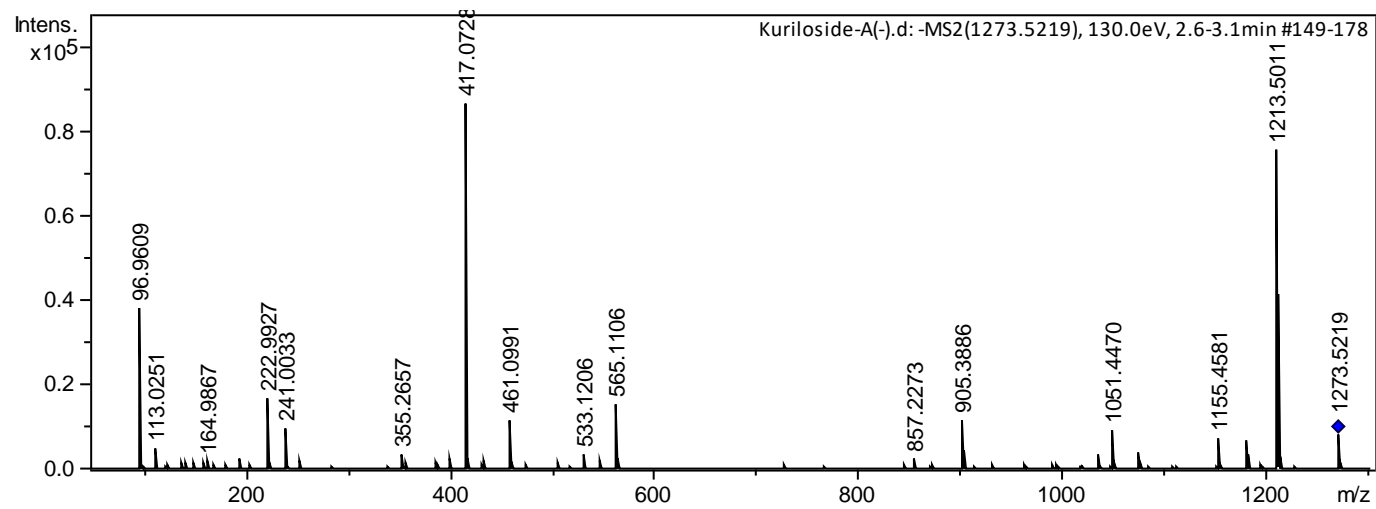
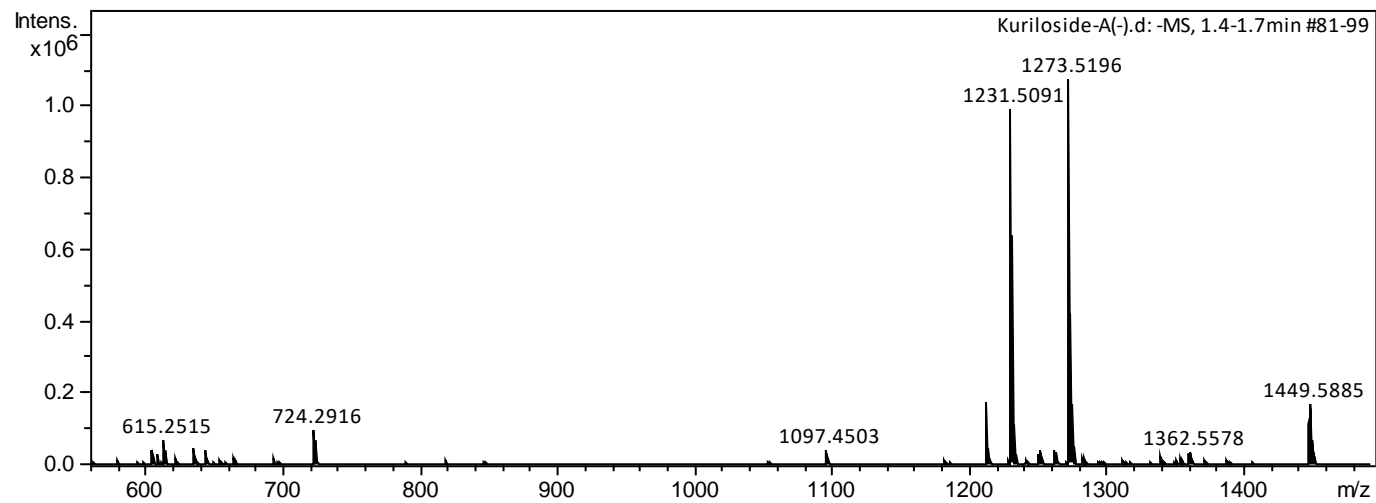


Figure S58. HR-ESI-MS and ESI-MS/MS spectra of kuriloside A (7)