

Unique Polyhalogenated Peptides from the Marine Sponge *Ircinia* sp.

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Experimental Procedure

Figure S1. Picture of the fresh sponge.

Figure S2. ¹H NMR spectrum of Haloircinamide A (**1**) 500 MHz, DMSO.

Figure S3. ¹³C NMR spectrum of Haloircinamide A (**1**) 125 MHz, DMSO.

Figure S4. g-HSQC spectrum of Haloircinamide A (**1**) 500 MHz, DMSO.

Figure S5. g-COSY spectrum of Haloircinamide A (**1**) 500 MHz, DMSO.

Figure S6. TOCSY spectrum of Haloircinamide A (**1**) 500 MHz, DMSO.

Figure S7. g-HMBC spectrum of Haloircinamide A (**1**) 500 MHz, DMSO.

Figure S8. ¹H NMR spectrum of Haloircinamide A (**1**) 500 MHz, CD₃OD.

Figure S9. ¹³C NMR spectrum of Haloircinamide A (**1**) 125 MHz, CD₃O).

Figure S10. g-HSQC spectrum of Haloircinamide A (**1**) 500 MHz, CD₃OD.

Figure S11. g-COSY spectrum of Haloircinamide A (**1**) 500 MHz, CD₃OD.

Figure S12. g-HMBC spectrum of Haloircinamide A (**1**) 500 MHz, CD₃OD.

Figure S13. ¹H NMR spectrum of Haloircinamide A (**1**) 500 MHz, CD₃OH.

Figure S14. g-COSY spectrum of Haloircinamide A (**1**) 500 MHz, CD₃OH.

Figure S15. g-HMBC spectrum of Haloircinamide A (**1**) 500 MHz, CD₃OH.

Figure S16. ROESY spectrum of Haloircinamide A (**1**) 500 MHz, CD₃OH.

Figure S17. ¹H NMR spectrum of Seribunamide A (**2**) 500 MHz, DMSO.

Figure S18. ¹³C NMR spectrum of Seribunamide A (**2**) 125 MHz, DMS).

Figure S19. g-HSQC spectrum of Seribunamide A (**2**) 500 MHz, DMSO.

Figure S20. g-COSY spectrum of Seribunamide A (**2**) 500 MHz, DMSO.

Figure S21. g-HMBC spectrum of Seribunamide A (**2**) 500 MHz, DMSO.

Figure S22. ROESY spectrum of Seribunamide A (**2**) 500 MHz, DMSO.

Figure S23. ¹H NMR spectrum of Seribunamide A (**2**) 500 MHz, CD₃OD.

Figure S24. ¹³C NMR spectrum of Seribunamide A (**2**) 125 MHz, CD₃OD.

Figure S25. g-HSQC spectrum of Seribunamide A (**2**) 500 MHz, CD₃OD.

Figure S26. g-COSY spectrum of Seribunamide A (**2**) 500 MHz, CD₃OD.

Figure S27. TOCSY spectrum of Seribunamide A (**2**) 500 MHz, CD₃OD.

Figure S28. g-HMBC spectrum of Seribunamide A (**2**) 500 MHz, CD₃OD.

Figure S29. ROESY spectrum of Seribunamide A (**2**) 500 MHz, CD₃OD.

Figure S30. QTOFMS spectrum of Haloirciamide A (**1**) and fragments found

Figure S31. Marfey of Haloirciamide A (**1**) and iSer standards

Figure S32. Marfey of Haloirciamide A (**1**) and Asp standards

Figure S33. Marfey of Haloirciamide A (**1**) and Dap standards

Figure S34. Marfey of Seribunamide A (2) and Ile standards

Figure S35. Marfey of Seribunamide A (2)

Figure S36. Marfey of Seribunamide A (2). Pro standards

Figure S37. Marfey of Seribunamide A (2). NMeLeu standards

Figure S38. Marfey of Seribunamide A (2). Glu standards

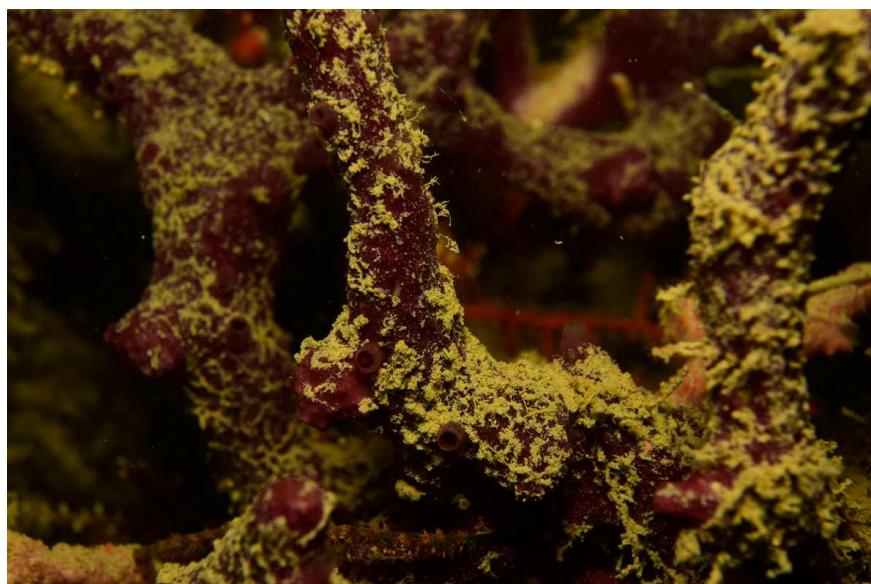


Figure S1. Picture of the fresh sponge.

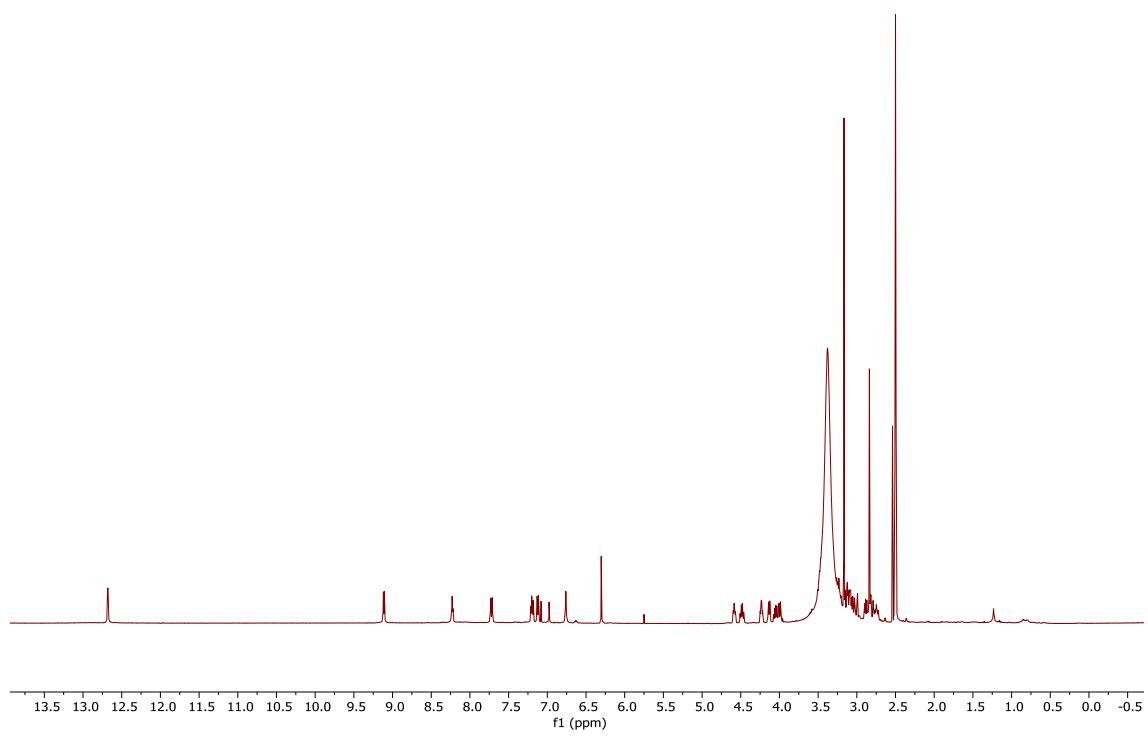


Figure S2 ¹H NMR spectrum of Haloircinamide A (**1**) 500 MHz, DMSO.

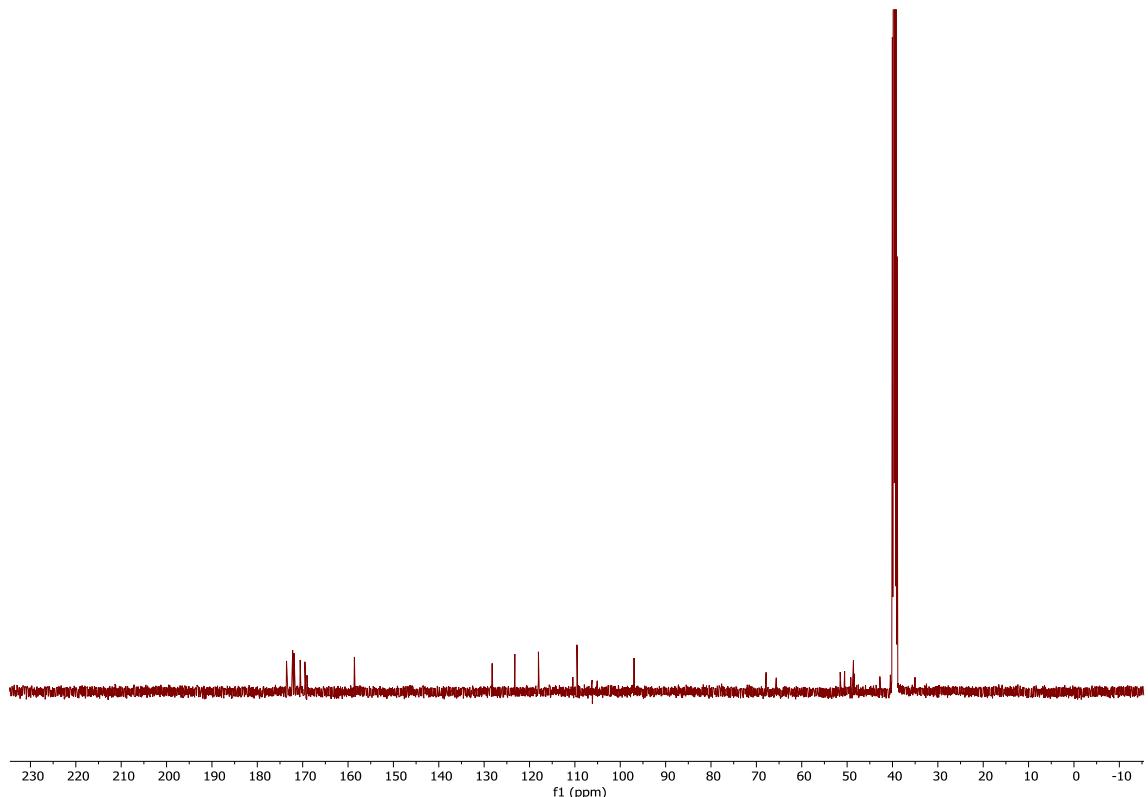


Figure S3 ¹³C NMR spectrum of Haloircinamide A (**1**) 125 MHz, DMSO.

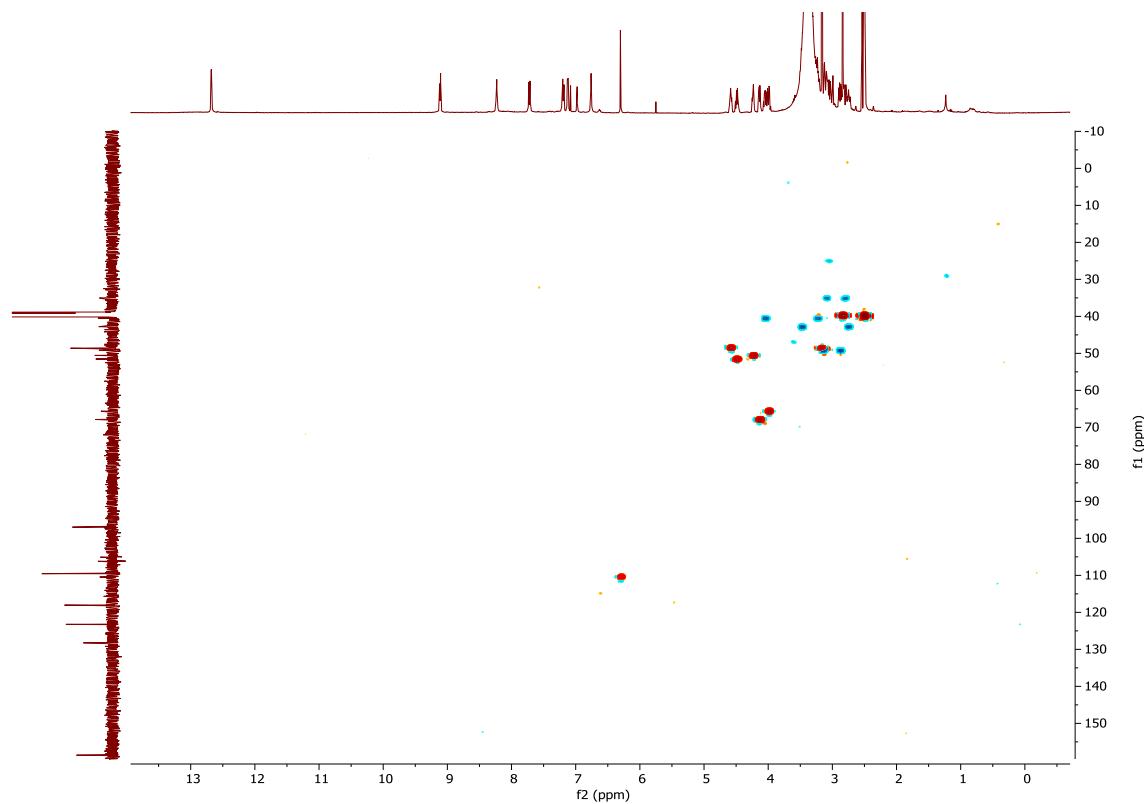


Figure S4 *g*-HSQC spectrum of Haloircinamide A (**1**) 500 MHz, DMSO

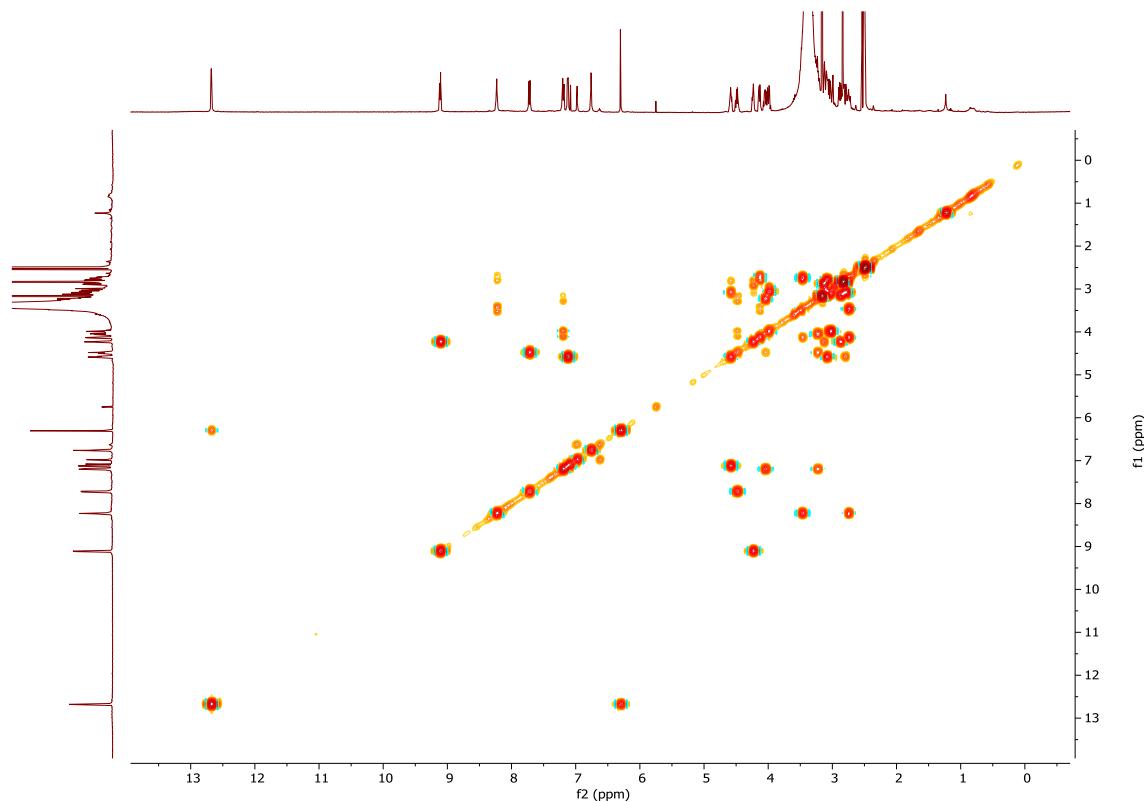


Figure S5 *g*-COSY spectrum of Haloircinamide A (**1**) 500 MHz, DMSO

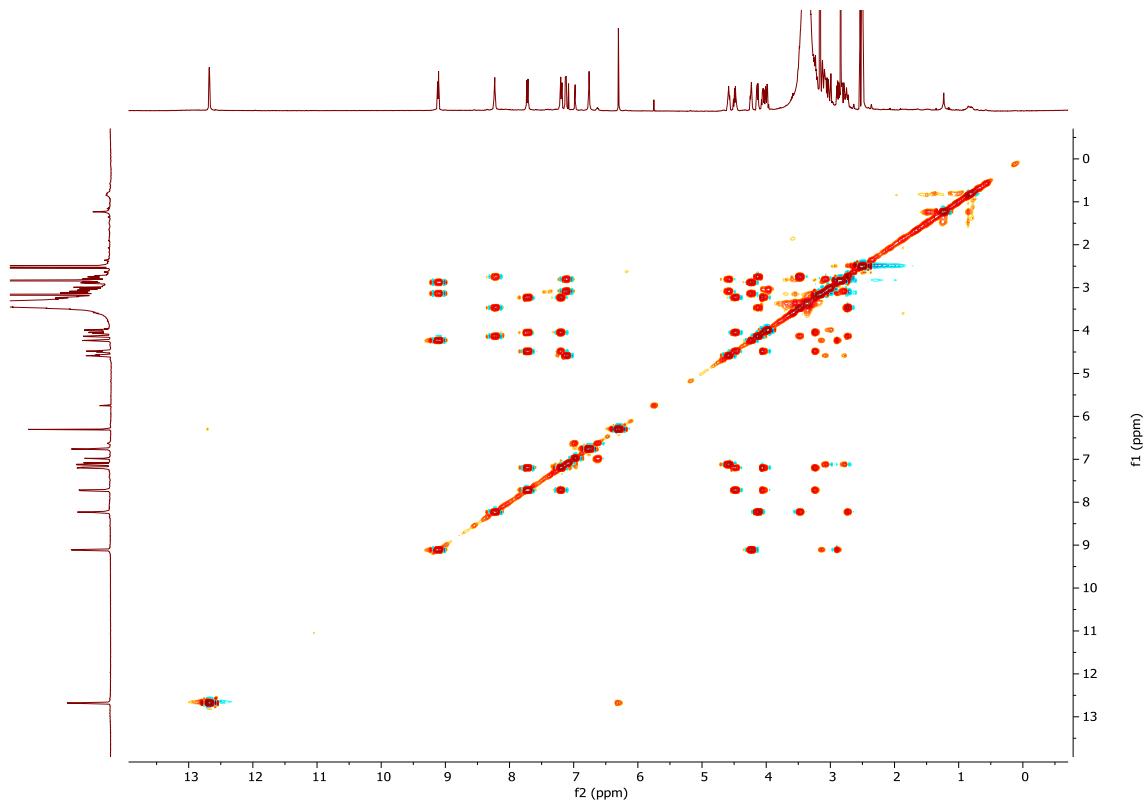


Figure S6 TOCSY spectrum of Haloircinamide A (**1**) 500 MHz, DMSO

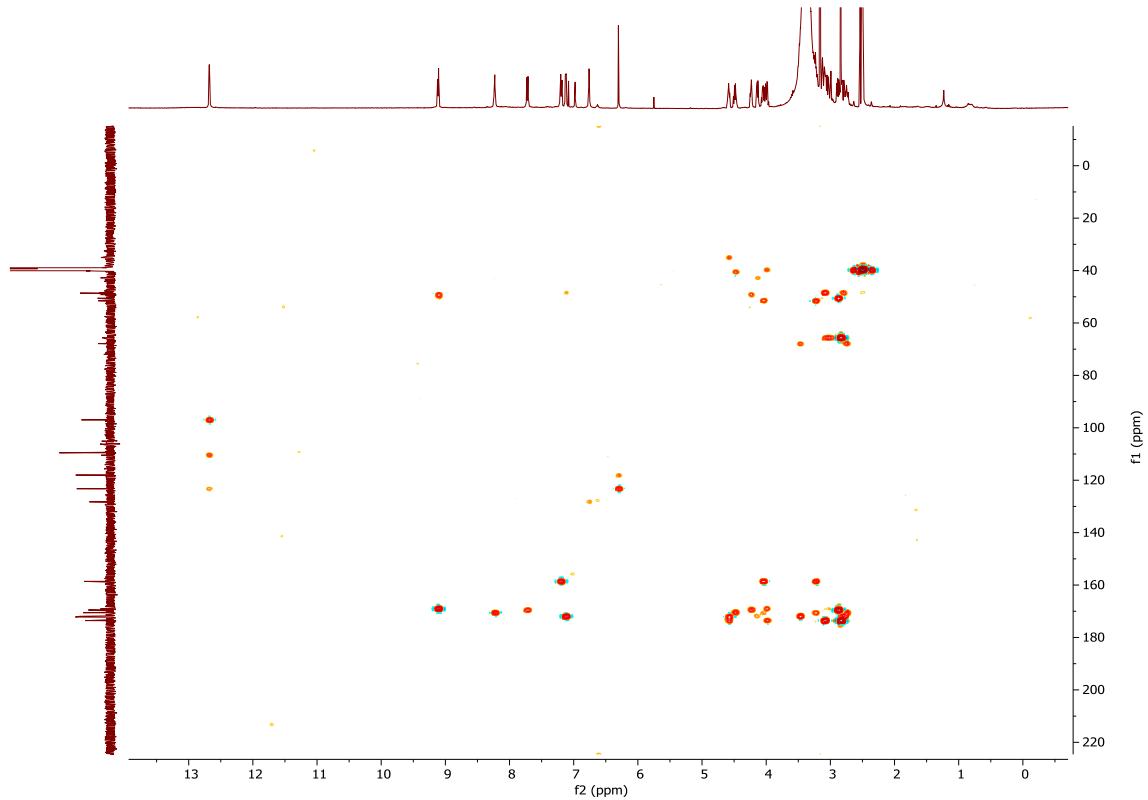


Figure S7 *g*-HMBC spectrum of Haloircinamide A (**1**) 500 MHz, DMSO

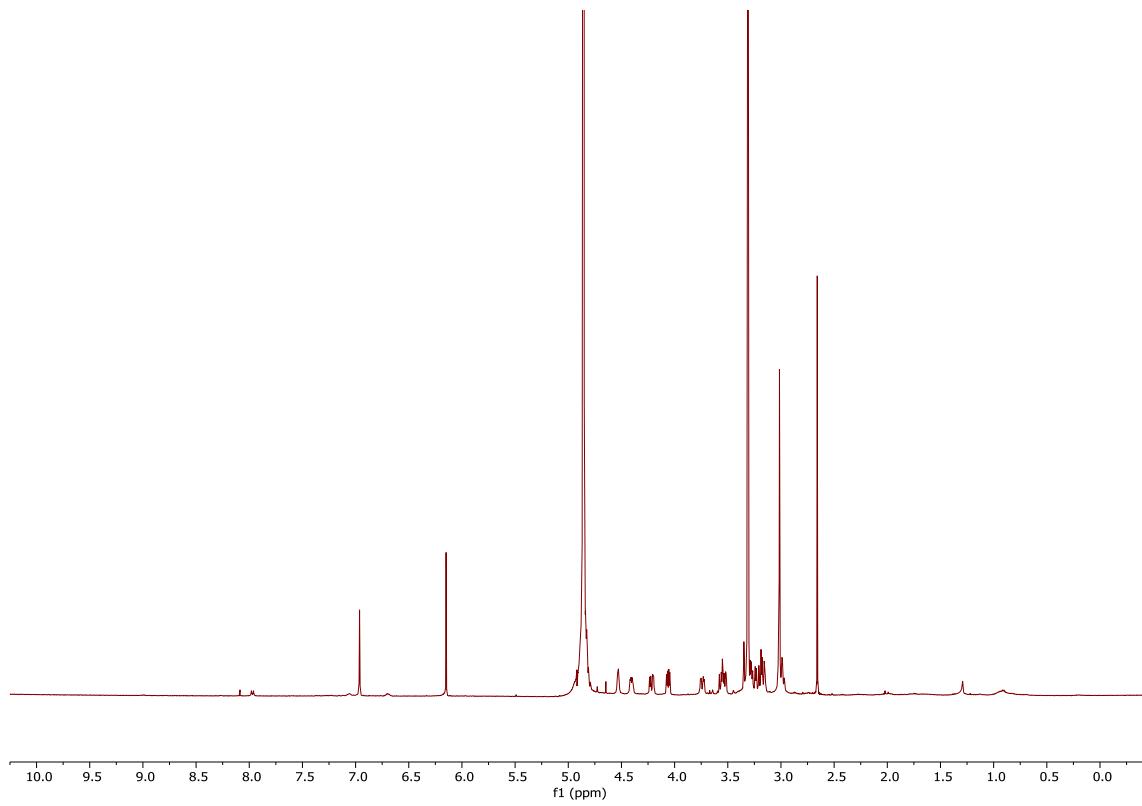


Figure S8 ¹H NMR spectrum of Haloircinamide A (**1**) 500 MHz, CD₃OD.

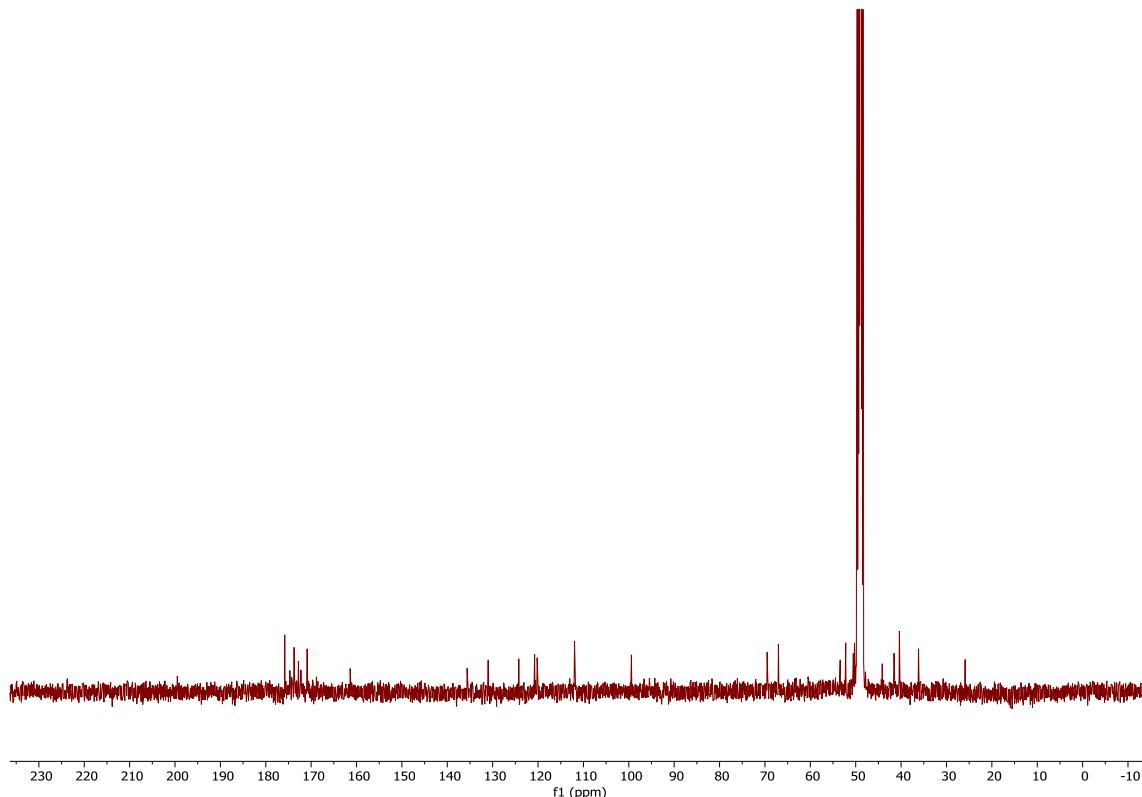


Figure S9 ¹³C NMR spectrum of Haloircinamide A (**1**) 125 MHz, CD₃OD.

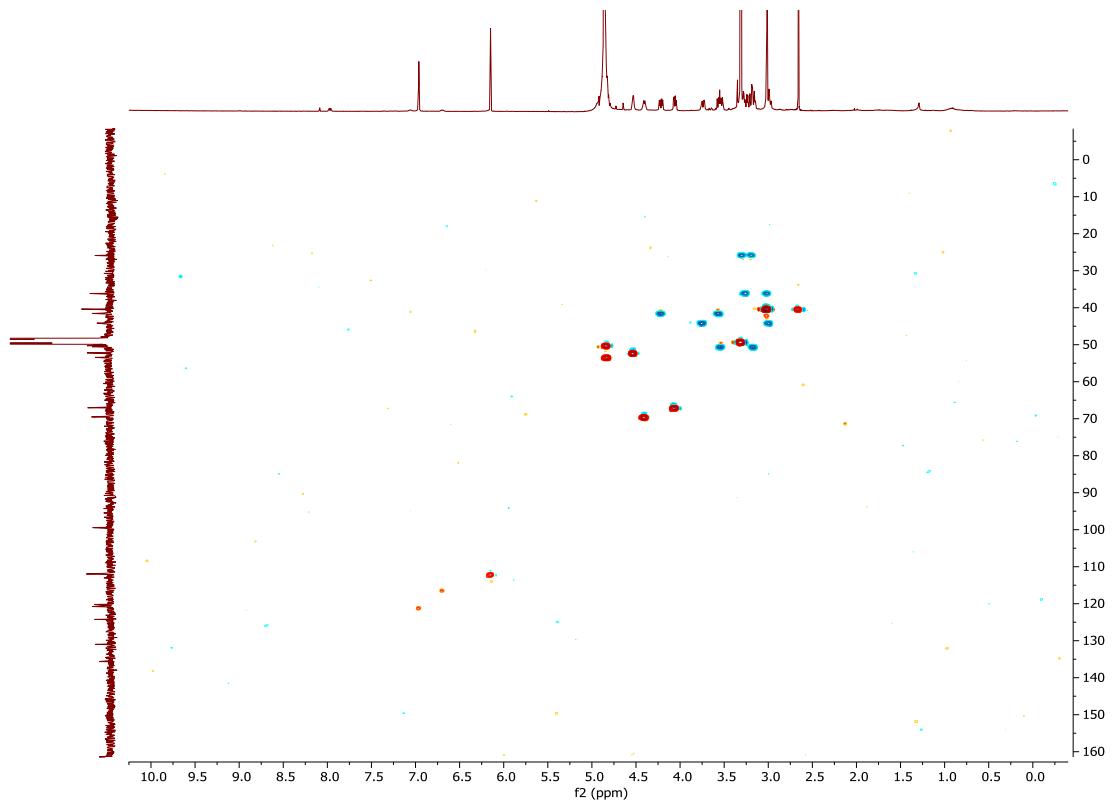


Figure S10 g-HSQC spectrum of Haloircinamide A (**1**) 500 MHz, CD_3OD

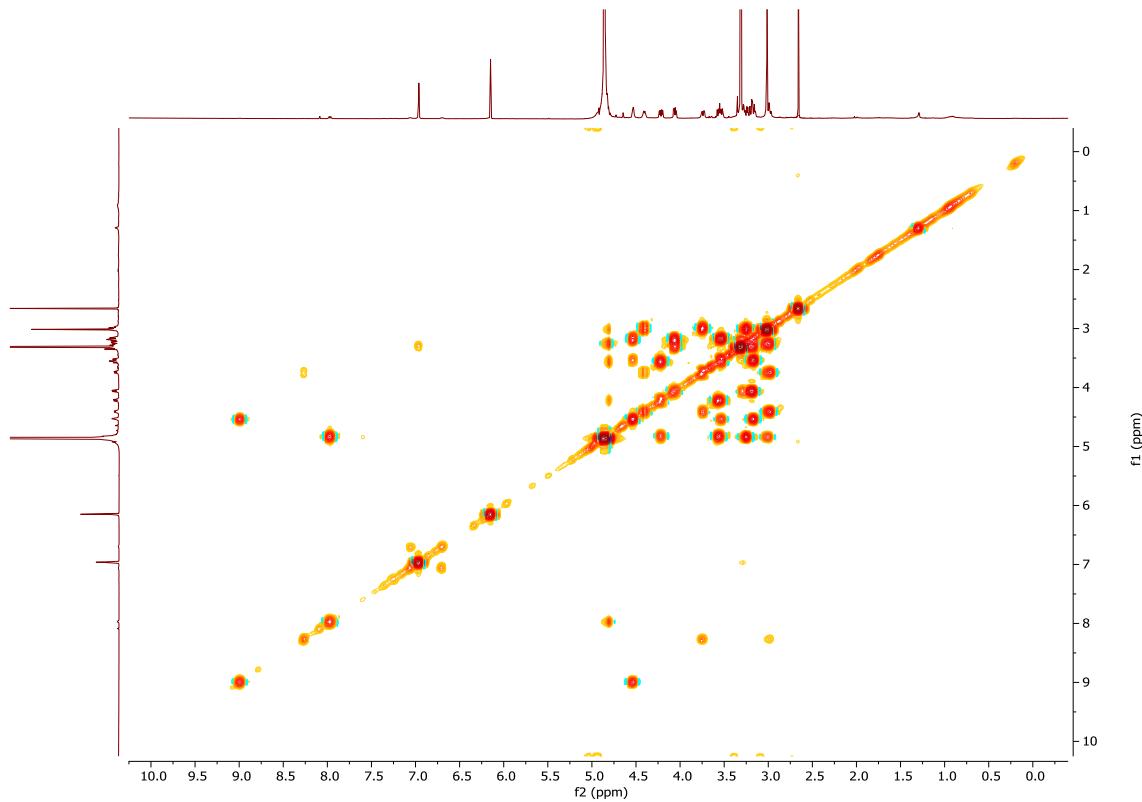


Figure S11 g-COSY spectrum of Haloircinamide A (**1**) 500 MHz, CD_3OD

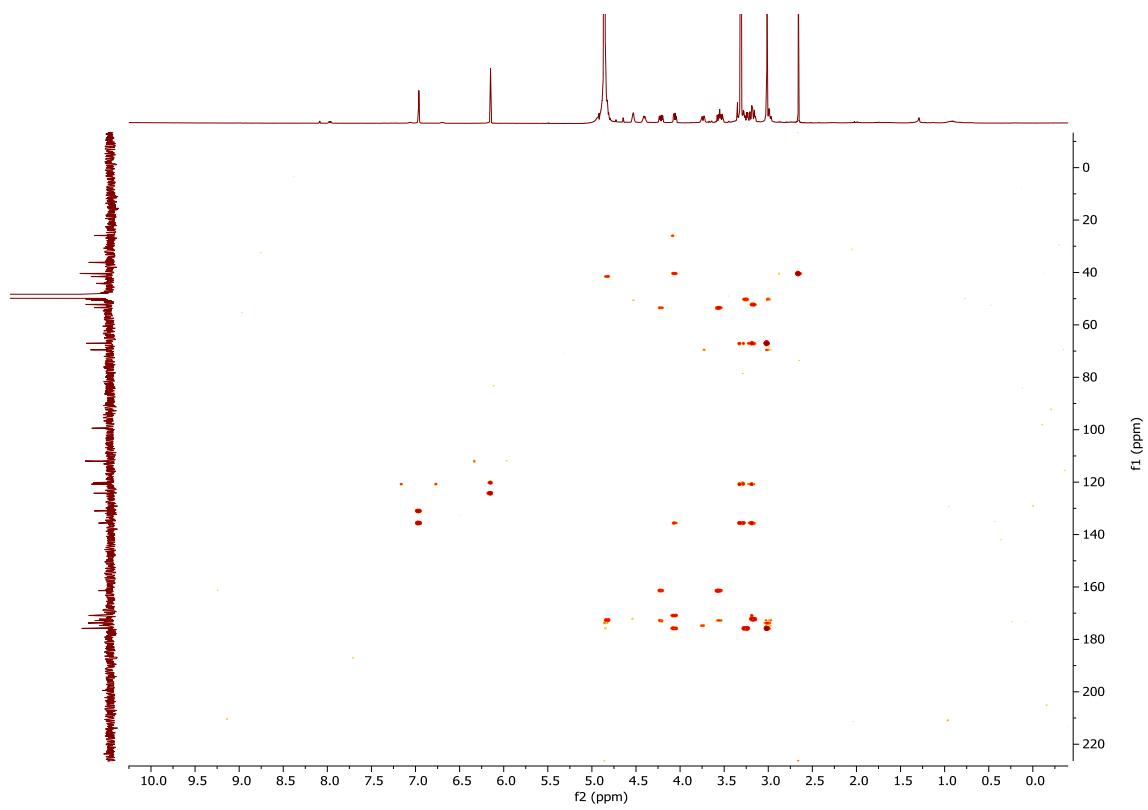


Figure S12 g-HMBC spectrum of Haloircinamide A (**1**) 500 MHz, CD₃OD

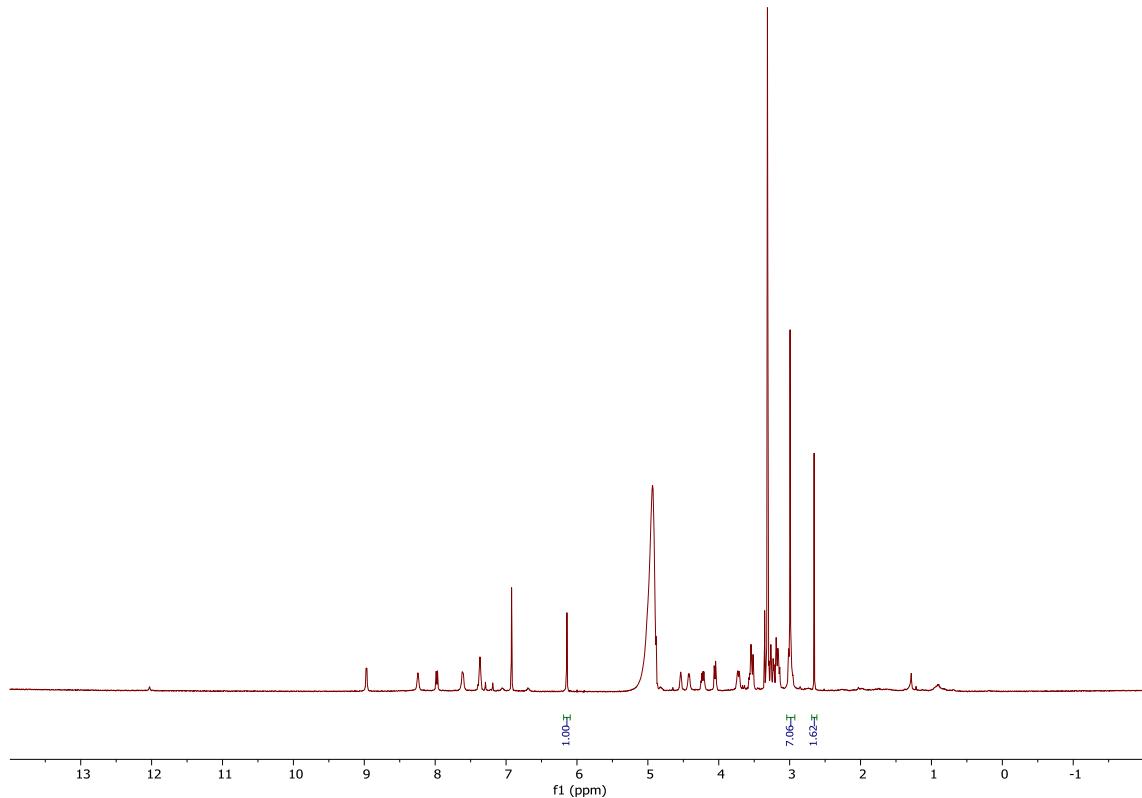


Figure S13 ¹H NMR spectrum of Haloircinamide A (**1**) 500 MHz, CD₃OH.

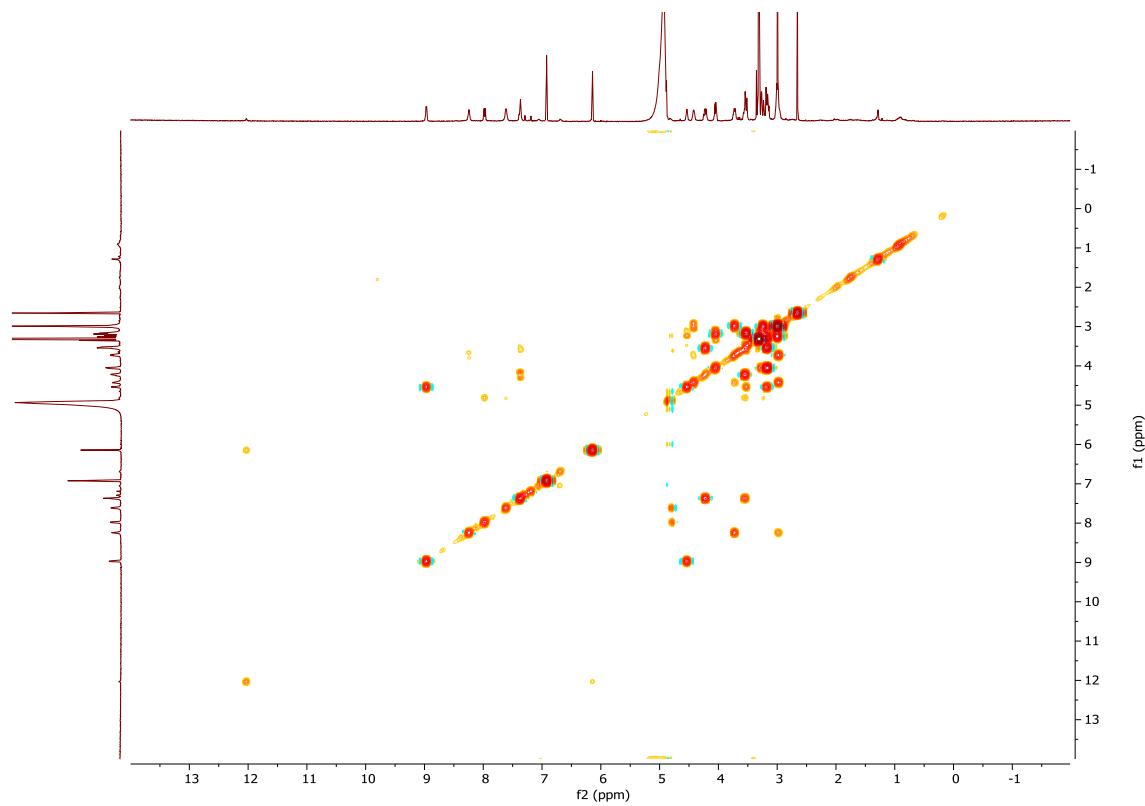


Figure S14 g-COSY spectrum of Haloircinamide A (**1**) 500 MHz, CD_3OH

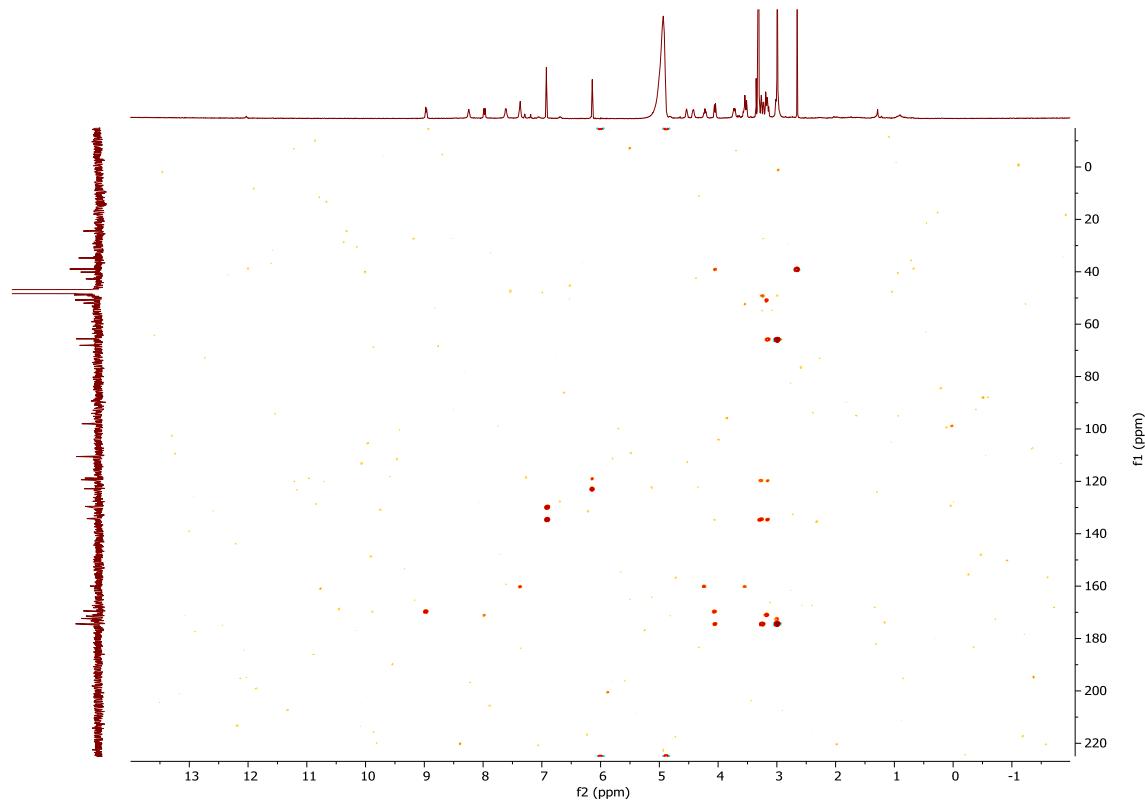


Figure S15 g-HMBC spectrum of Haloircinamide A (**1**) 500 MHz, CD_3OH

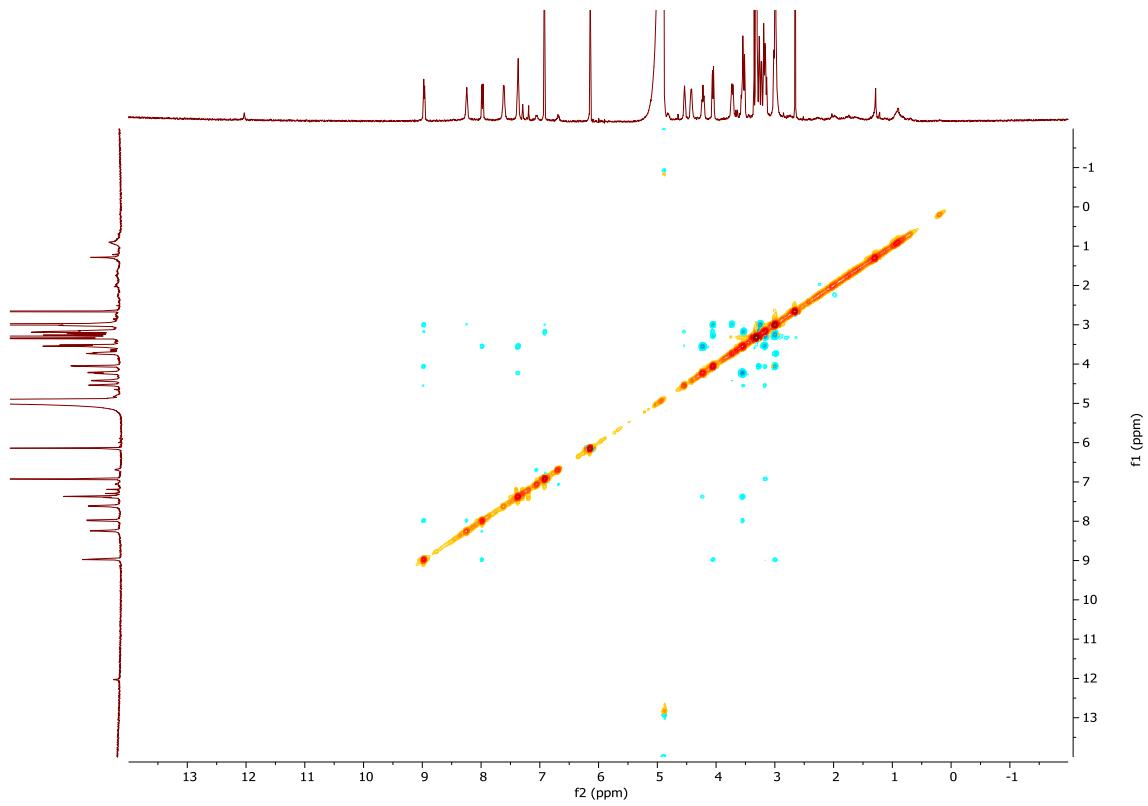


Figure S16 ROESY spectrum of Haloircinamide A (**1**) 500 MHz, CD₃OH

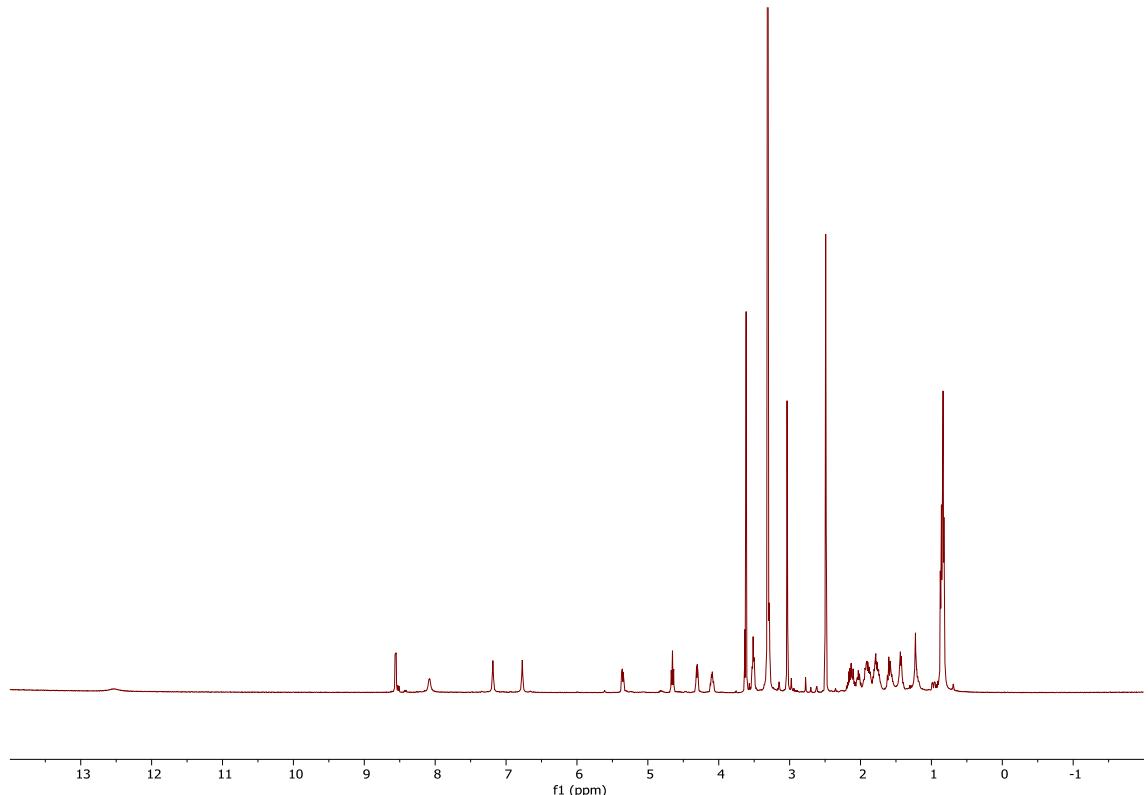


Figure S17 ¹H NMR spectrum of Seribunamide A (**2**), 500 MHz, DMSO

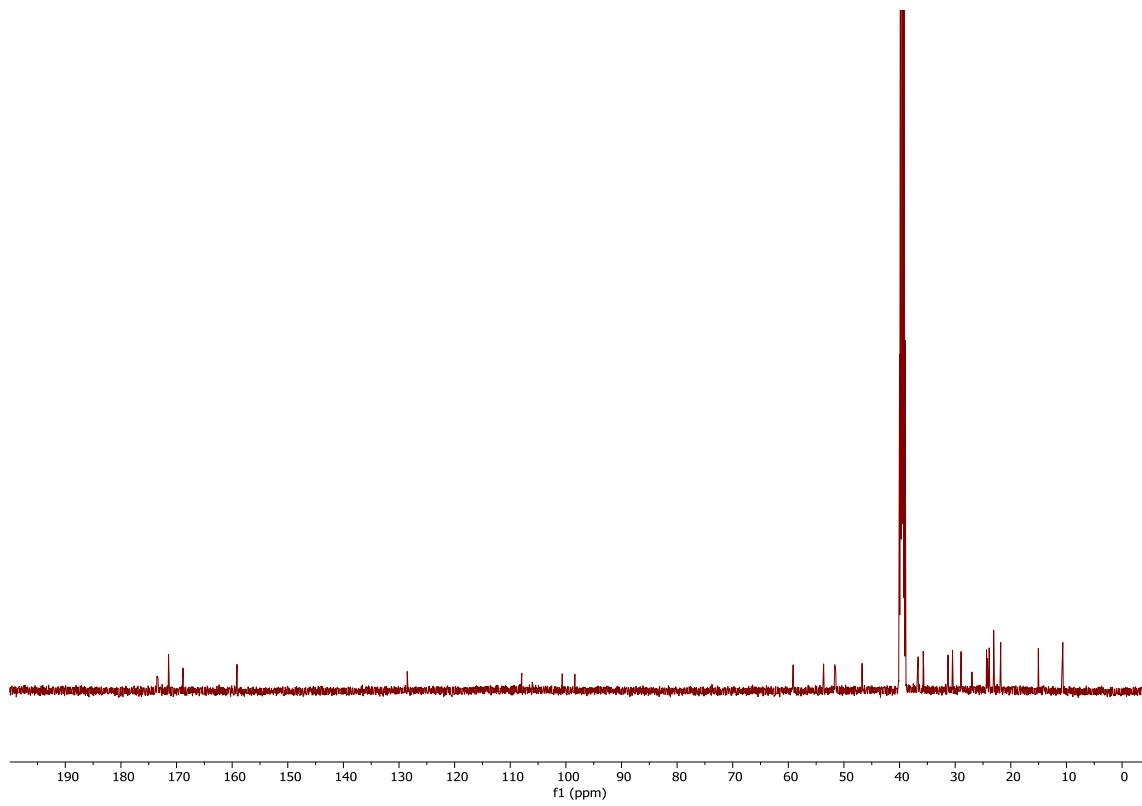


Figure S18 ^{13}C NMR spectrum of Seribunamide A (2), 125 MHz, DMSO

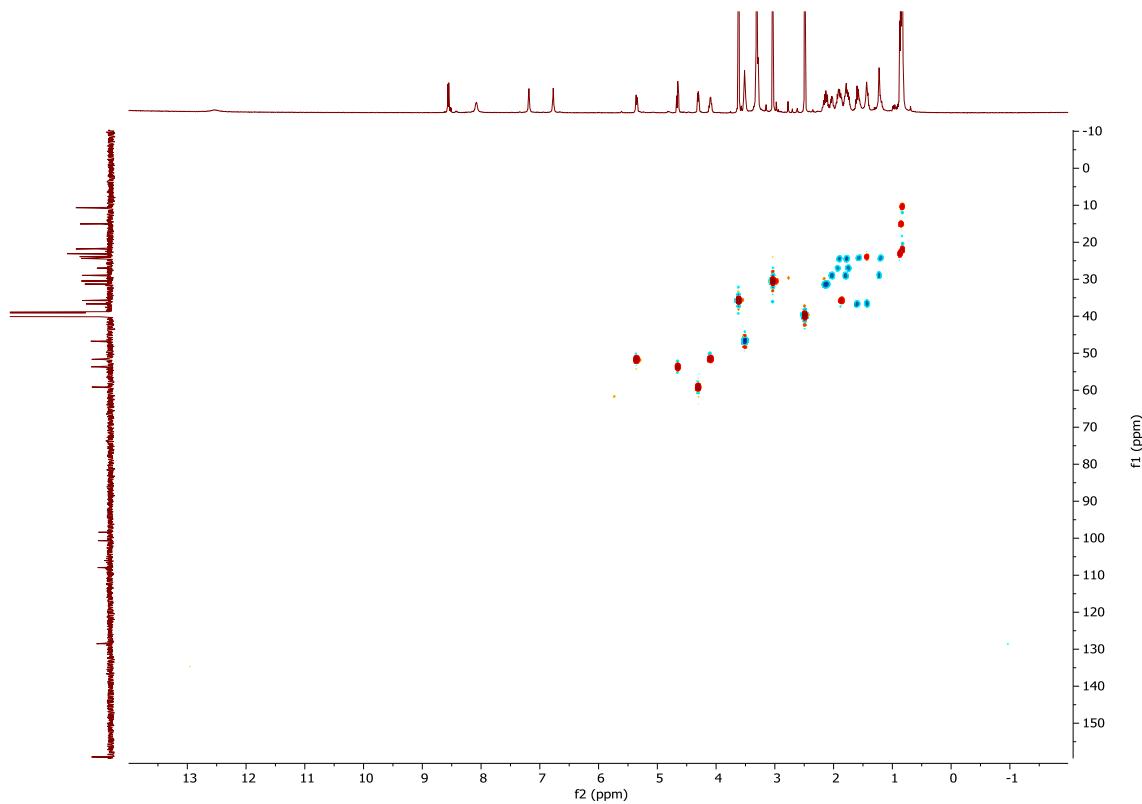


Figure S19 gHSQC spectrum of Seribunamide A (2), 500 MHz, DMSO

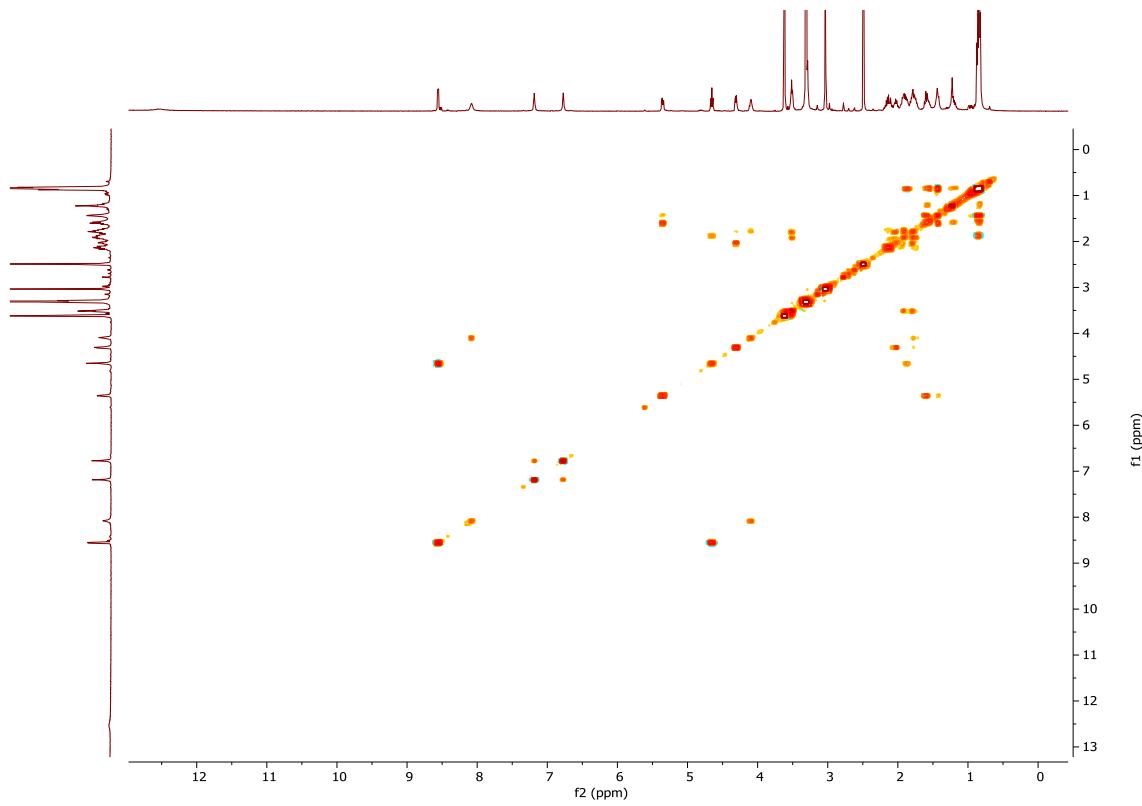


Figure S20 gCOSY spectrum of Seribunamide A (**2**), 500 MHz, DMSO

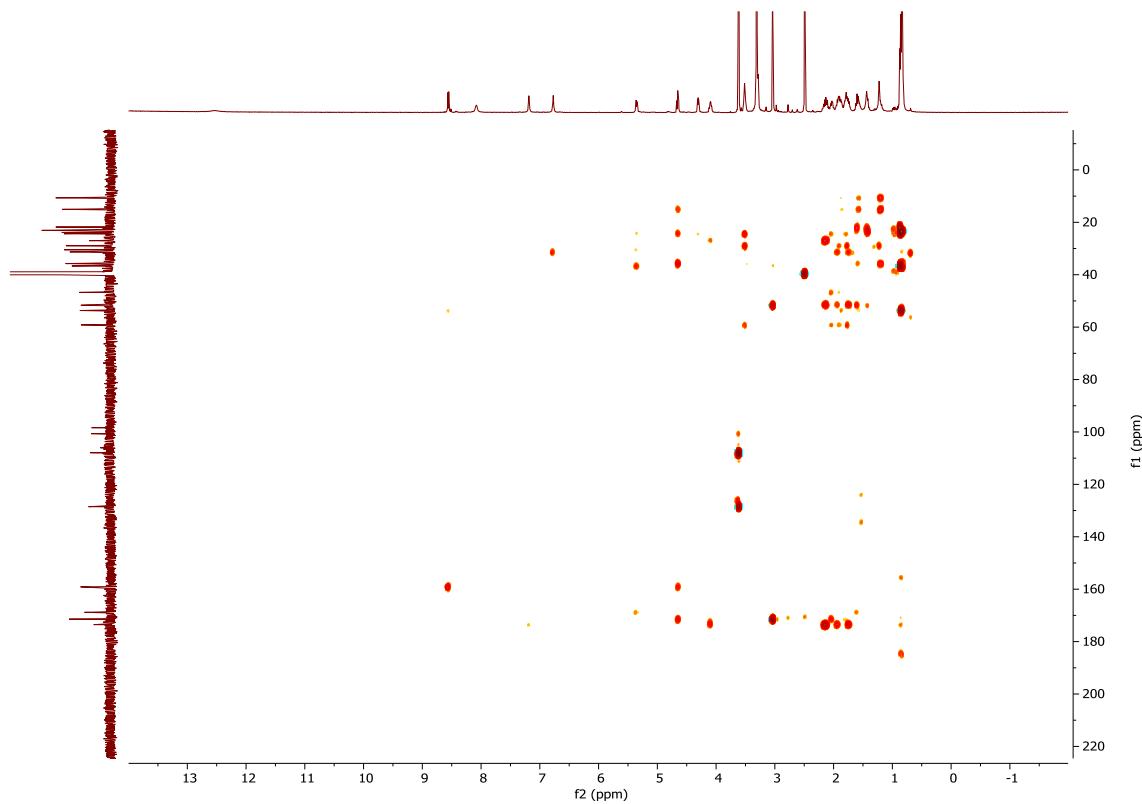


Figure S21 gHMBC spectrum of Seribunamide A (**2**), 500 MHz, DMSO

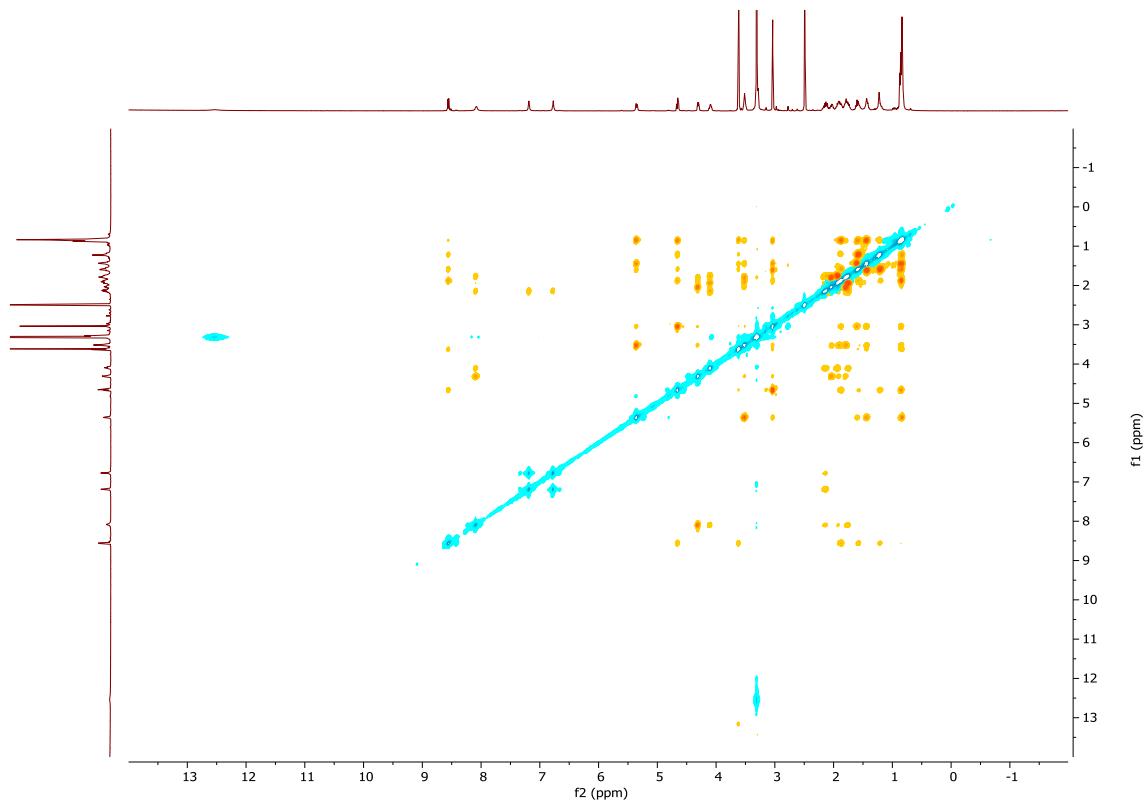


Figure S22 ROESY spectrum of Seribunamide A (**2**), 500 MHz, DMSO

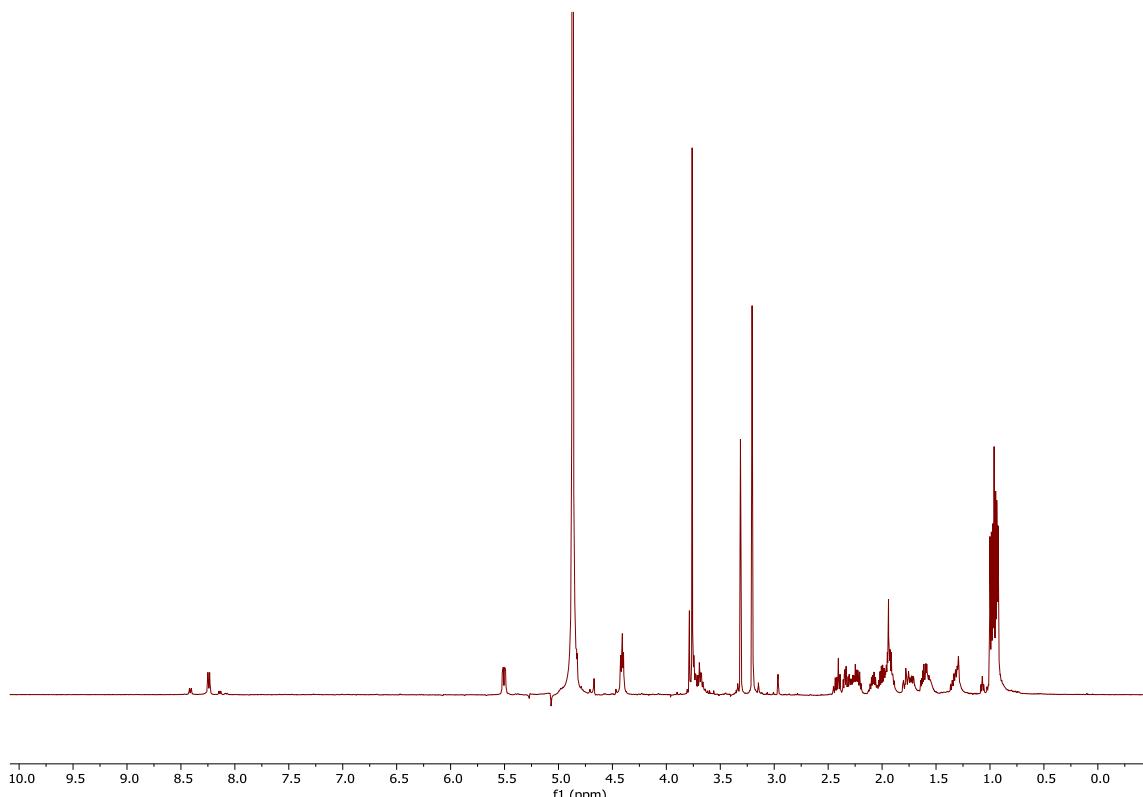


Figure S23 ^1H NMR spectrum of Seribunamide A (**2**), 500 MHz, CD_3OD

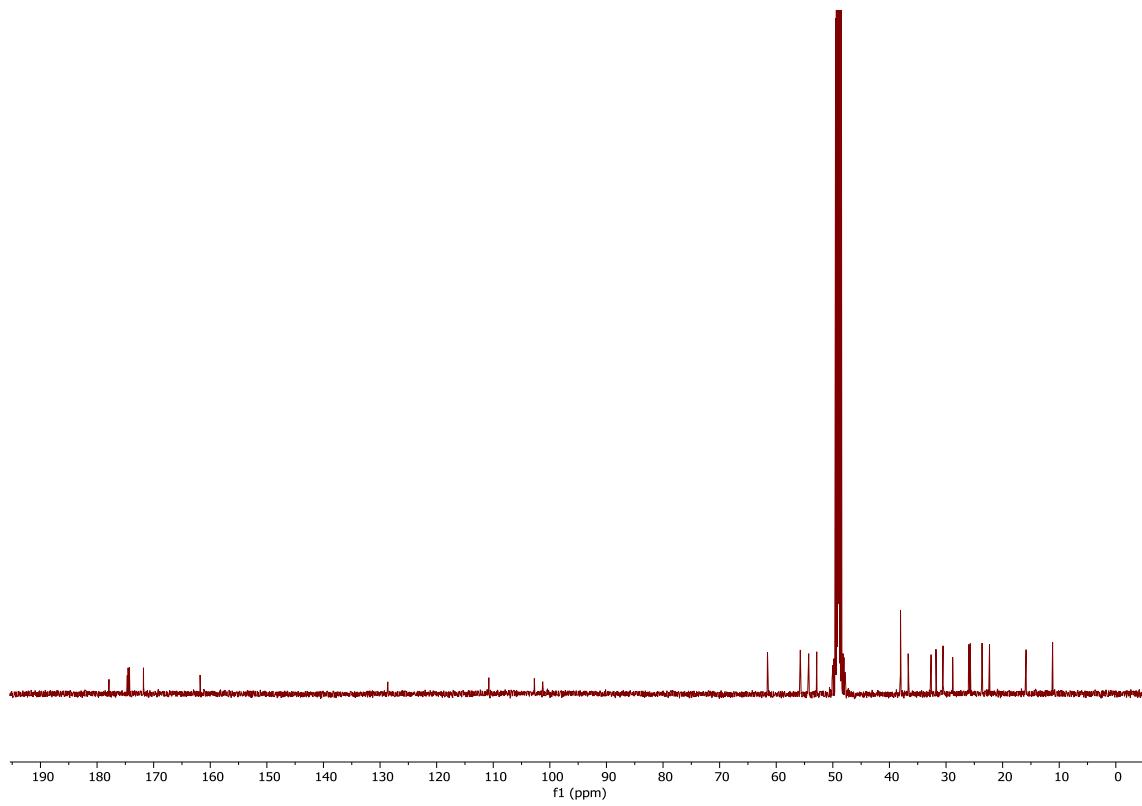


Figure S24 ^{13}C NMR spectrum of Seribunamide A (2), 125 MHz, CD_3OD

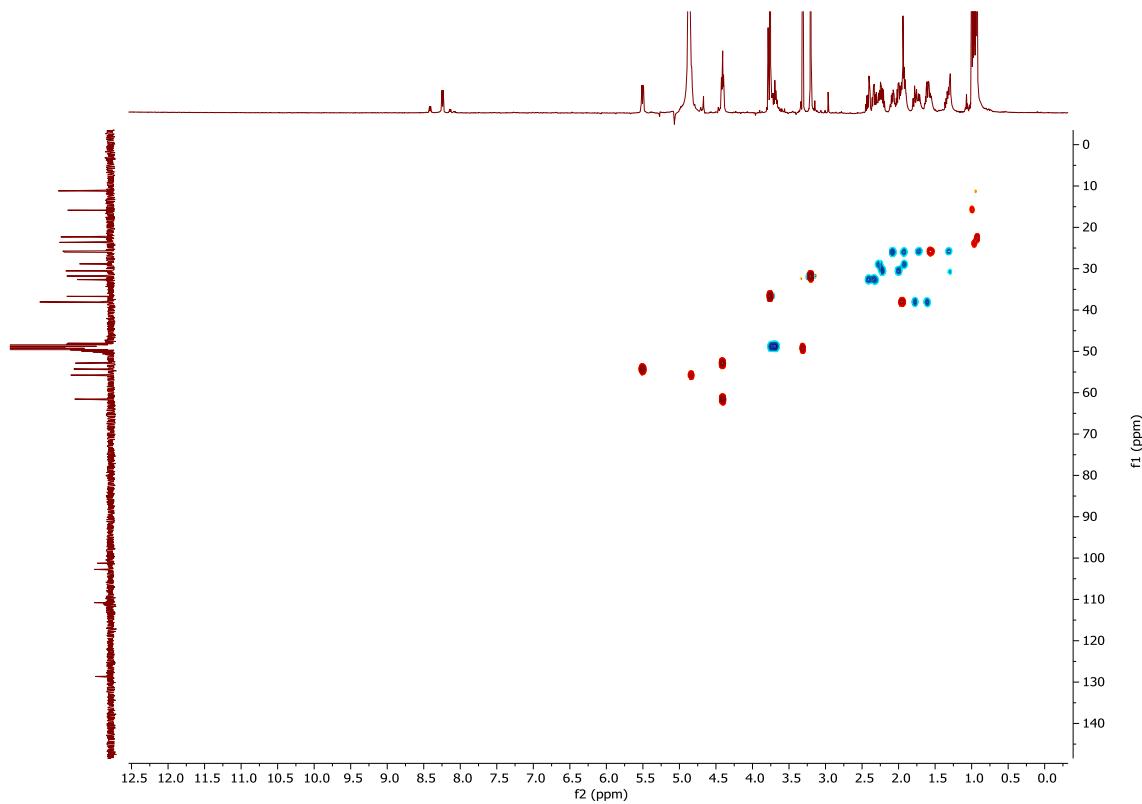


Figure S25 gHSQC spectrum of Seribunamide A (2), 500 MHz, CD_3OD

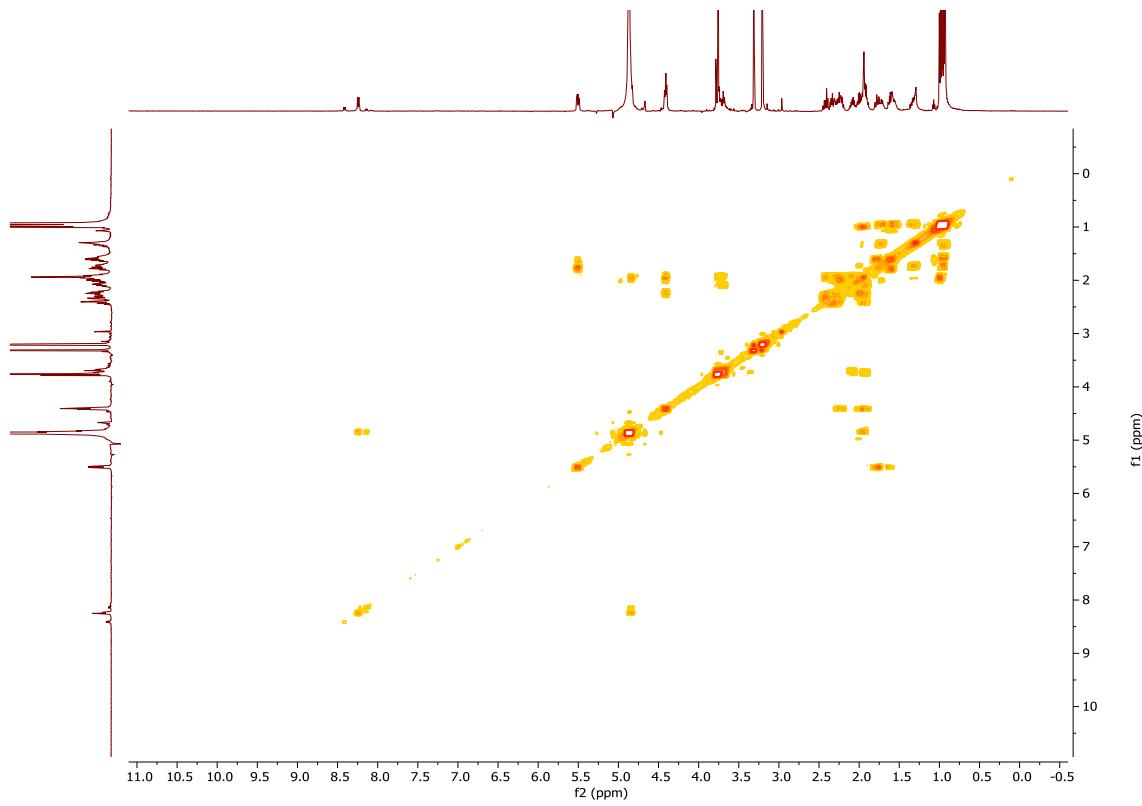


Figure S26 gCOSY spectrum of Seribunamide A (**2**), 500 MHz, CD_3OD

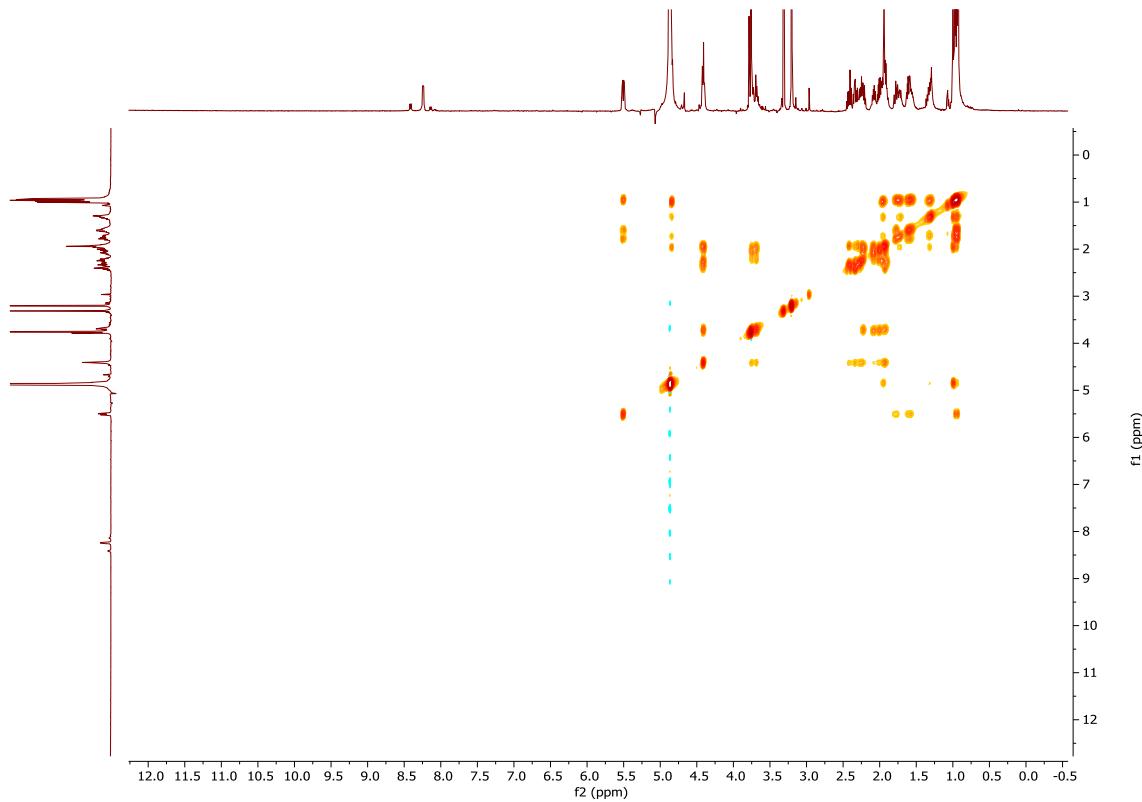


Figure S27 TOCSY spectrum of Seribunamide A (**2**), 500 MHz, CD_3OD

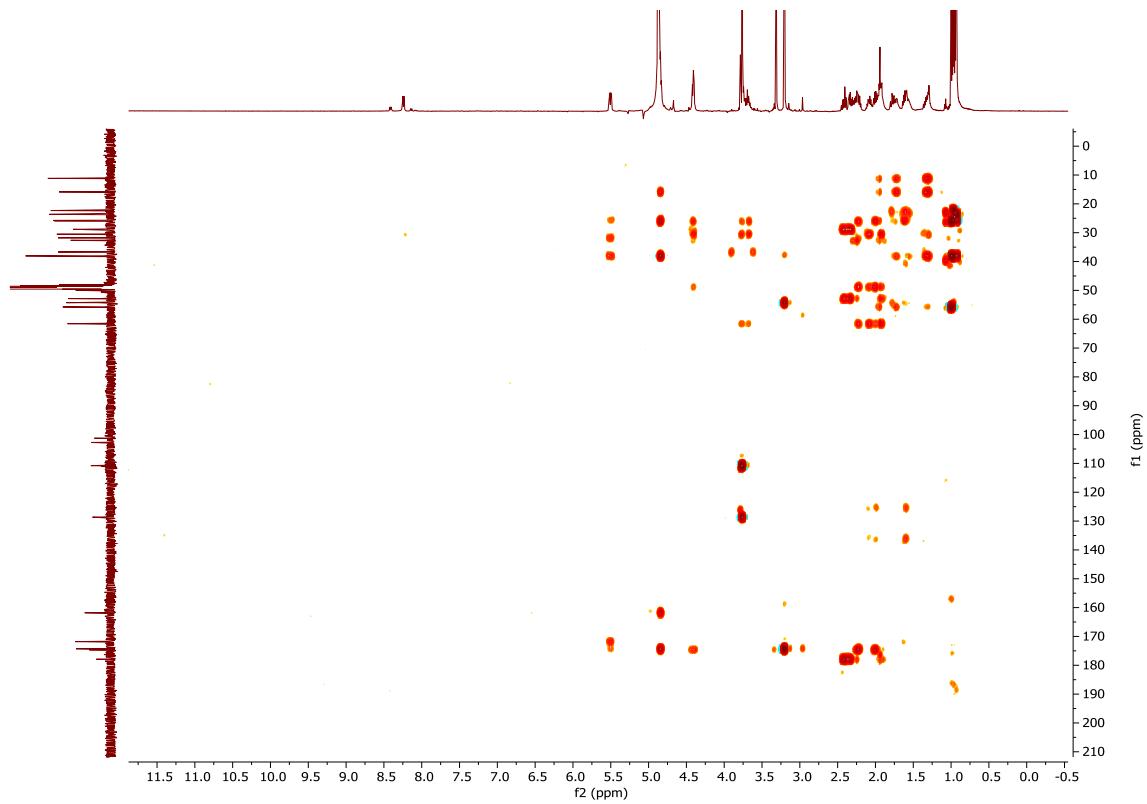


Figure S28 gHMBC spectrum of Seribunamide A (**2**), 500 MHz, CD_3OD

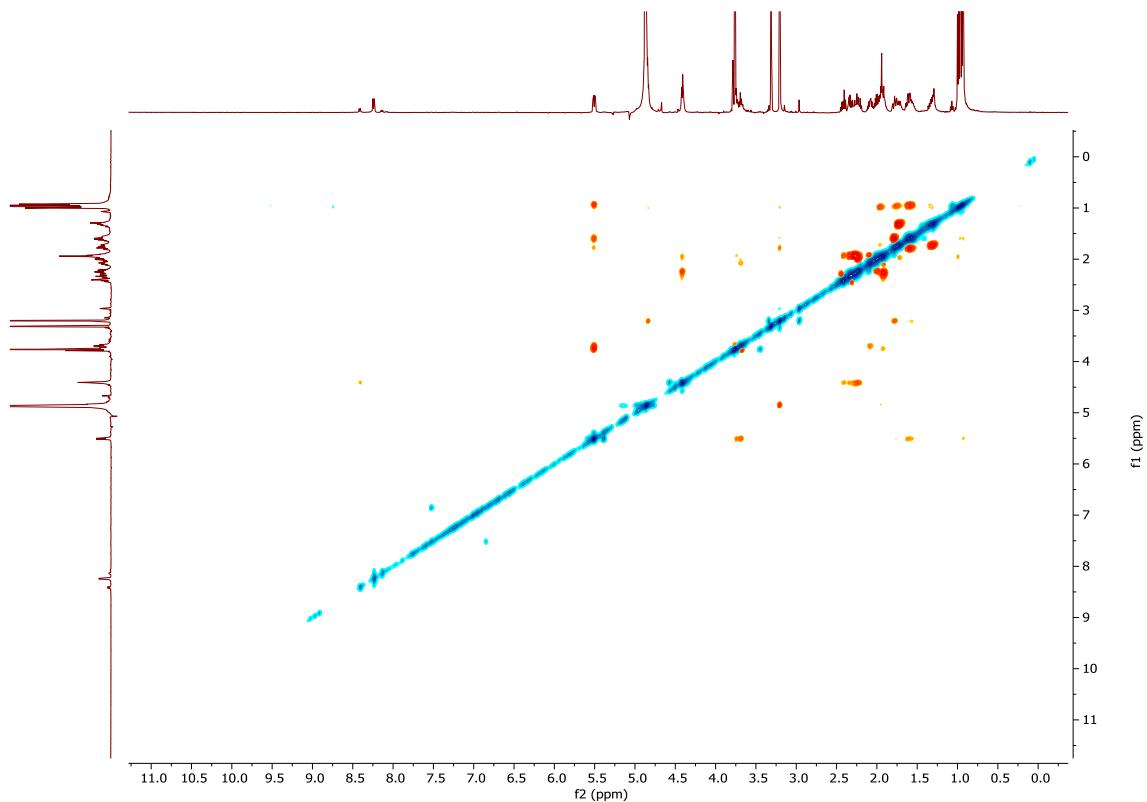
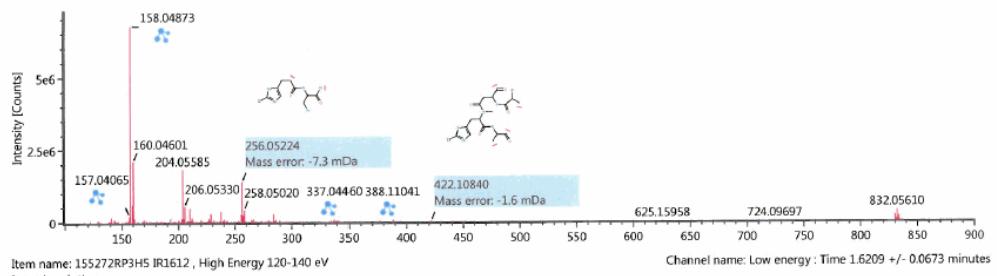


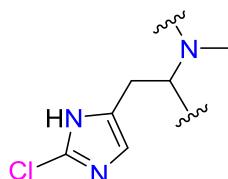
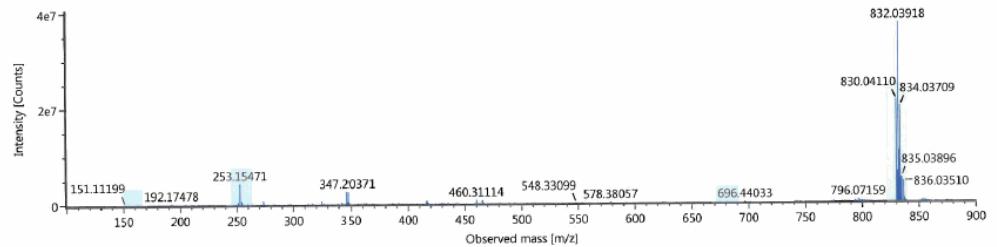
Figure S29 ROESY spectrum of Seribunamide A (**2**), 500 MHz, CD_3OD

Item name: 155272RP3HS IR1612 , High Energy 60-80 eV
 Item description:



Item name: 155272RP3HS IR1612 , High Energy 120-140 eV

Item description:



Observed m/z 158.0487
 Expected m/z 158.0480
 Mass error 4.92 ppm

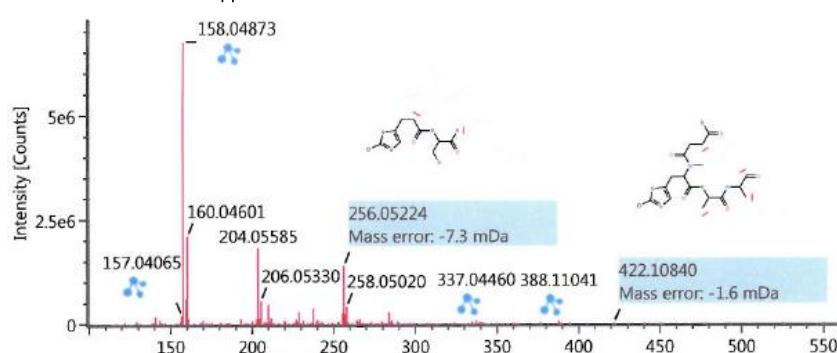


Figure S30 QTOFMS spectrum of Haloirciamide A (**1**) and fragments found

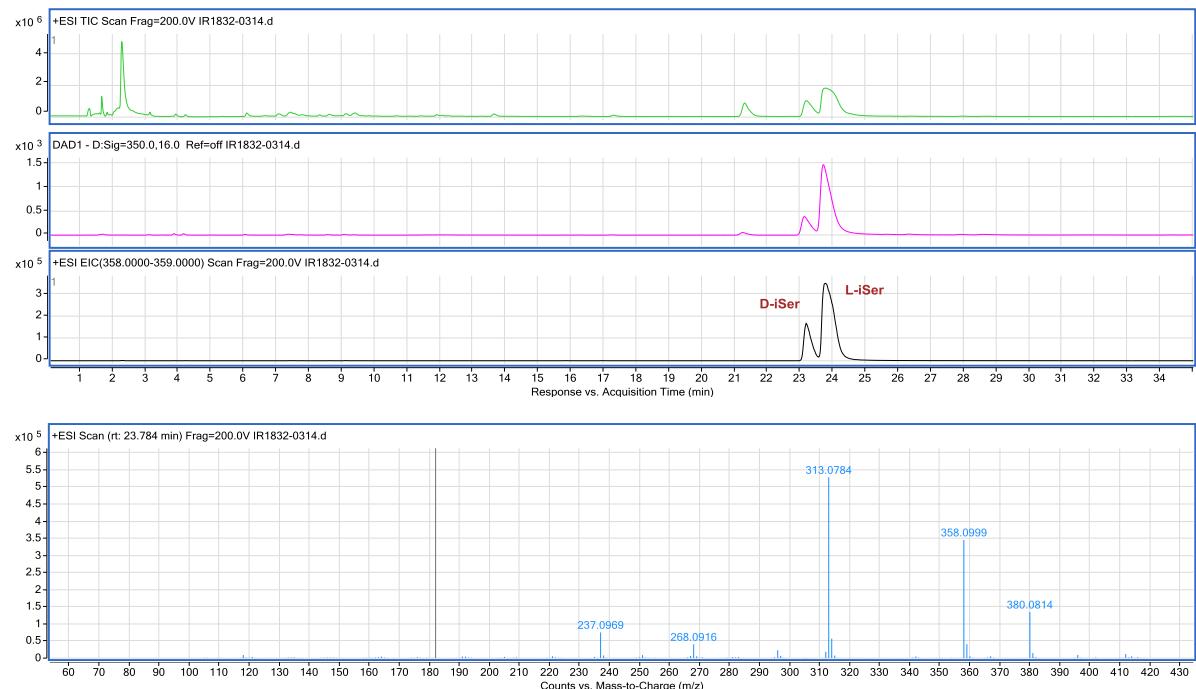
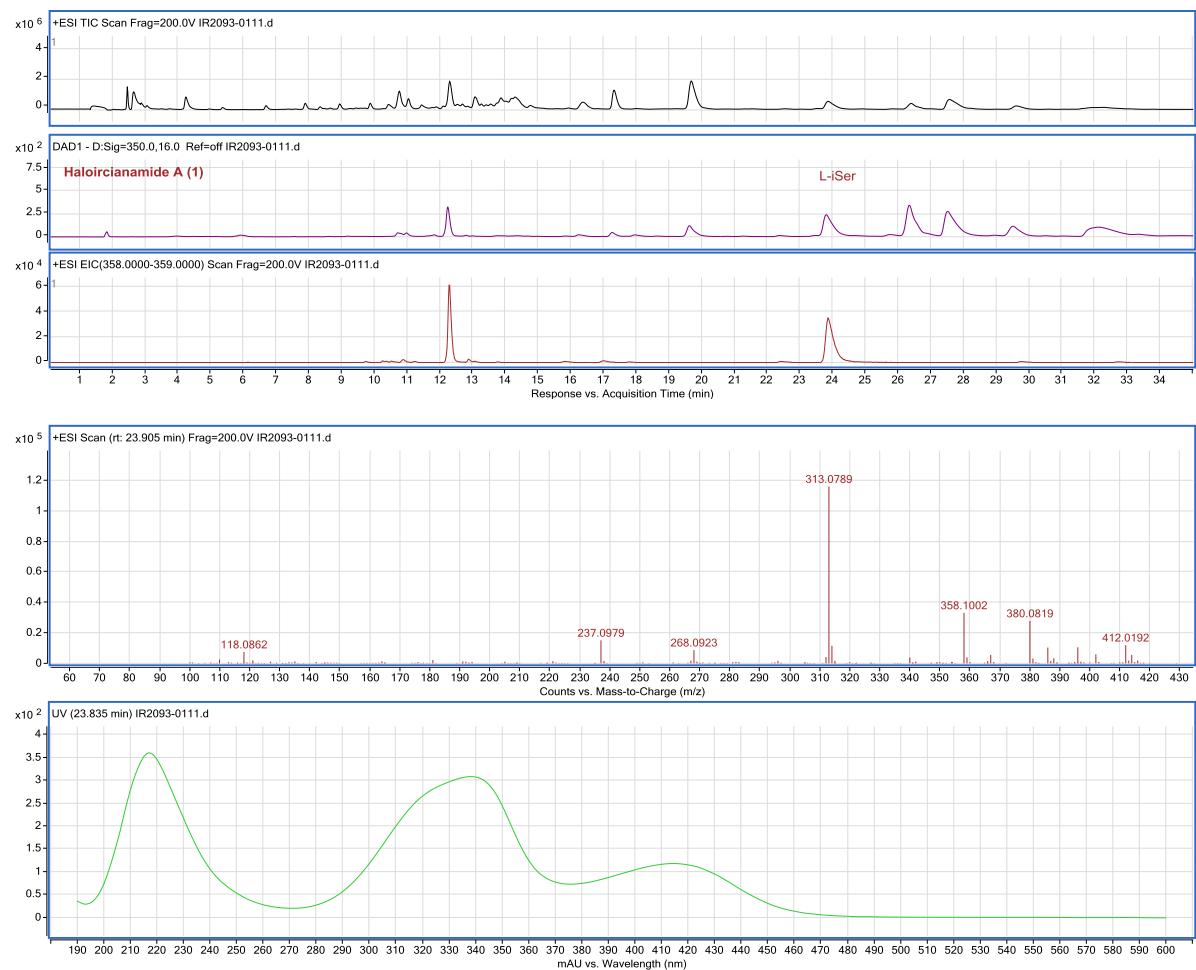
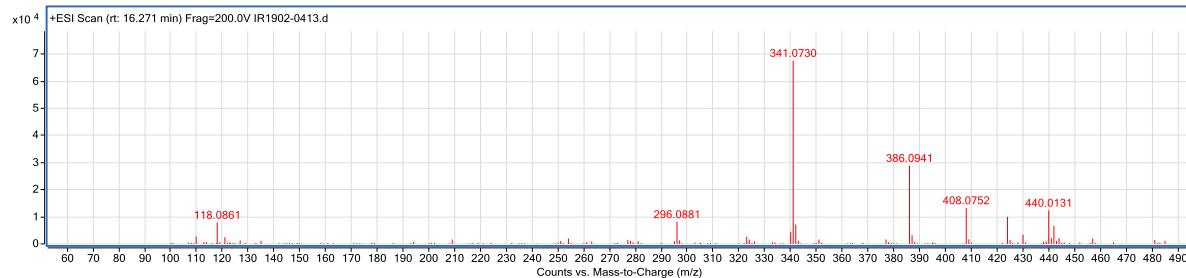
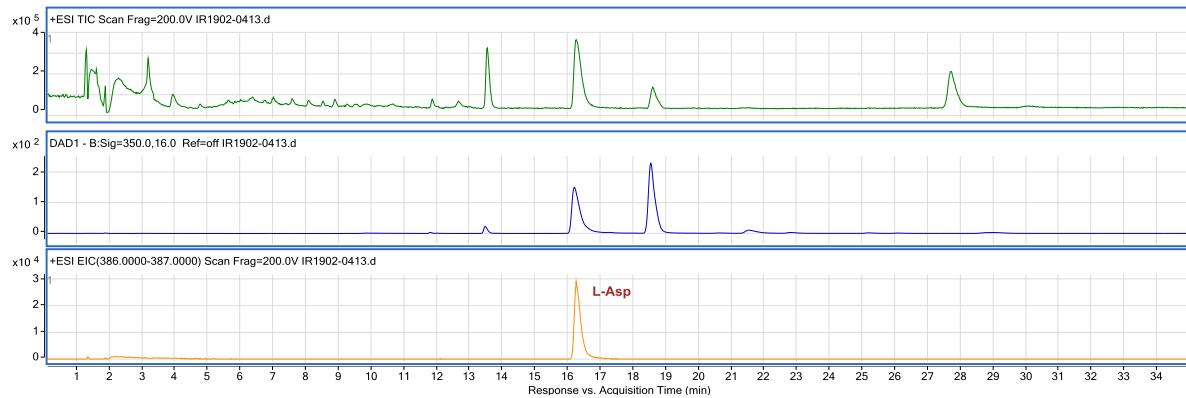
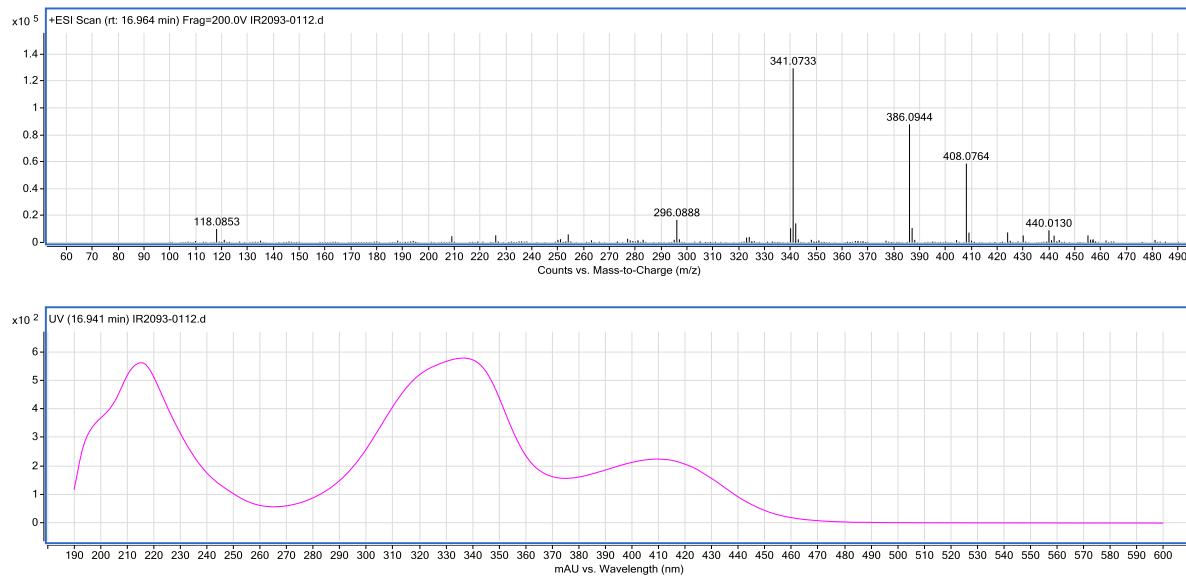
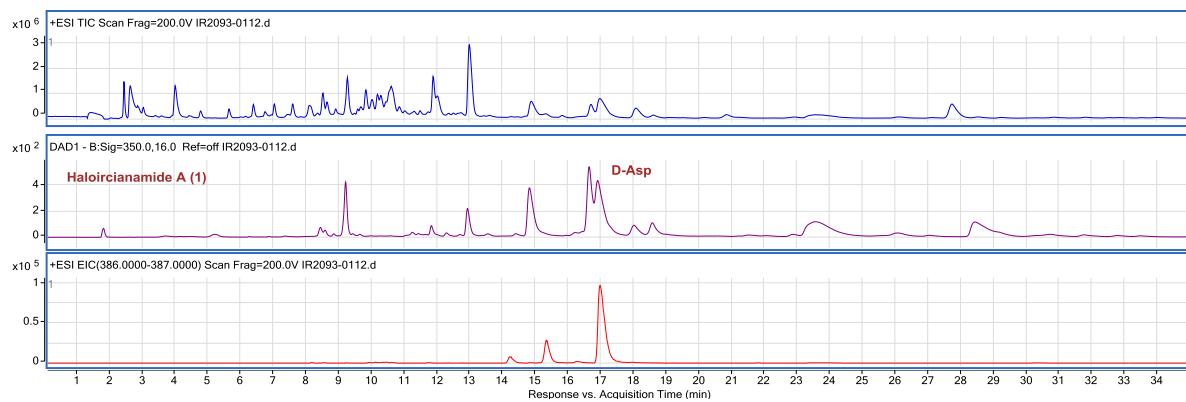


Figure S31 Marfey of Haloirciamide A (**1**) and iSer standards



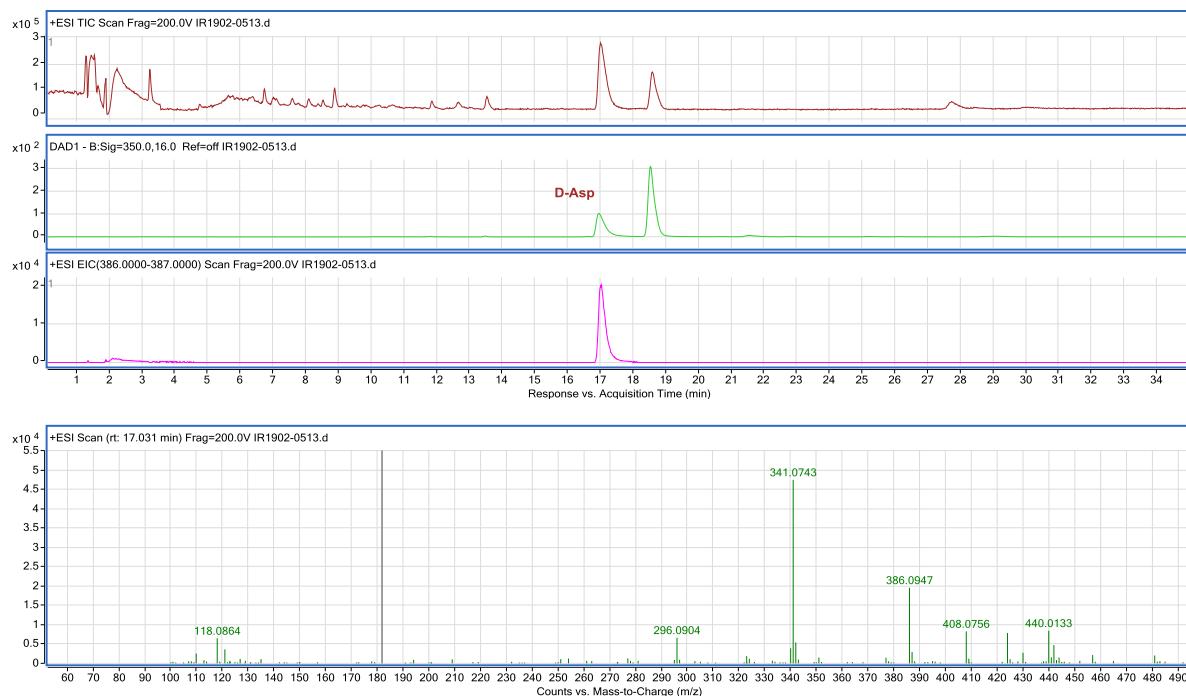
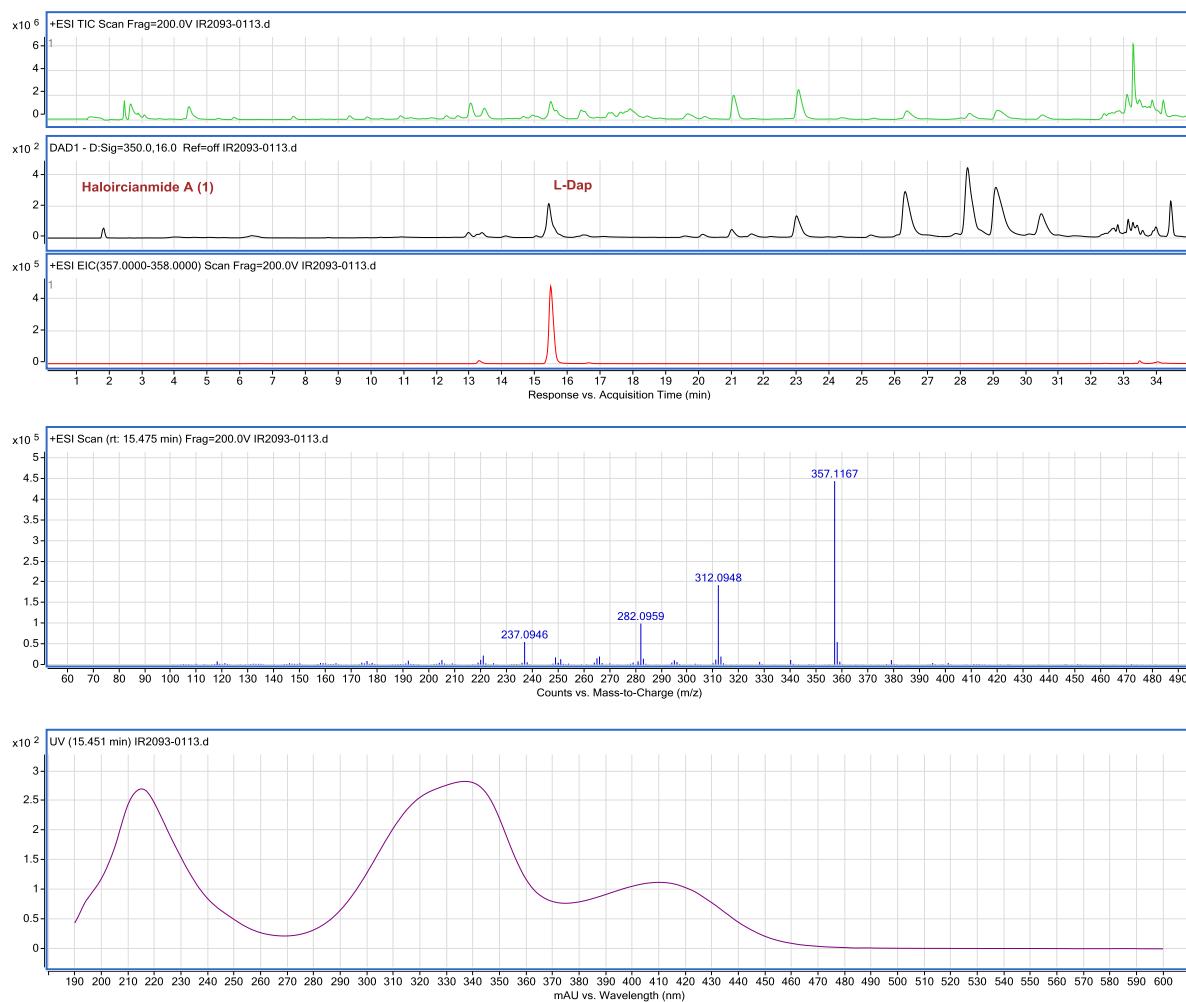


Figure S32 Marfey of Haloirciamide A (**1**) and Asp standards



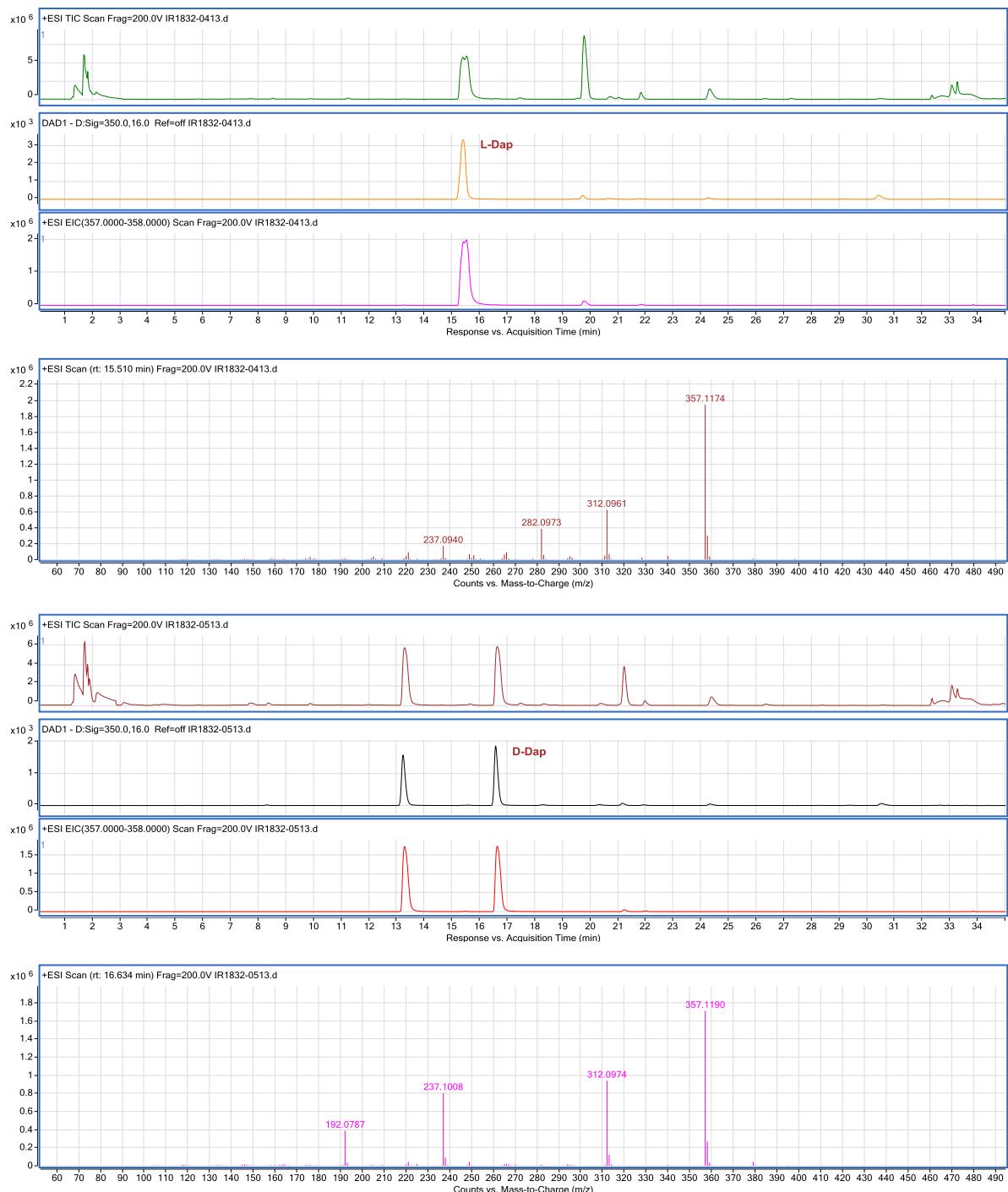


Figure S33 Marfey of Haloirciamide A (**1**) and Dap standards

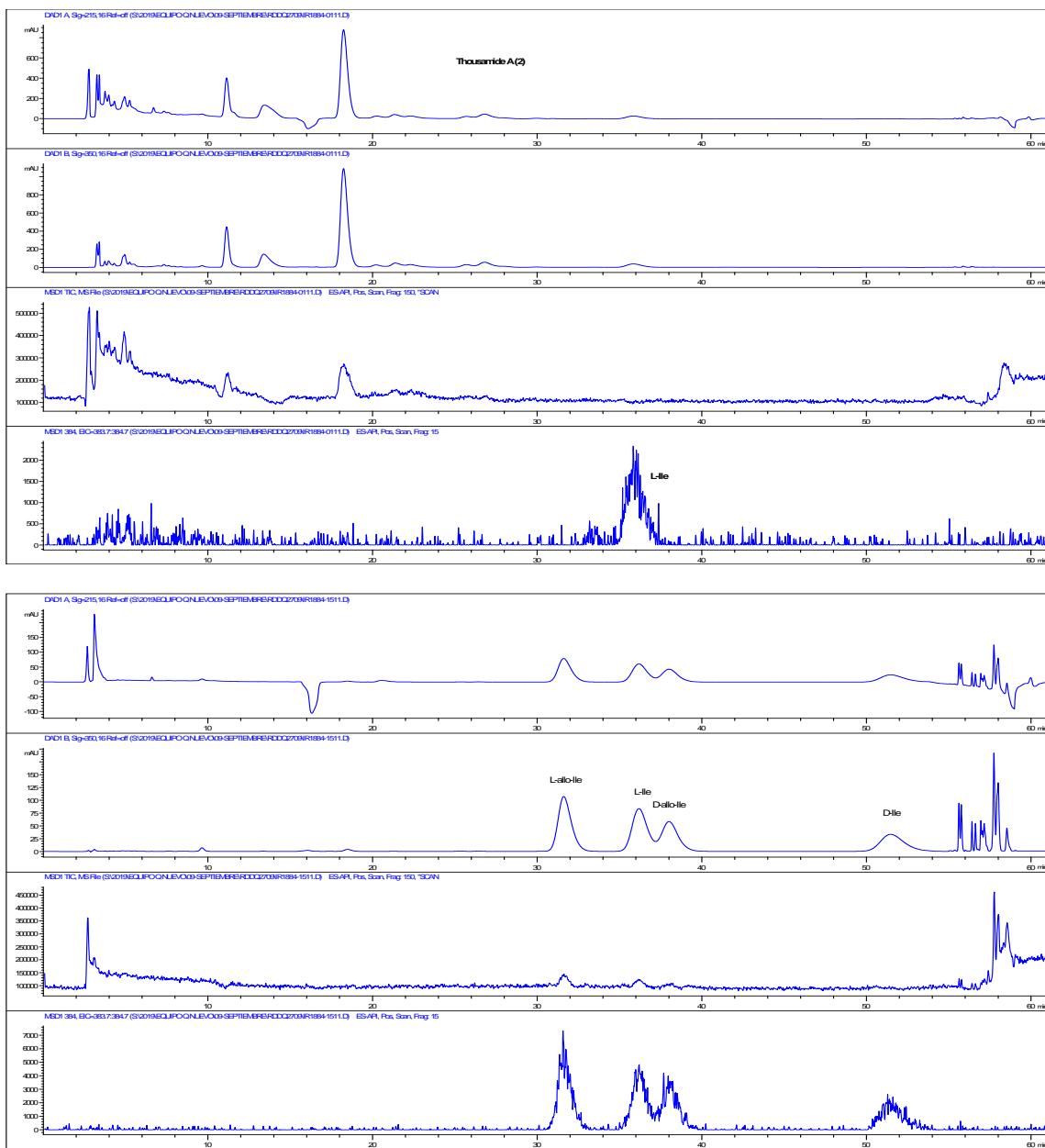


Figure S34 Marfey of Seribunamide A (**2**) and Ile standards

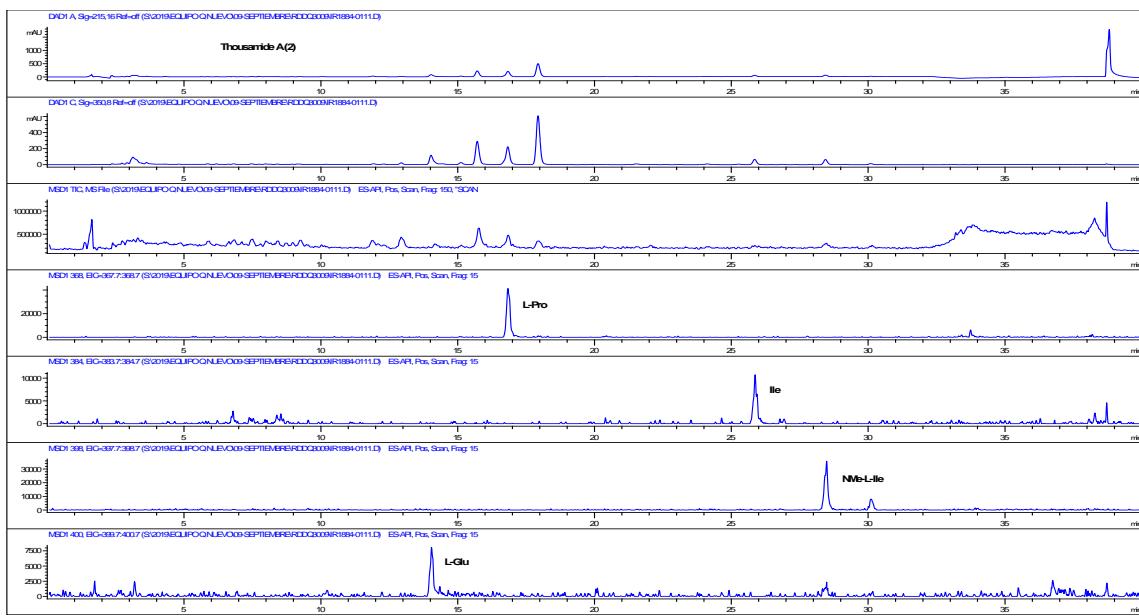


Figure S35 Marfey of Seribunamide A (**2**)

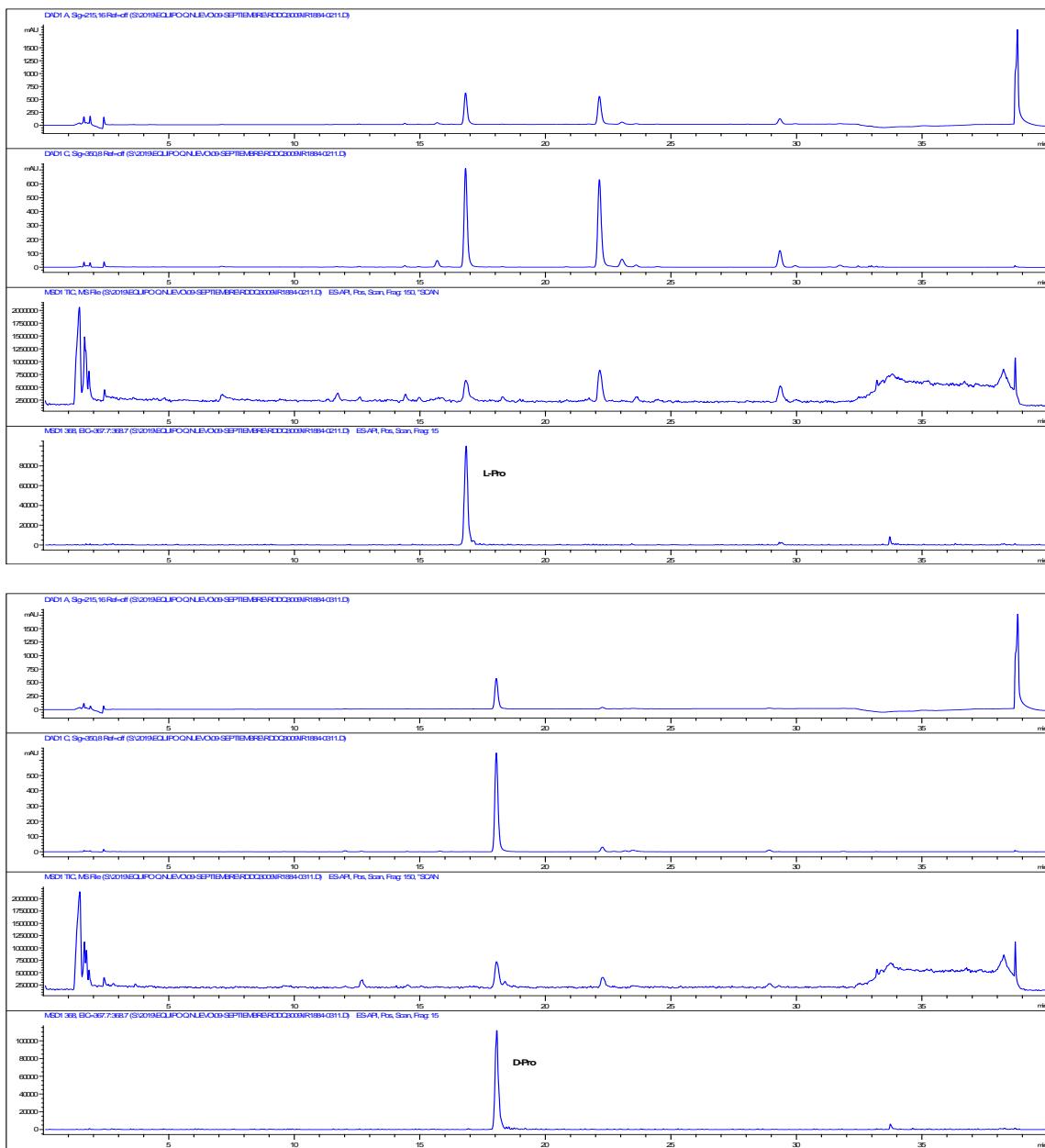


Figure S36 Marfey of Seribunamide A (**2**). Pro standards

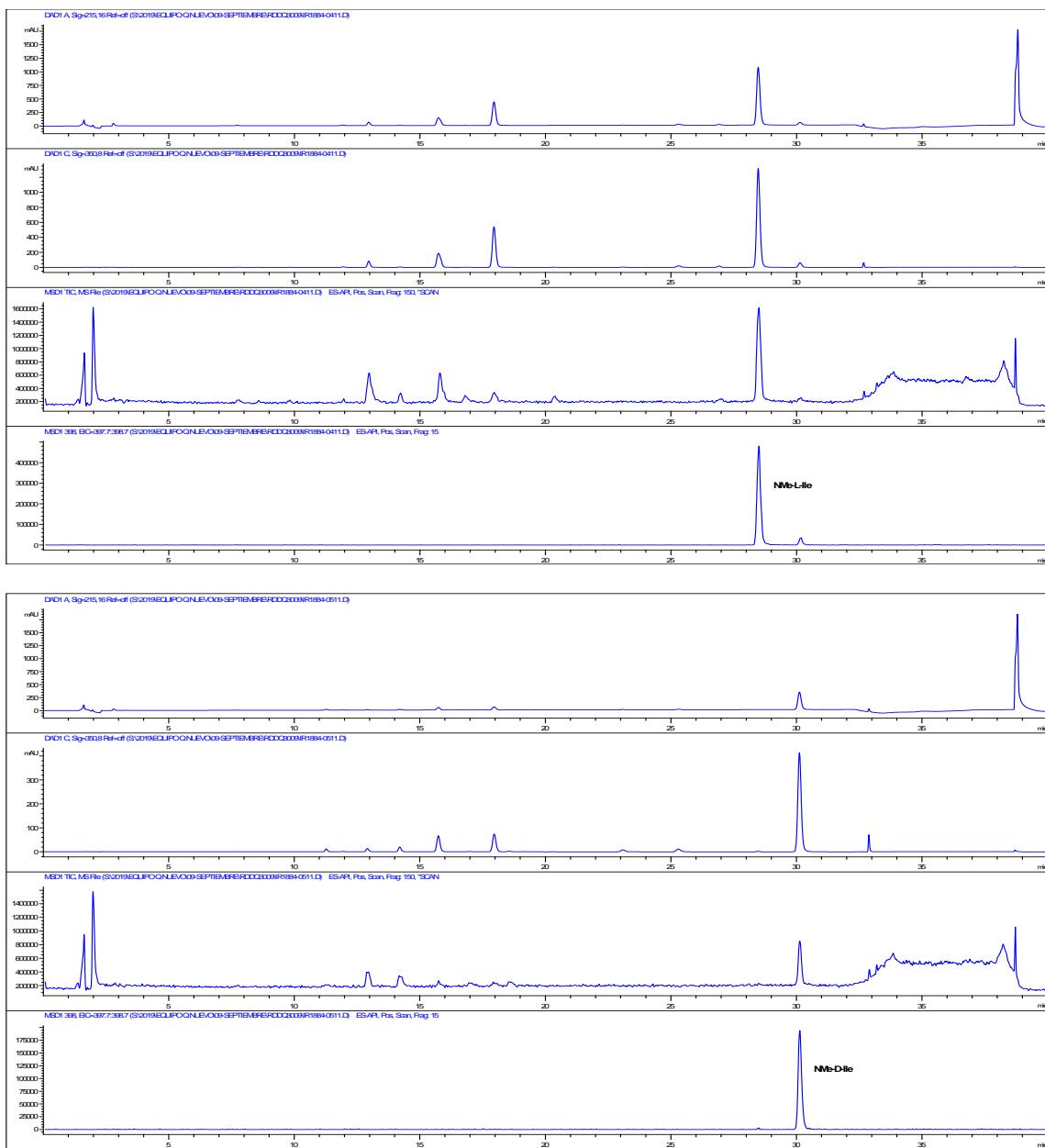


Figure S37 Marfey of Seribunamide A (**2**). NMeLeu standards

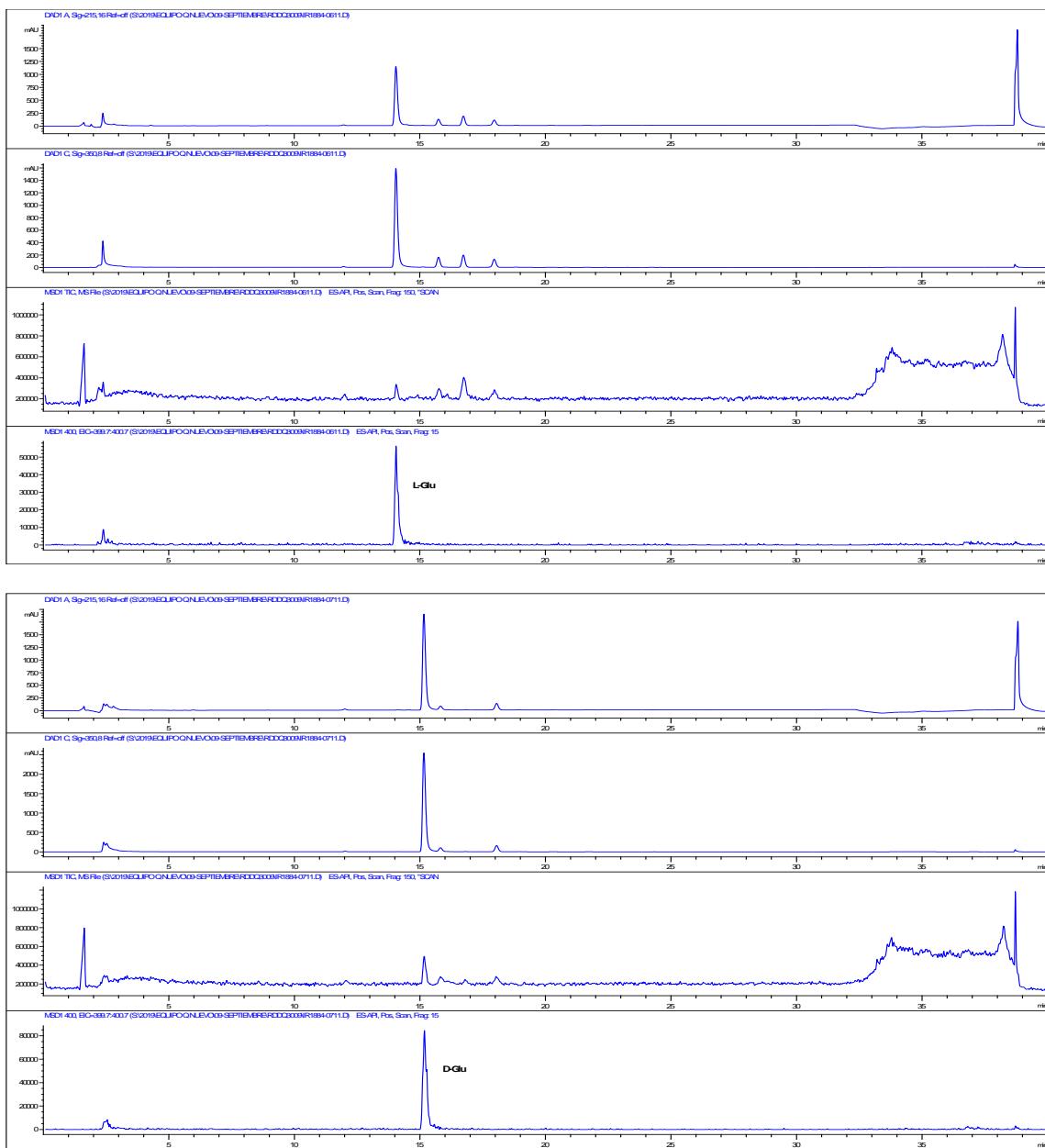


Figure S38 Marfey of Seribunamide A (**2**). Glu standards