

Supplementary Materials

Rare Ophiuroid Type Steroid 3 β ,21-, 3 β ,22-, and 3 α ,22-Disulfates from the Slime Sea Star *Pteraster marsippus* and Their Colony-Inhibiting Effects Against Human Breast Cancer Cells

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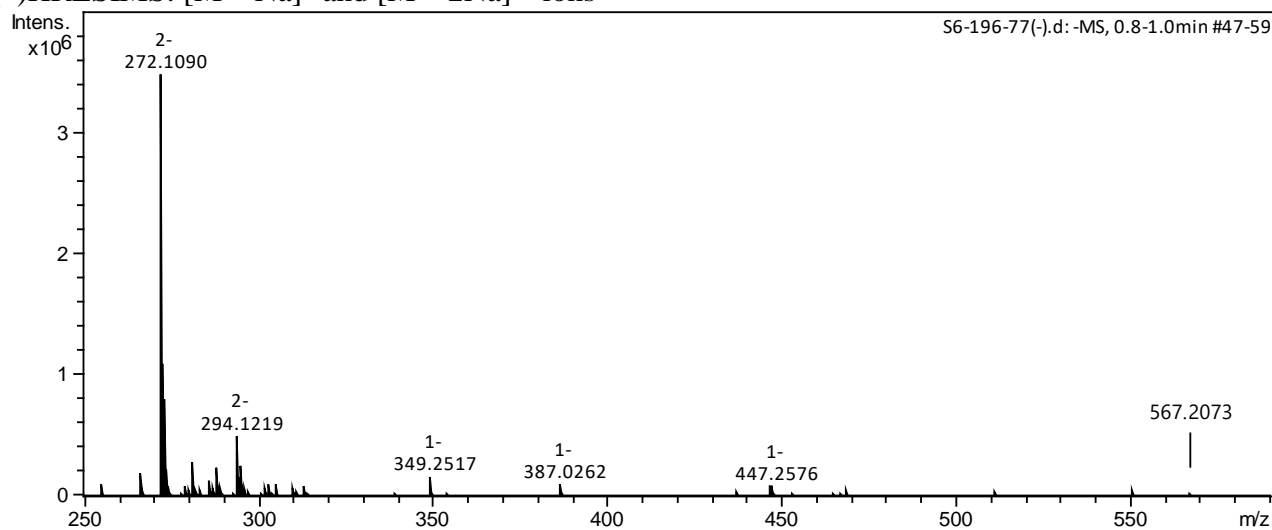
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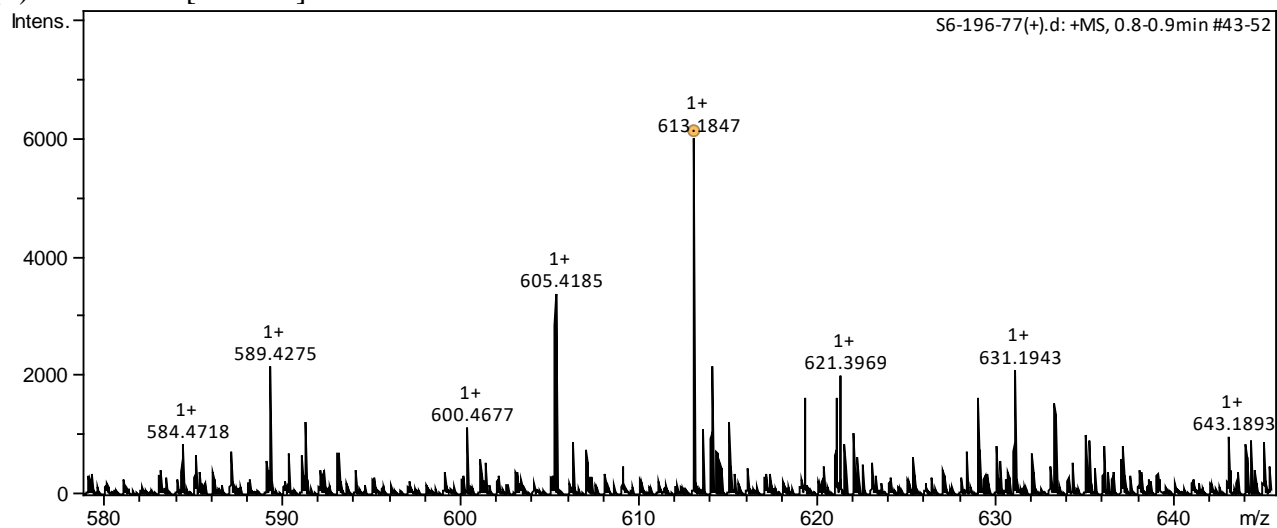
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Figure S1. HRESIMS and HRESIMS/MS spectra of compound **1**.

(-)HRESIMS: $[M - Na]^-$ and $[M - 2Na]^{2-}$ ions



(+)HRESIMS: $[M + Na]^+$ ion



(-)HRESIMS/MS of the $[M - 2Na]^{2-}$ ion at m/z 272.1076

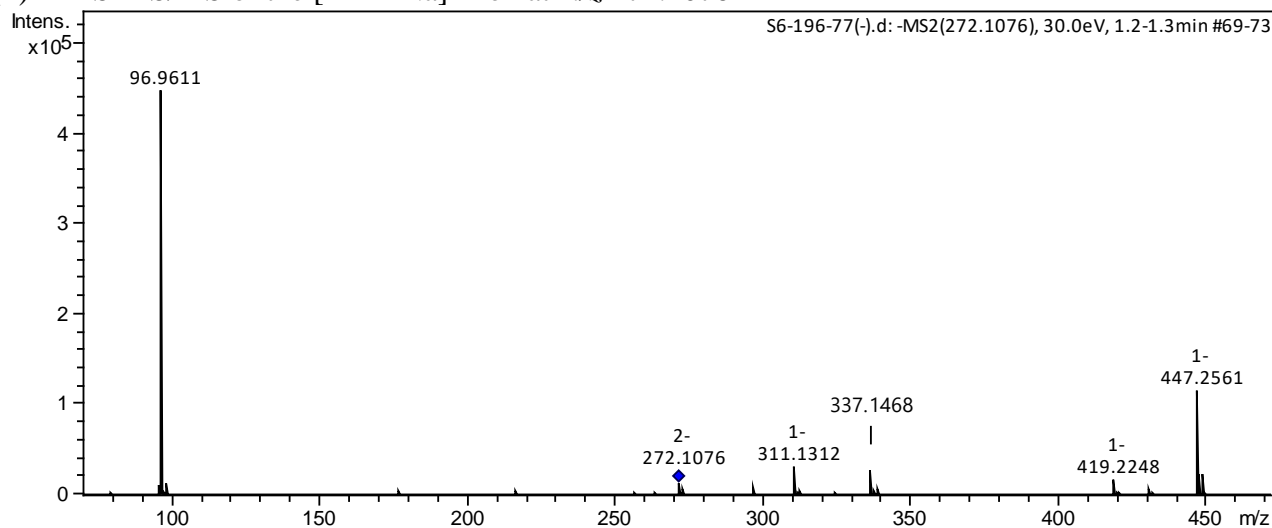


Figure S2. ^1H NMR (700.13 MHz, CD_3OD) spectrum of compound **1**.

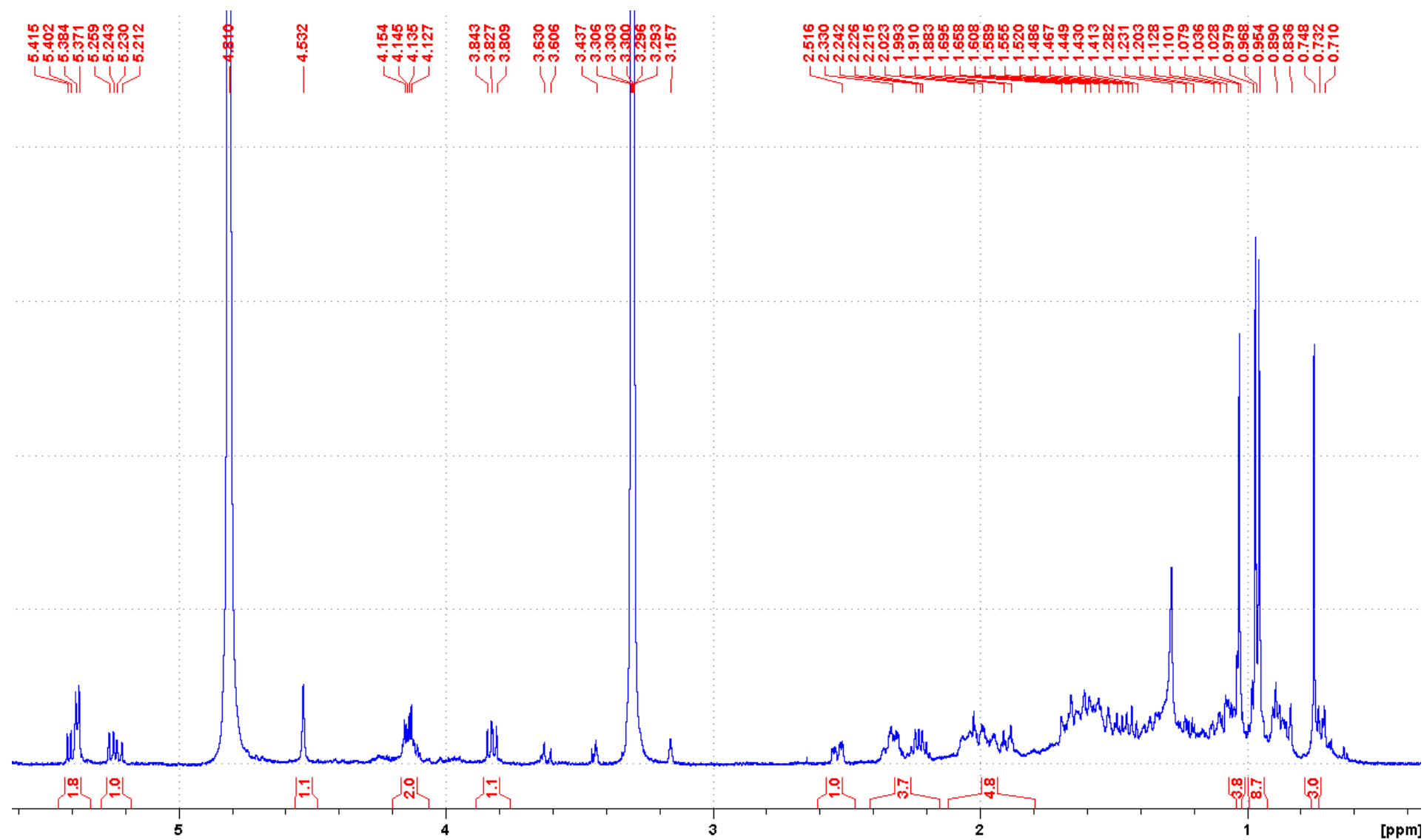


Figure S3. ^{13}C NMR (176.04 MHz, CD_3OD) spectrum of compound **1**.

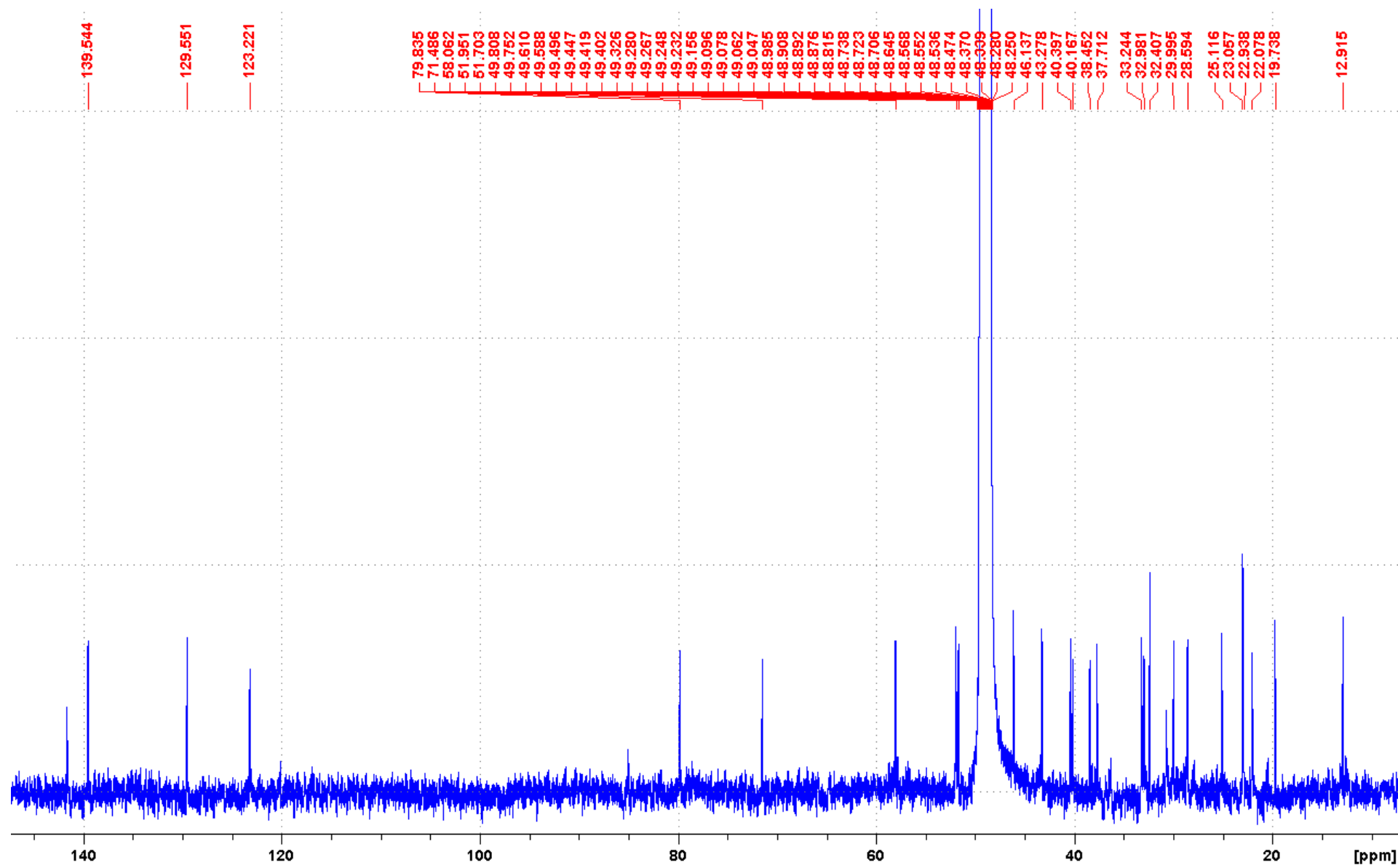


Figure S4. ^1H - ^1H COSY (700.13 MHz, CD_3OD) spectrum of compound **1**.

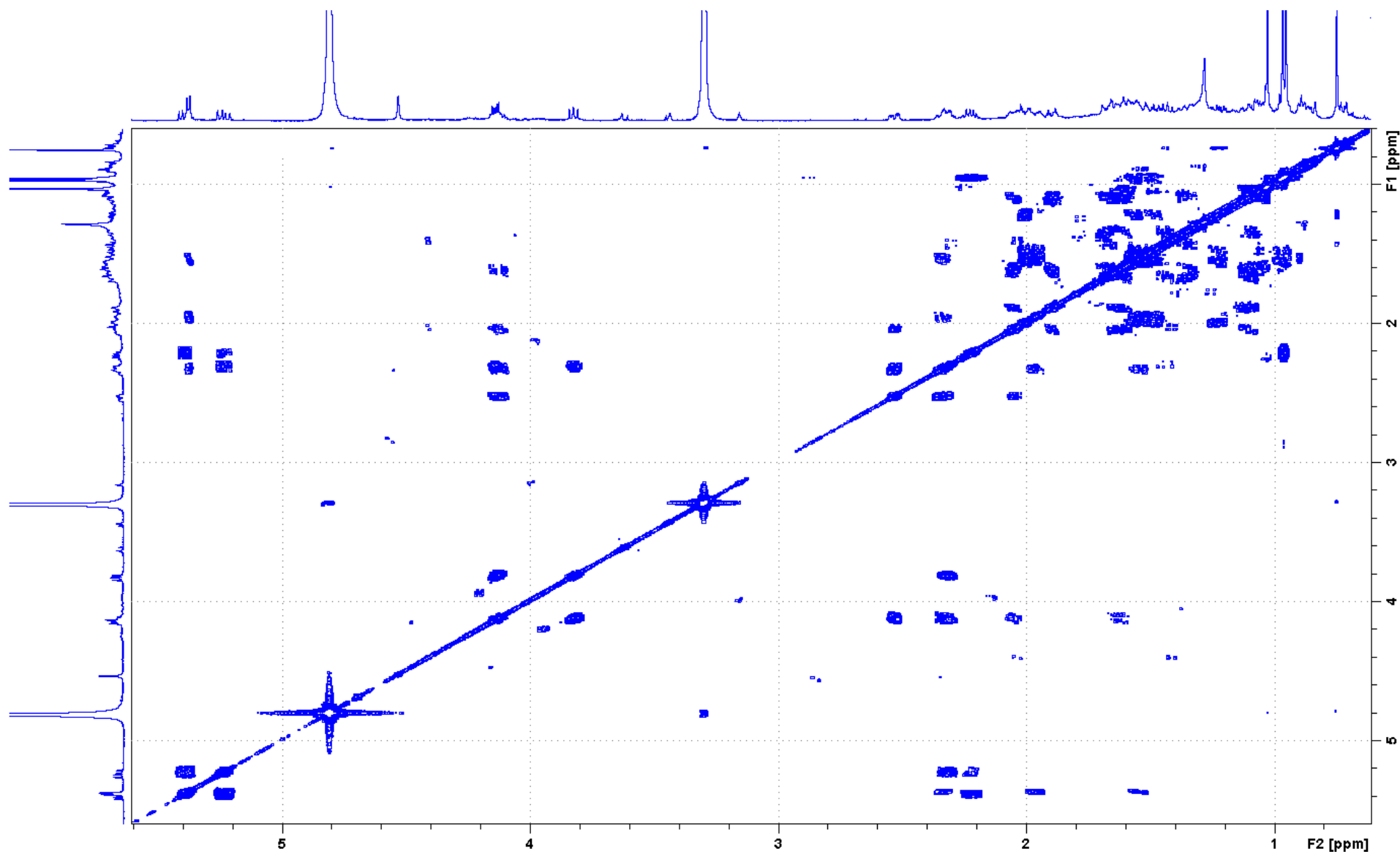


Figure S5. HSQC (700.13 MHz, CD₃OD) spectrum of compound **1**.

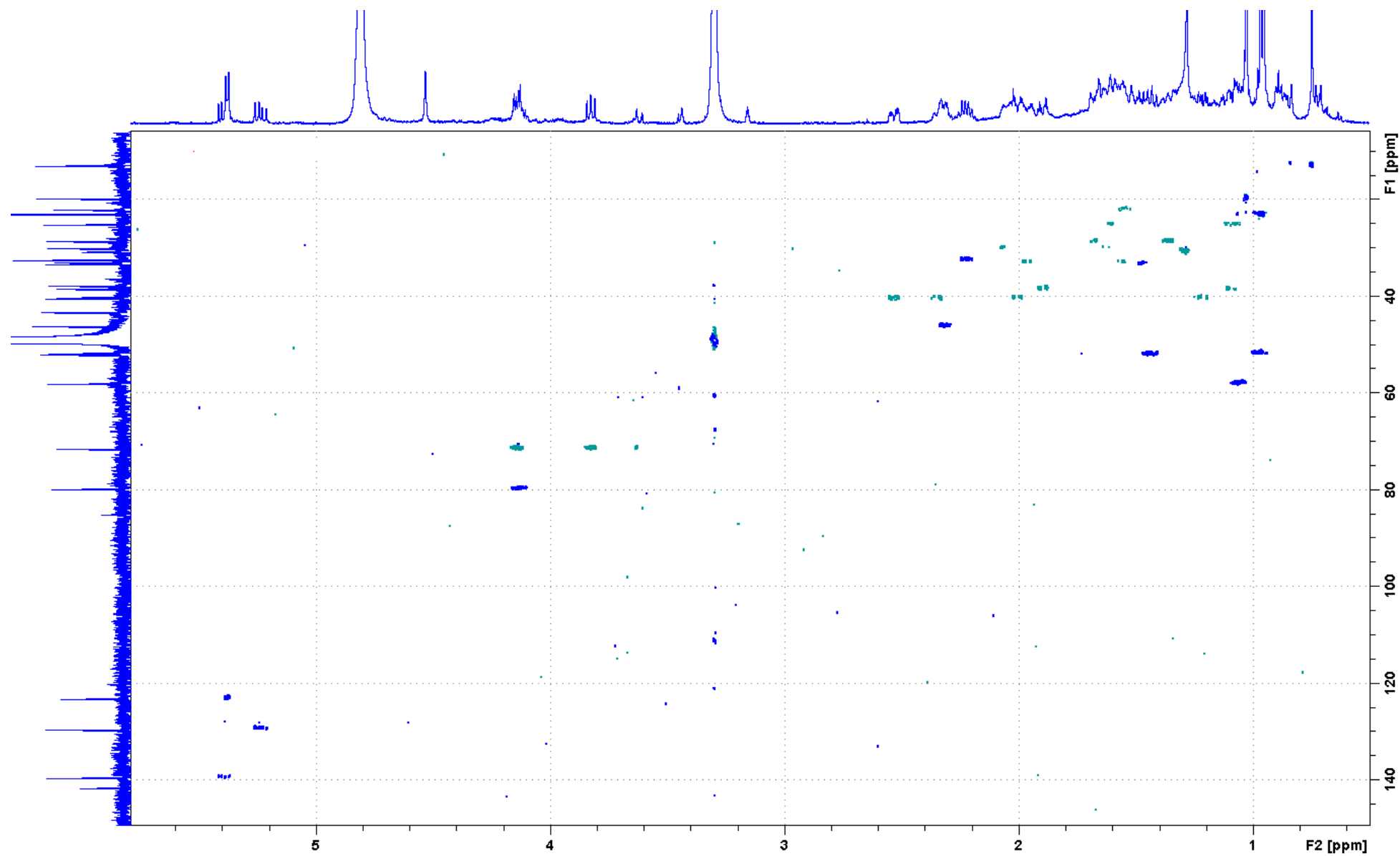


Figure S6. HMBC (700.13 MHz, CD₃OD) spectrum of compound **1**.

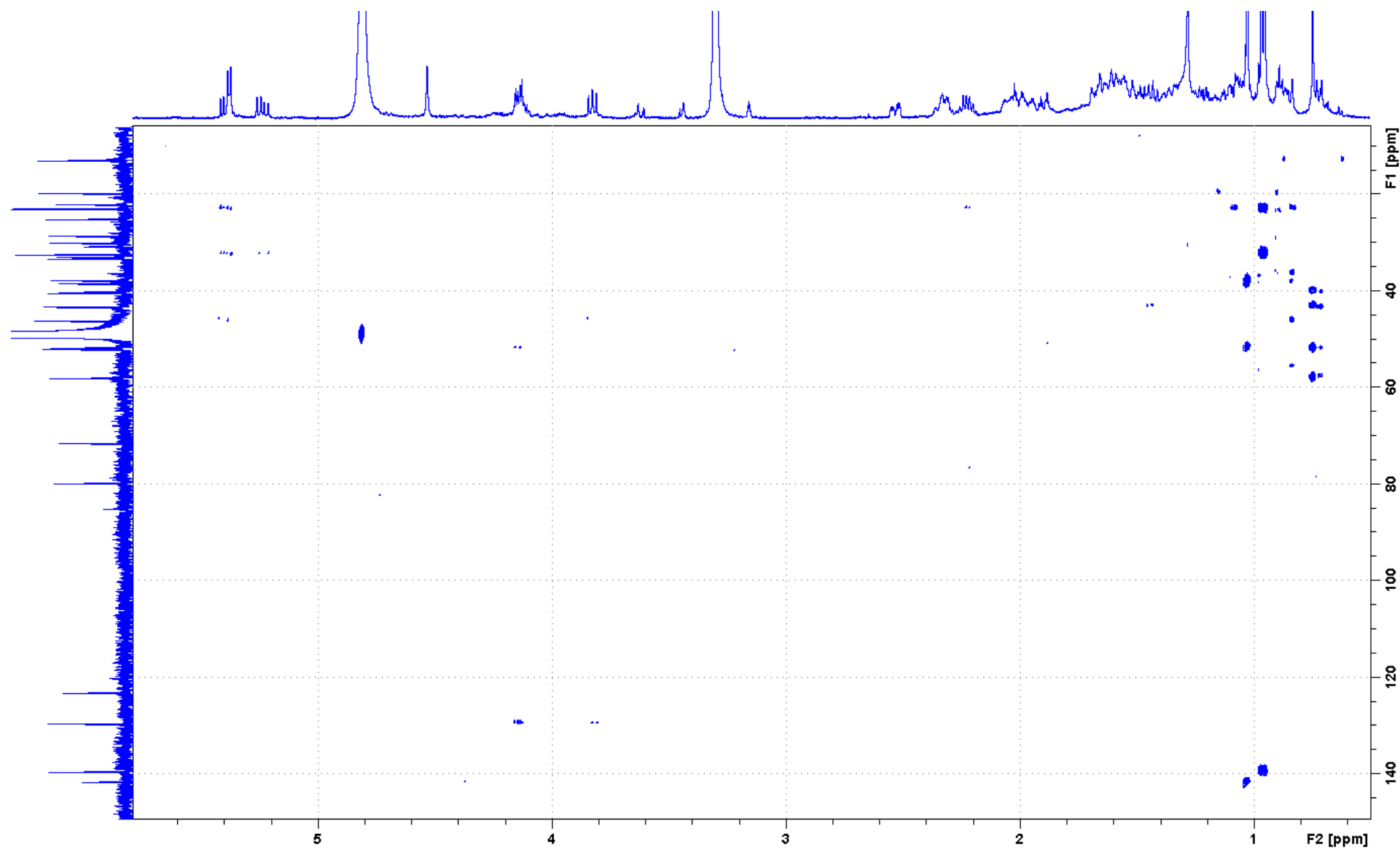


Figure S7. ROESY (700.13 MHz, CD₃OD) spectrum of compound **1**.

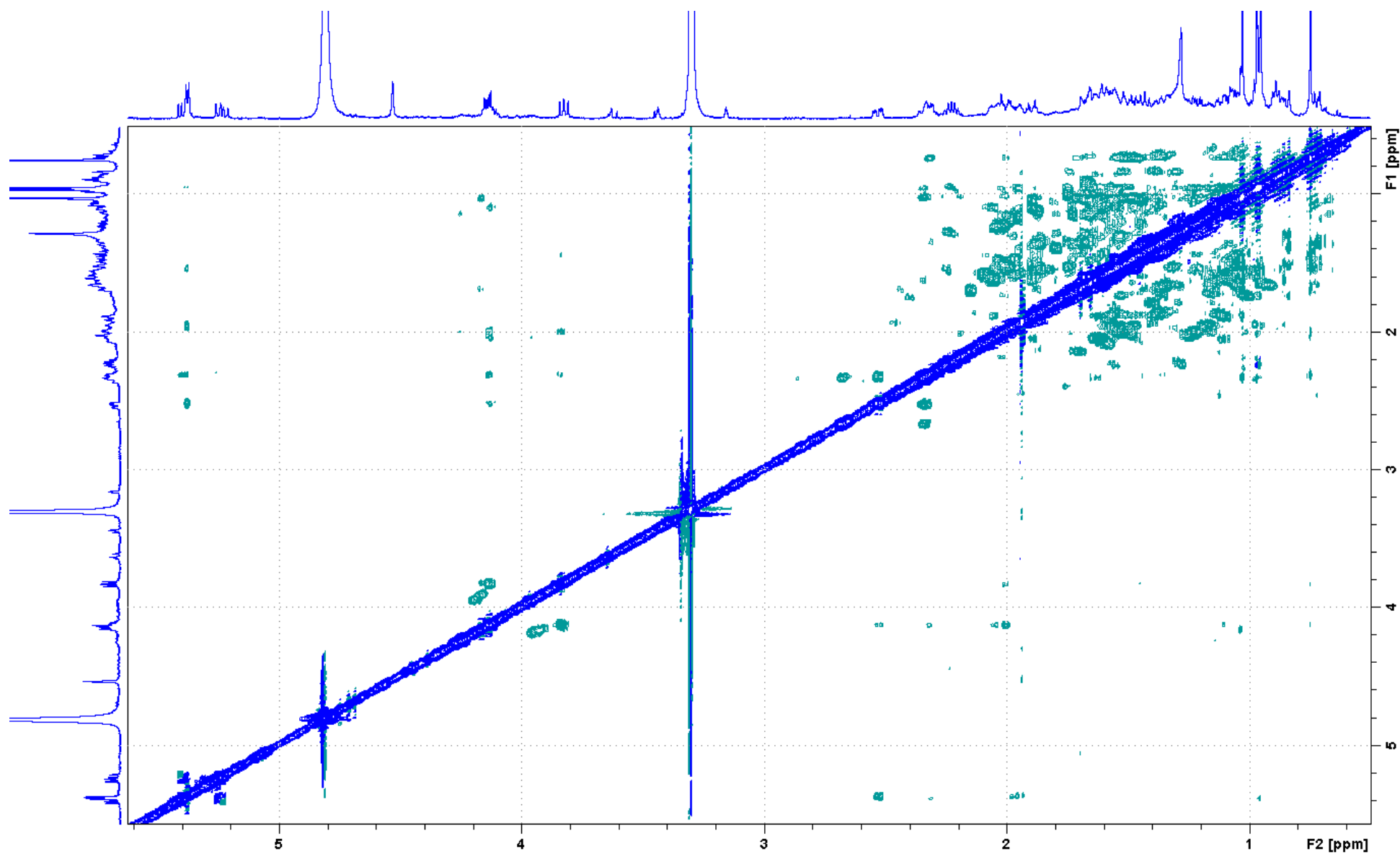
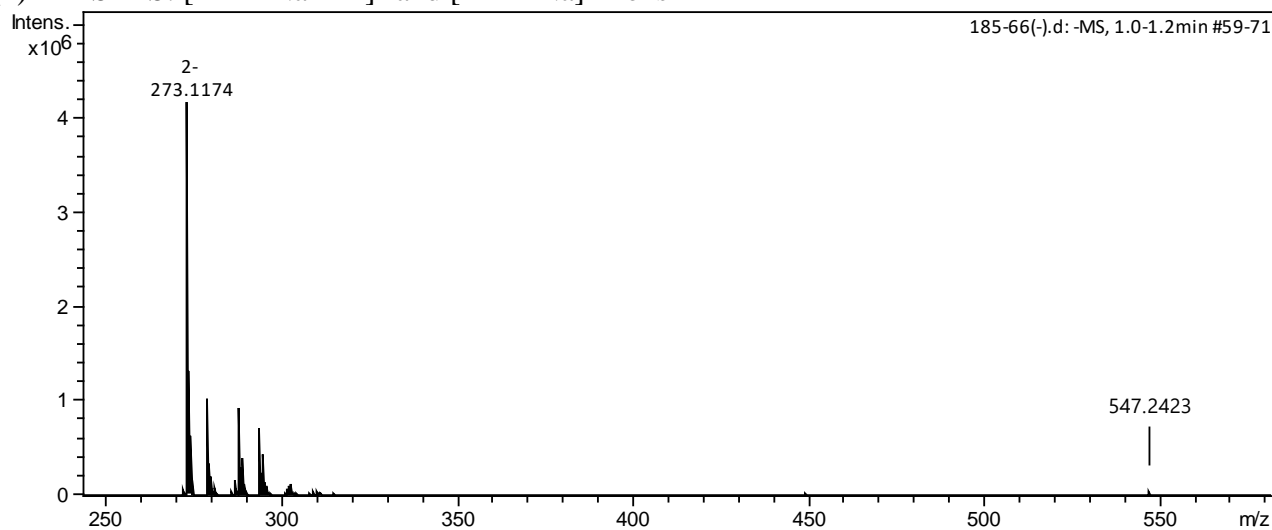
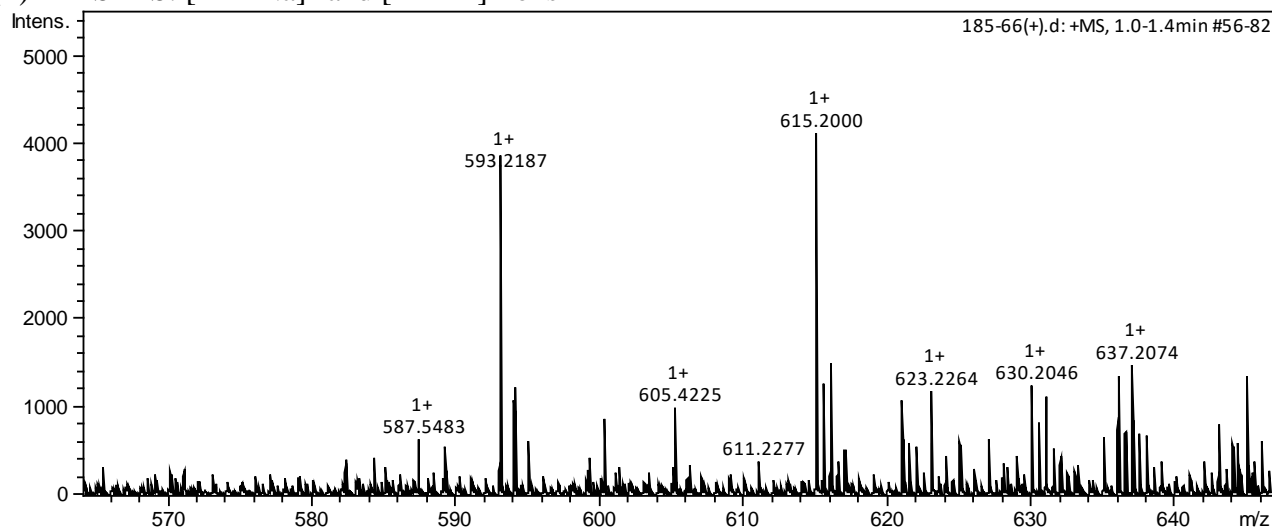


Figure S8. HRESIMS and HRESIMS/MS spectra of compound **2**.

(-)HRESIMS: $[M - 2Na + H]^-$ and $[M - 2Na]^{2-}$ ions



(+)HRESIMS: $[M + Na]^+$ and $[M + H]^+$ ions



(-)HRESIMS/MS of the $[M - 2Na]^{2-}$ ion at m/z 273.1171

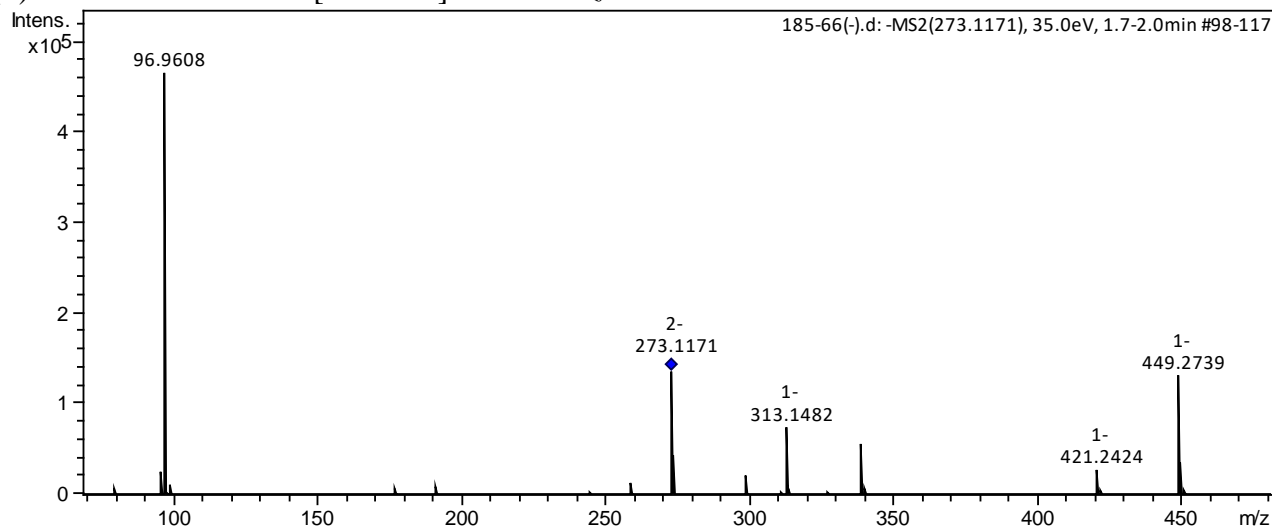


Figure S9. ^1H NMR (700.13 MHz, CD_3OD) spectrum of compound 2.

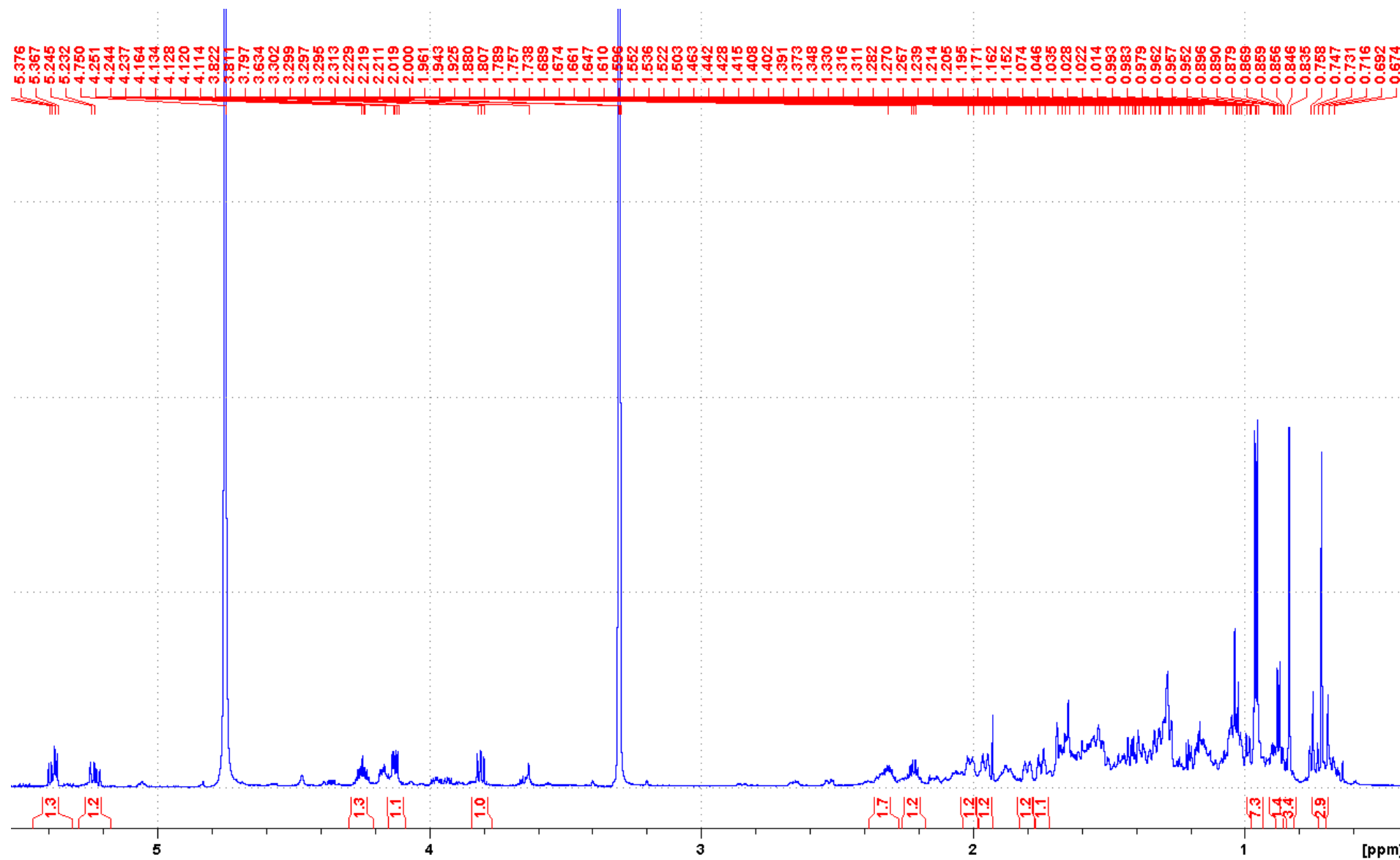


Figure S10. ^{13}C NMR (176.04 MHz, CD_3OD) spectrum of compound **2**.

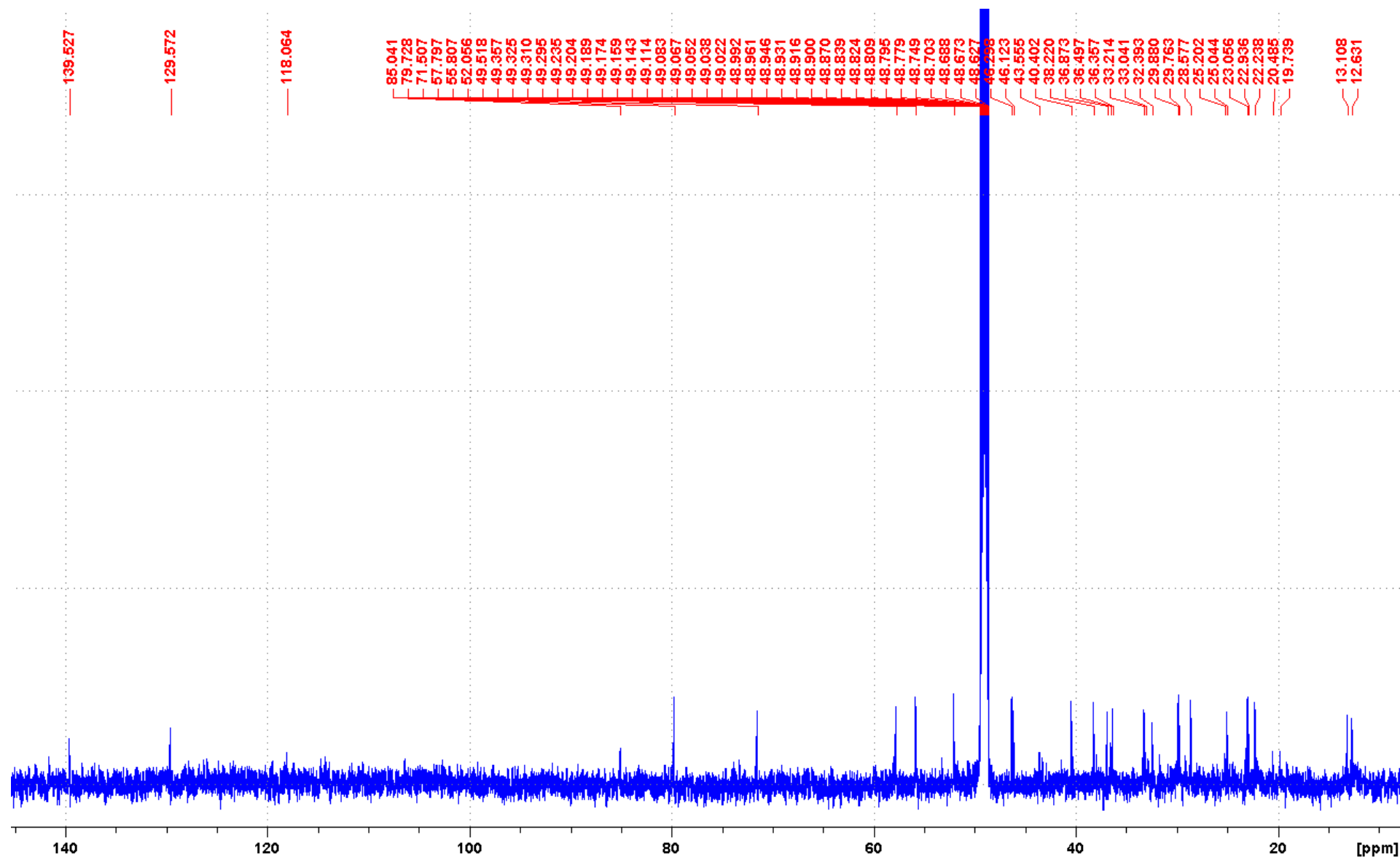


Figure S11. ^1H - ^1H COSY (700.13 MHz, CD_3OD) spectrum of compound **2**.

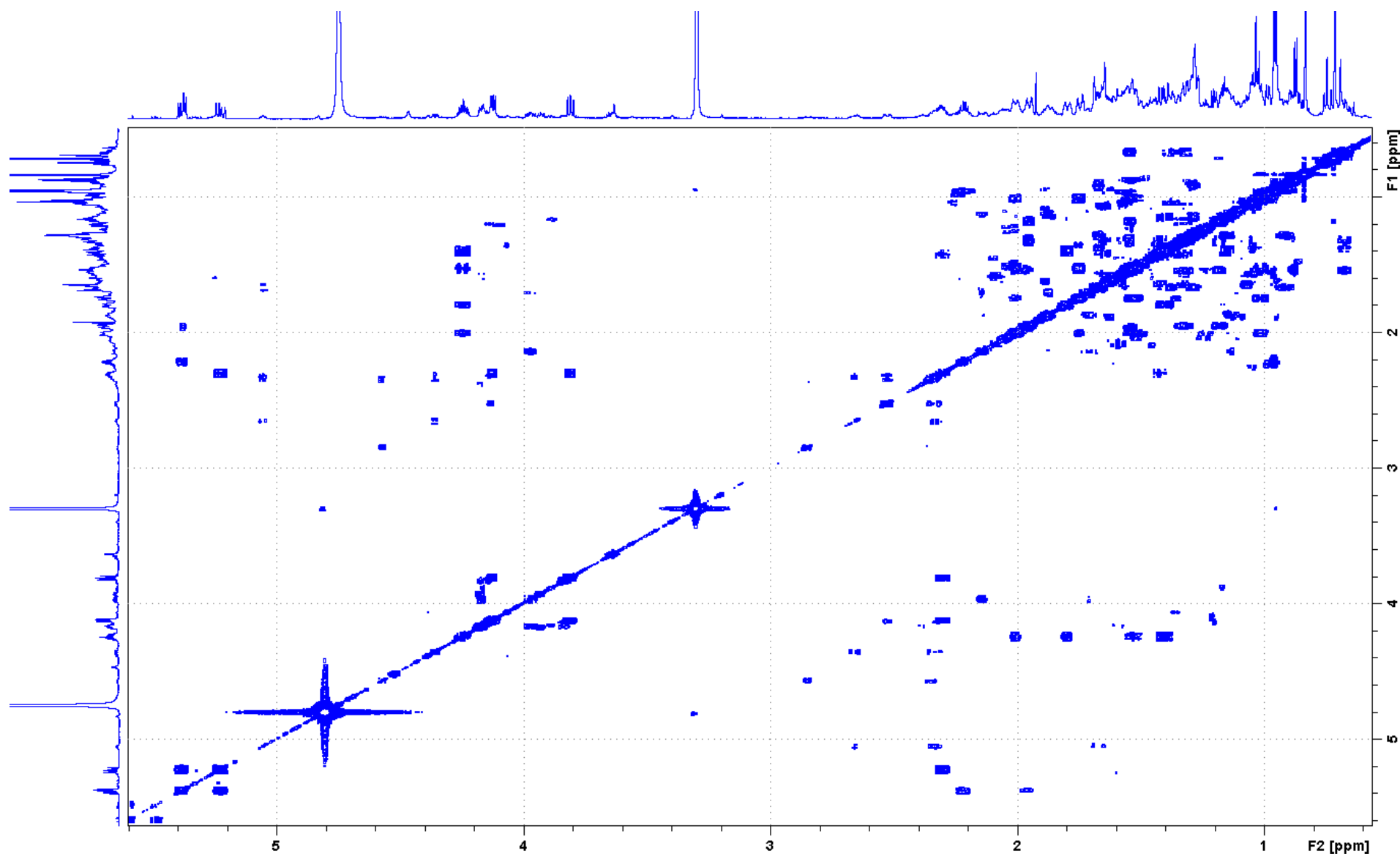


Figure S12. HSQC (700.13 MHz, CD₃OD) spectrum of compound **2**.

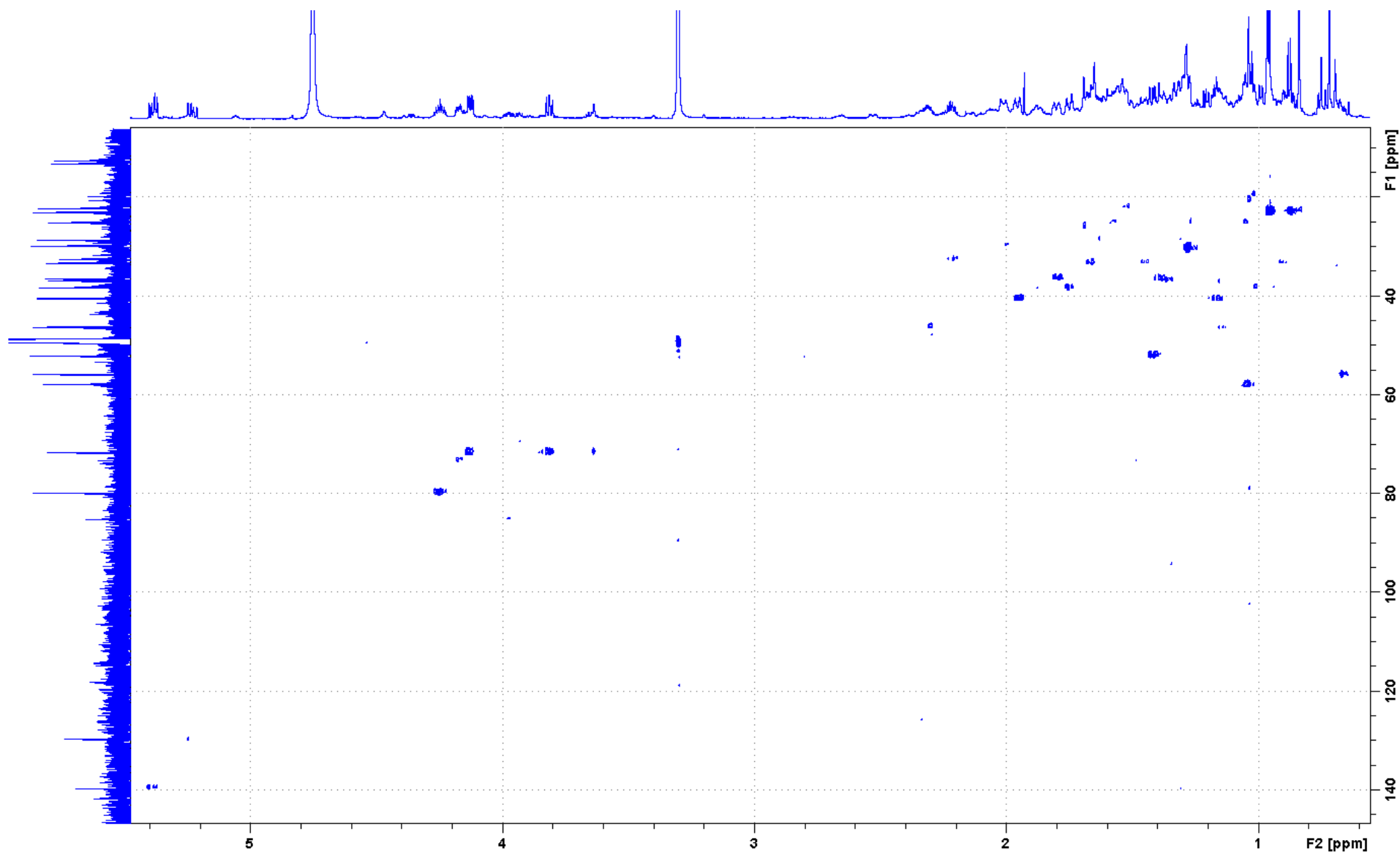


Figure S13. HMBC (700.13 MHz, CD₃OD) spectrum of compound **2**.

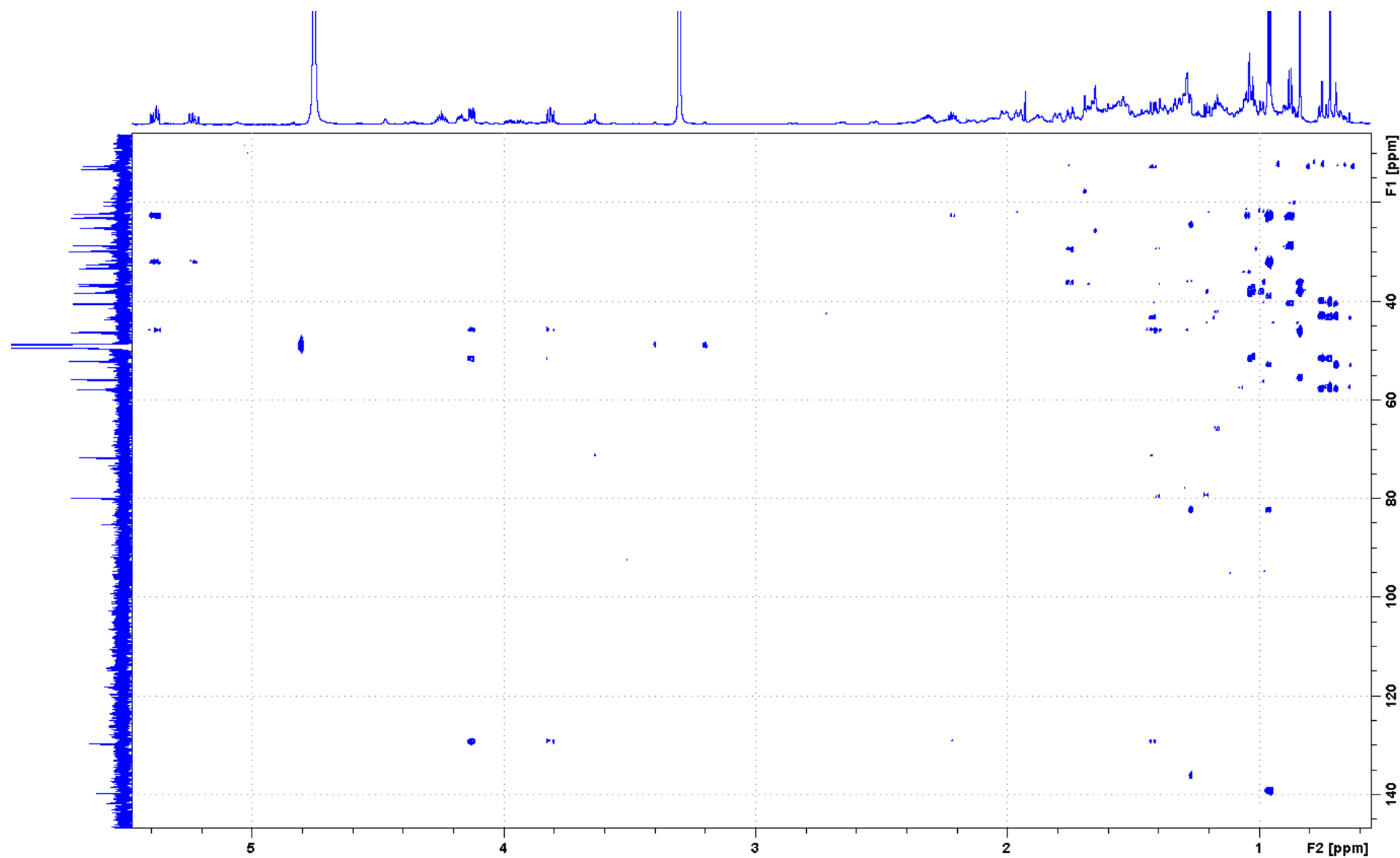


Figure S14. ROESY (700.13 MHz, CD₃OD) spectrum of compound **2**.

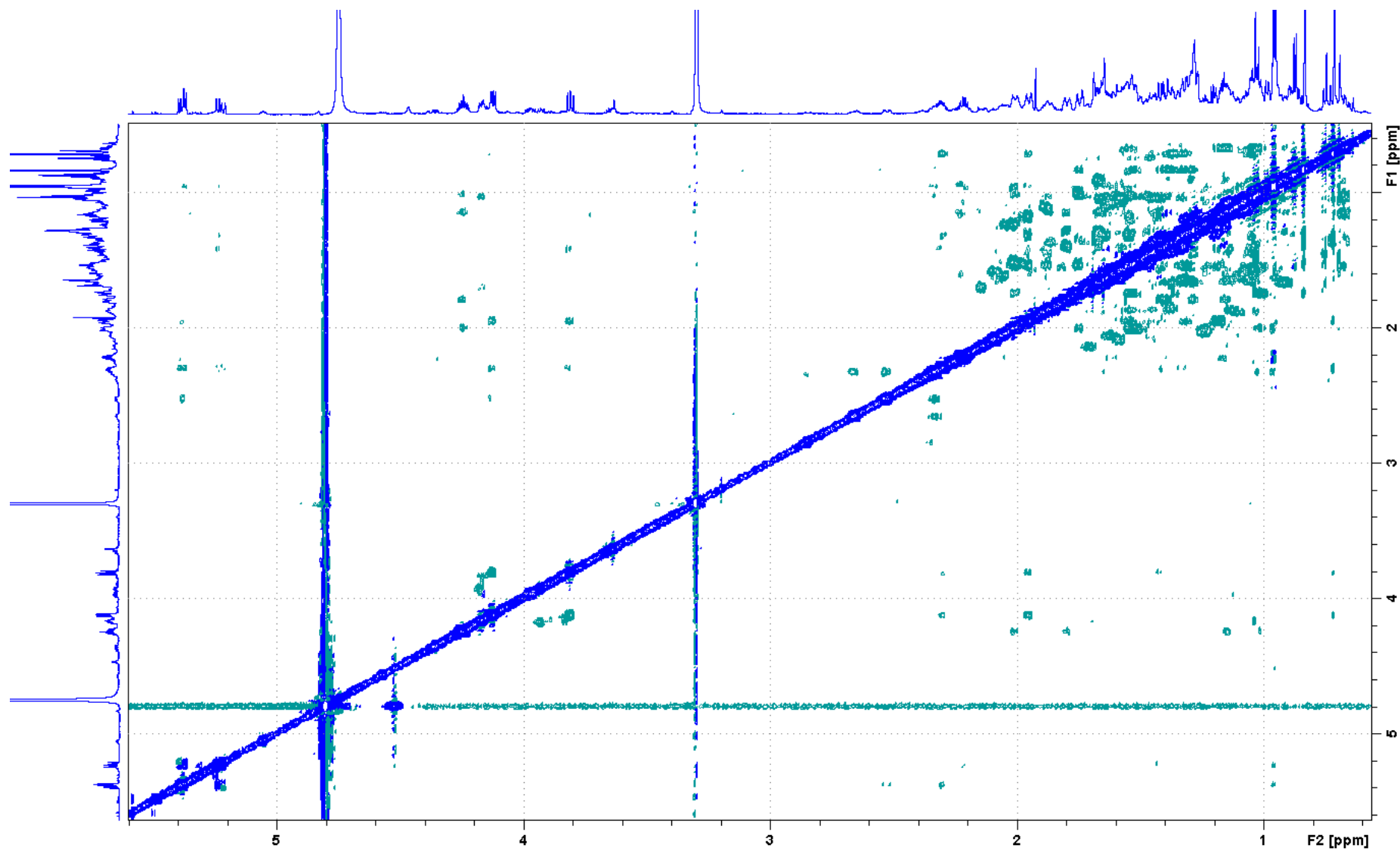
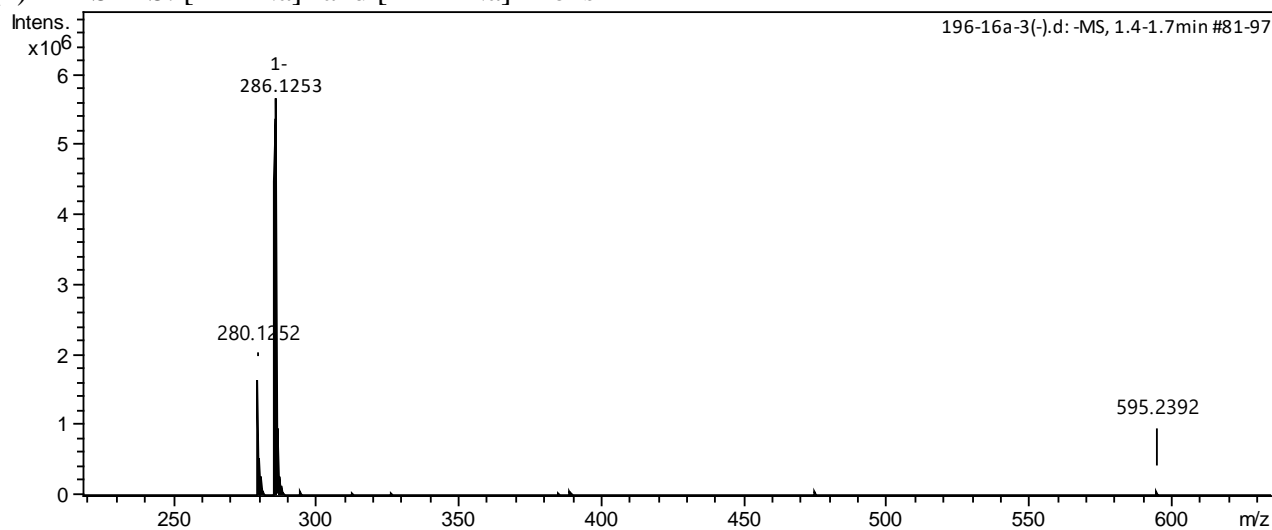
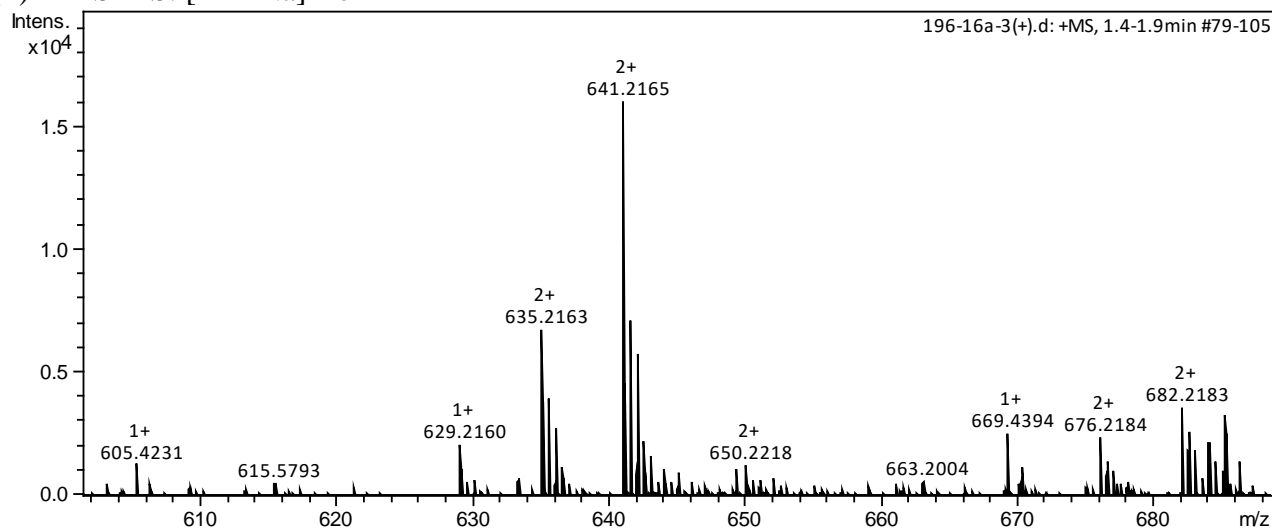


Figure S15. HRESIMS and HRESIMS/MS spectra of compound **3**.

(-)HRESIMS: $[M - Na]^-$ and $[M - 2Na]^{2-}$ ions



(+)HRESIMS: $[M + Na]^+$ ion



(-)HRESIMS/MS of the $[M - 2Na]^{2-}$ ion at m/z 286.1254

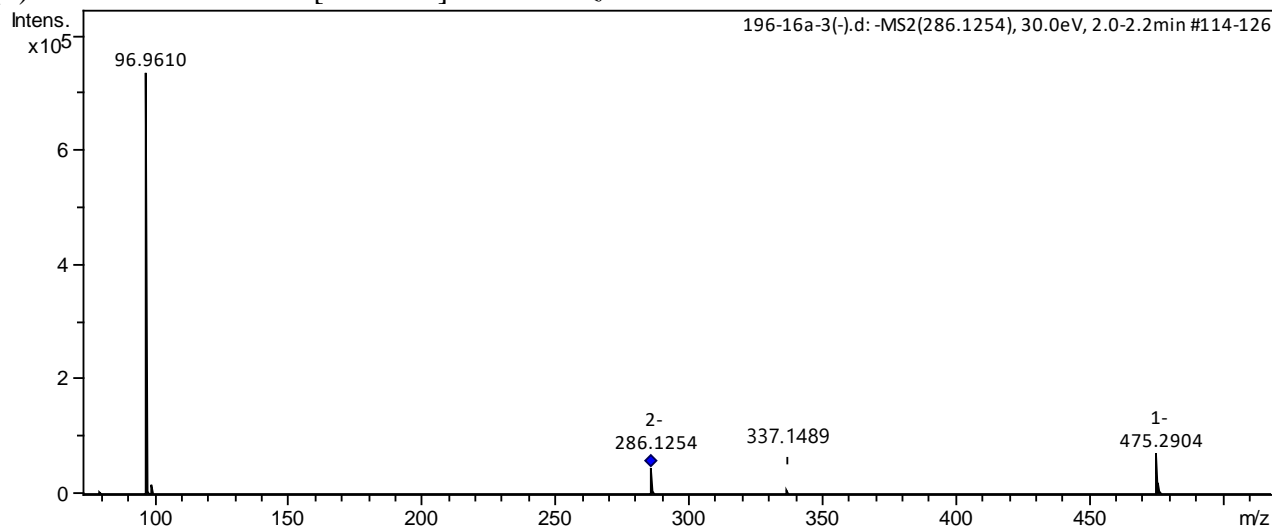


Figure S16. ^1H NMR (700.13 MHz, CD_3OD) spectrum of compound **3**.

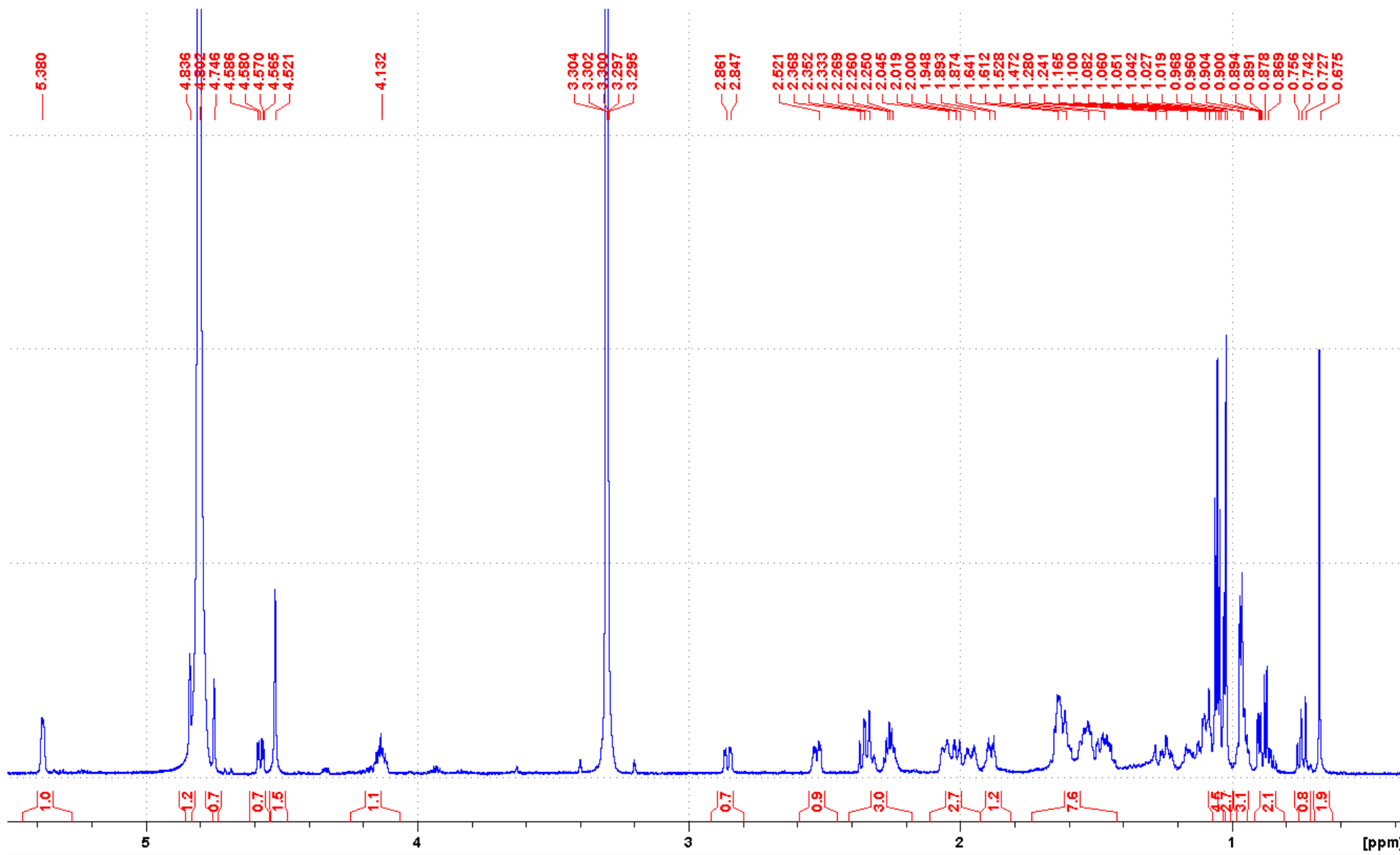


Figure S17. ^{13}C NMR (176.04 MHz, CD_3OD) spectrum of compound **3**.

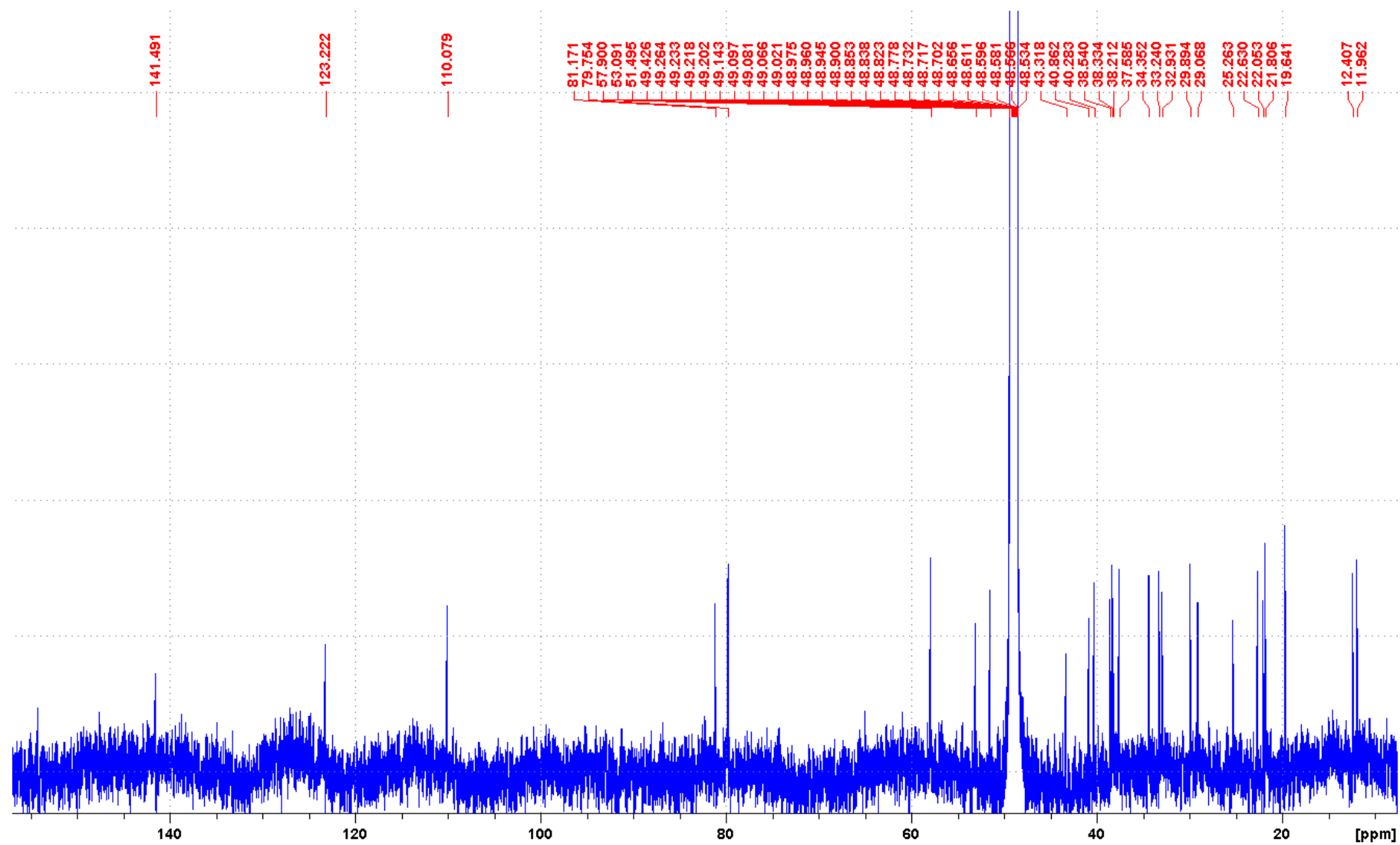


Figure S18. ^1H - ^1H COSY (700.13 MHz, CD_3OD) spectrum of compound **3**.

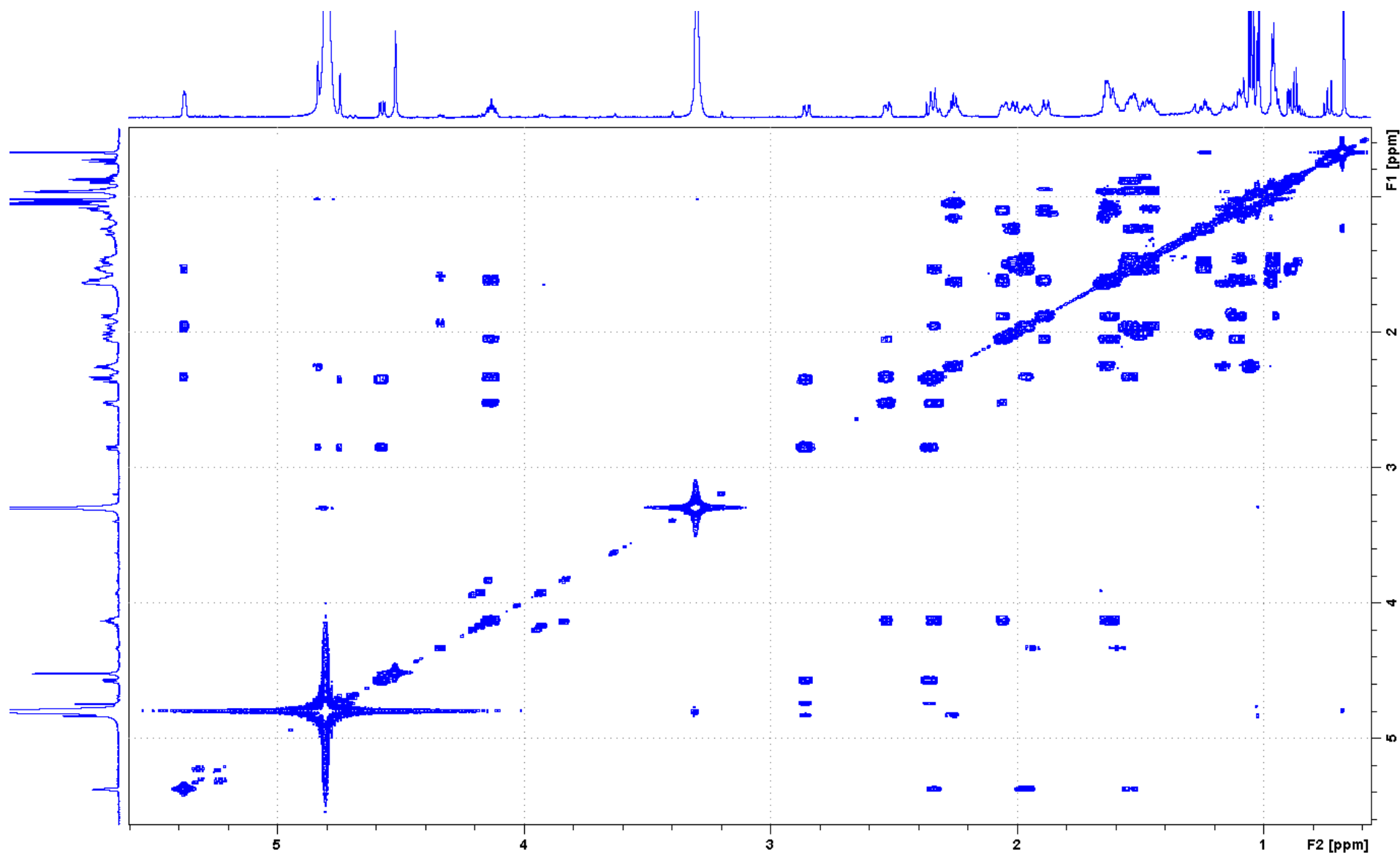


Figure S19. HSQC (700.13 MHz, CD₃OD) spectrum of compound **3**.

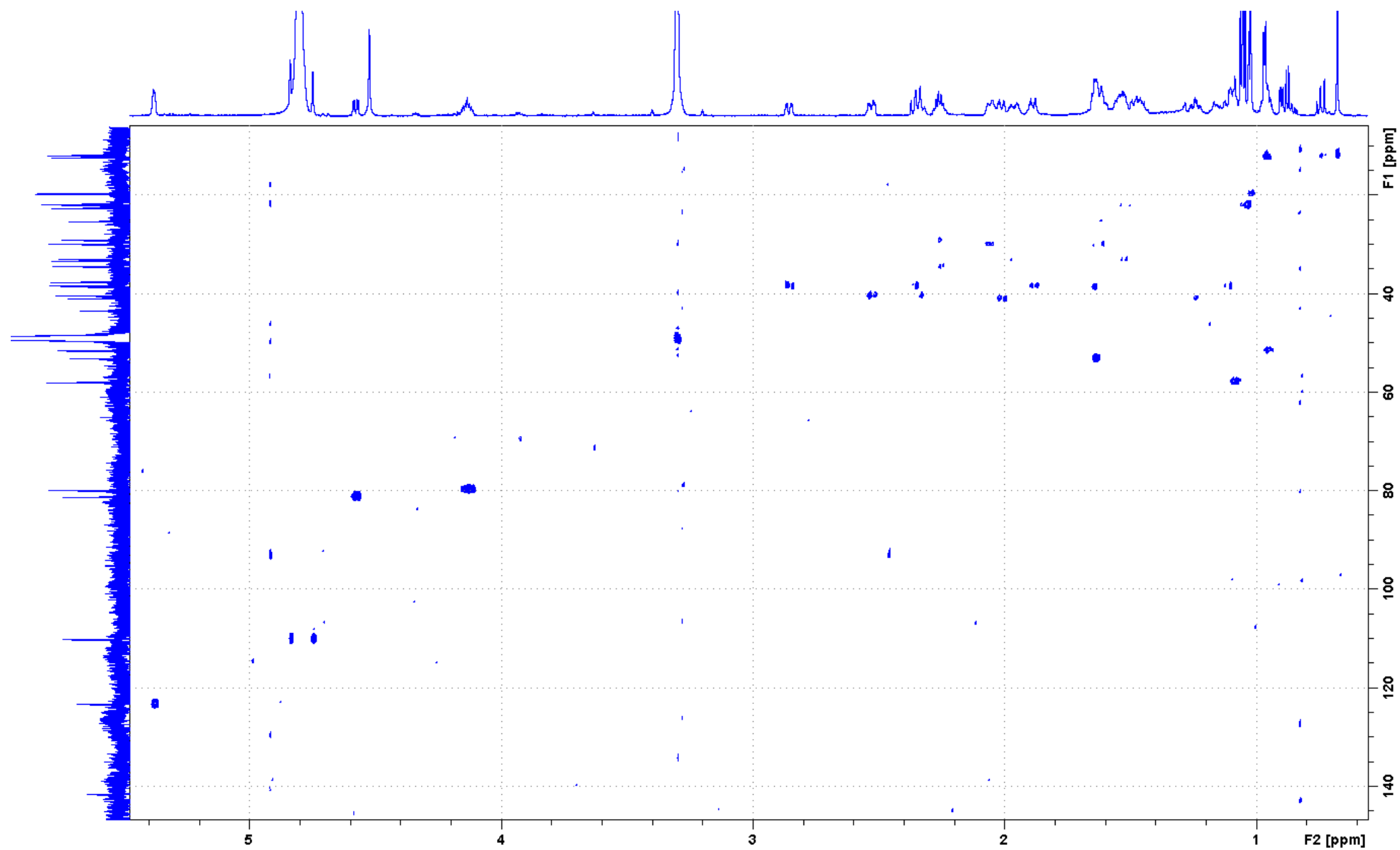


Figure S20. HMBC (700.13 MHz, CD₃OD) spectrum of compound **3**.

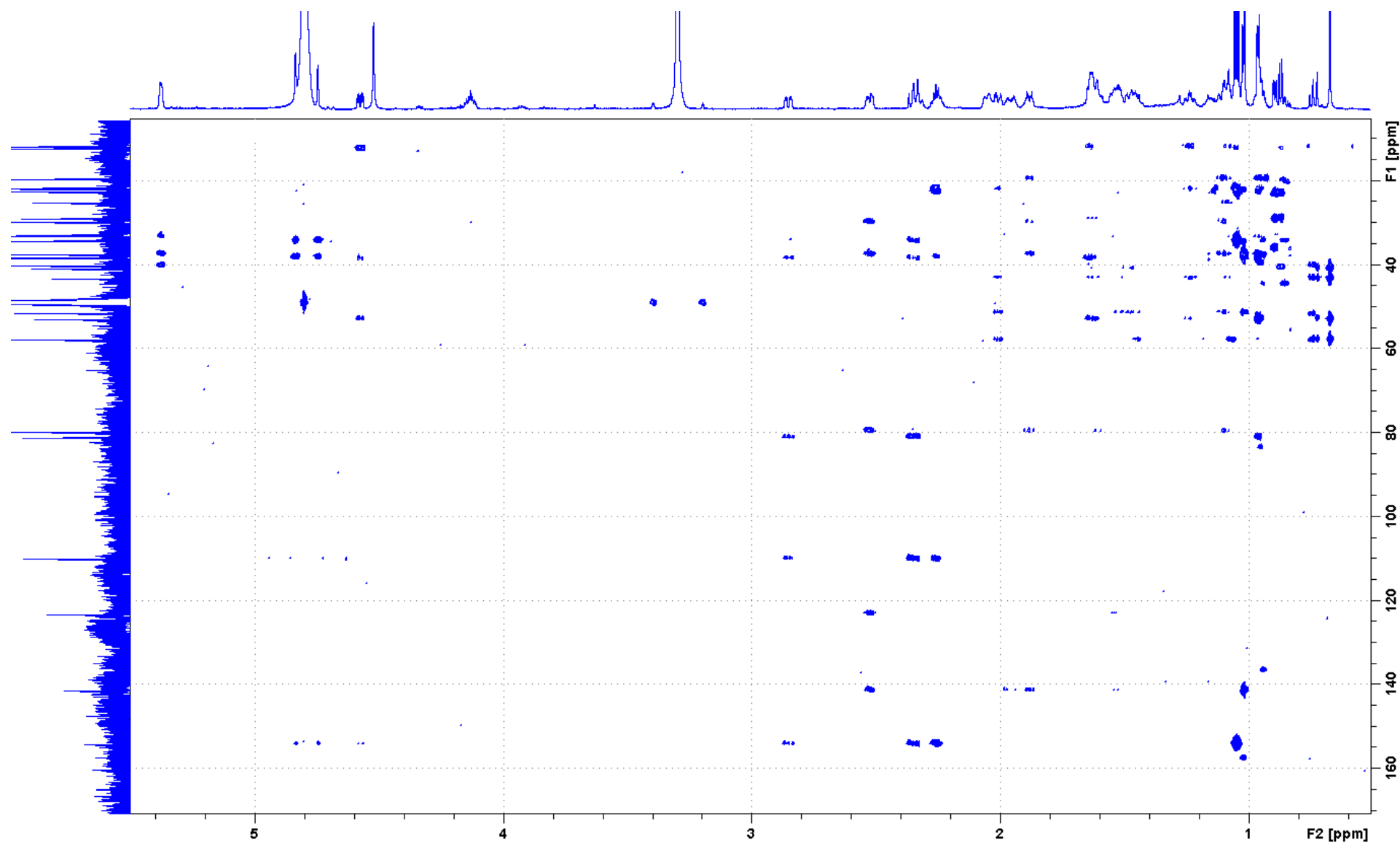


Figure S21. ROESY (700.13 MHz, CD₃OD) spectrum of compound **3**.

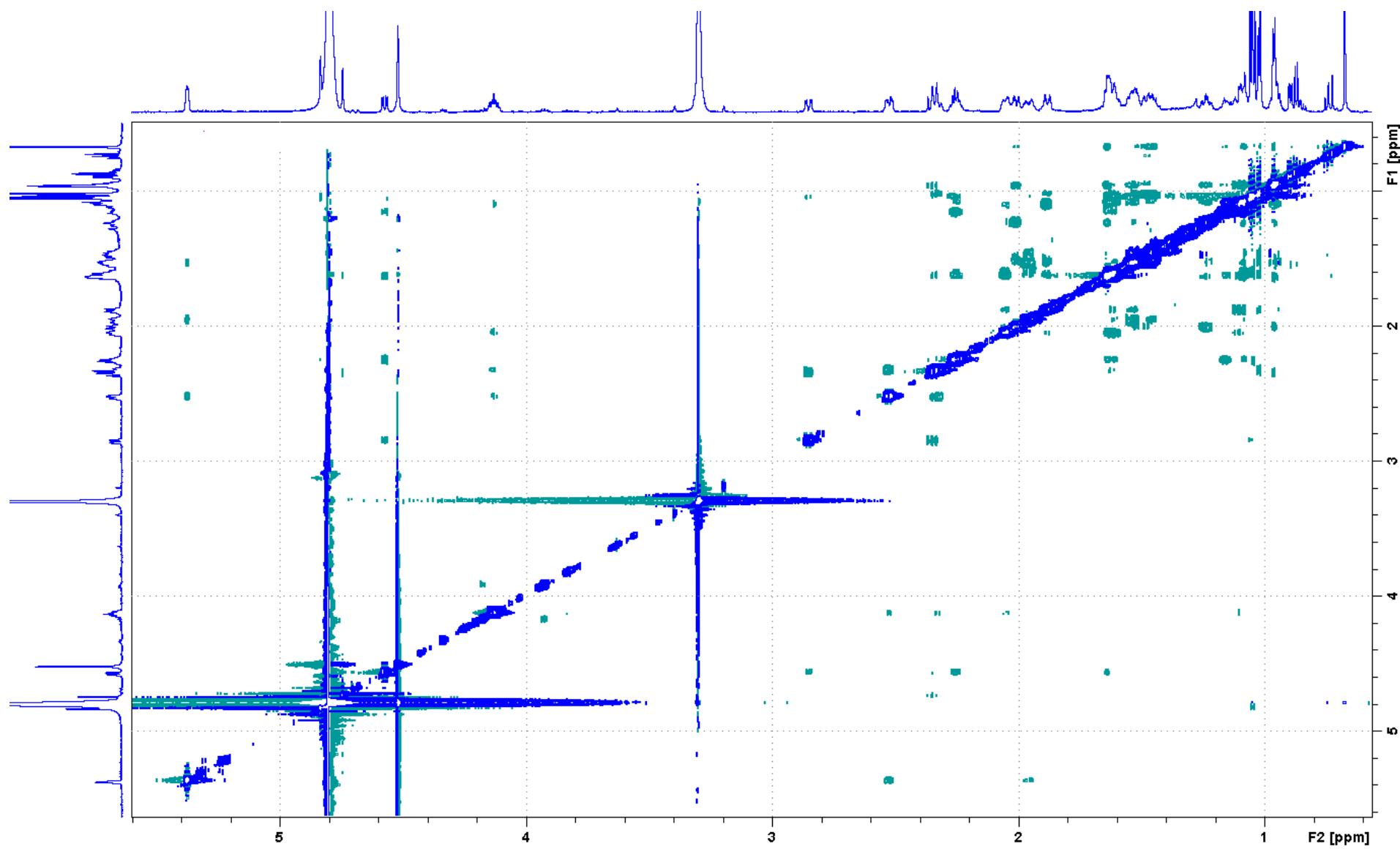
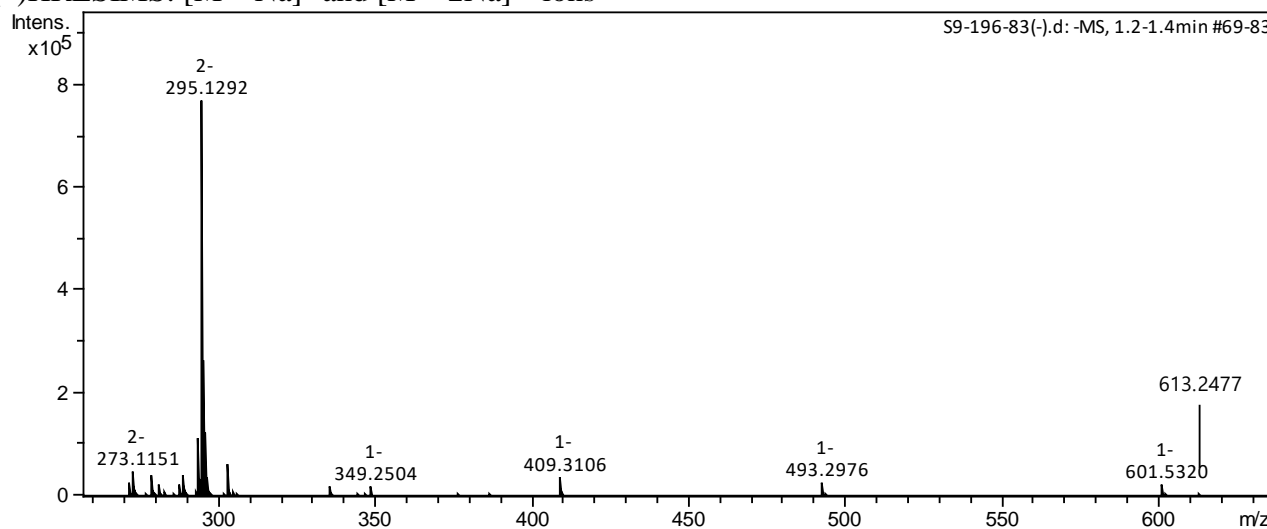
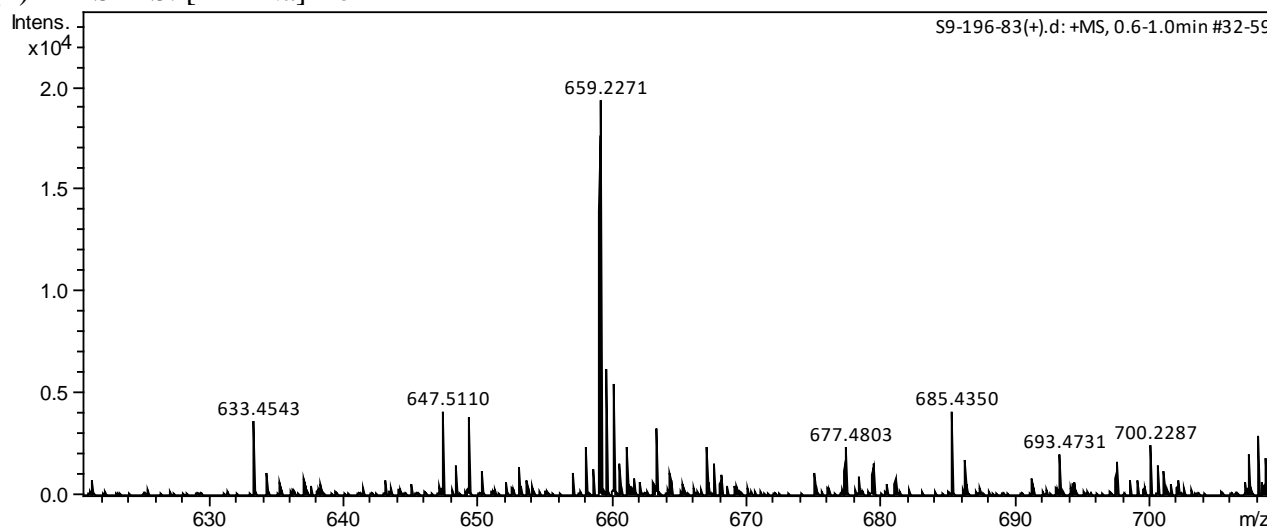


Figure S22. HRESIMS and HRESIMS/MS spectra of compound **4**

(-)HRESIMS: $[M - Na]^-$ and $[M - 2Na]^{2-}$ ions



(+)HRESIMS: $[M + Na]^+$ ion



(-)HRESIMS/MS of the $[M - 2Na]^{2-}$ ion at m/z 295.1280

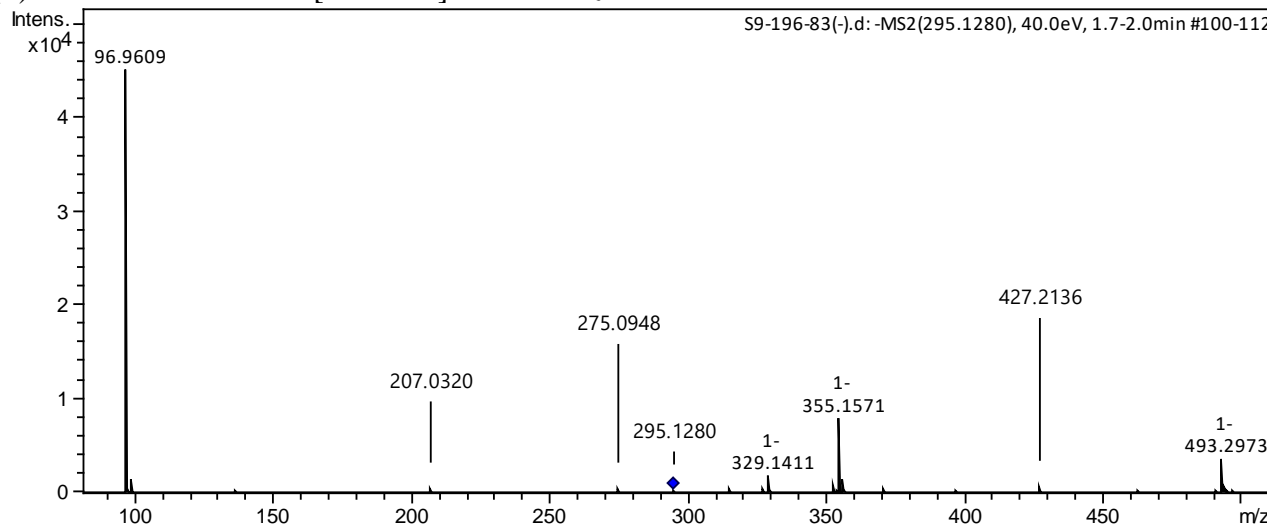


Figure S23. ^1H NMR (700.13 MHz, CD_3OD) spectrum of compound **4**.

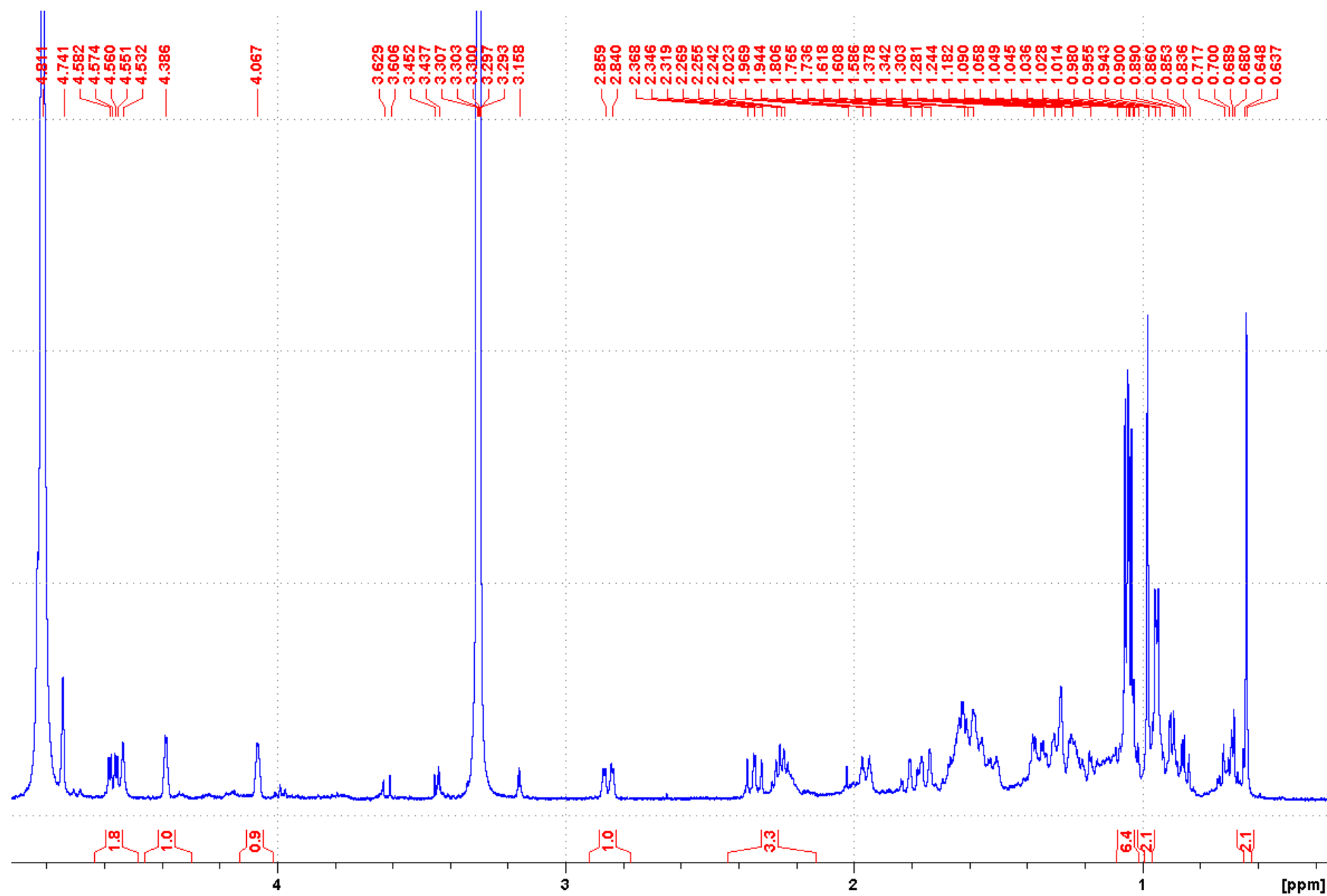


Figure S24. ^{13}C NMR (176.04 MHz, CD_3OD) spectrum of compound **4**.

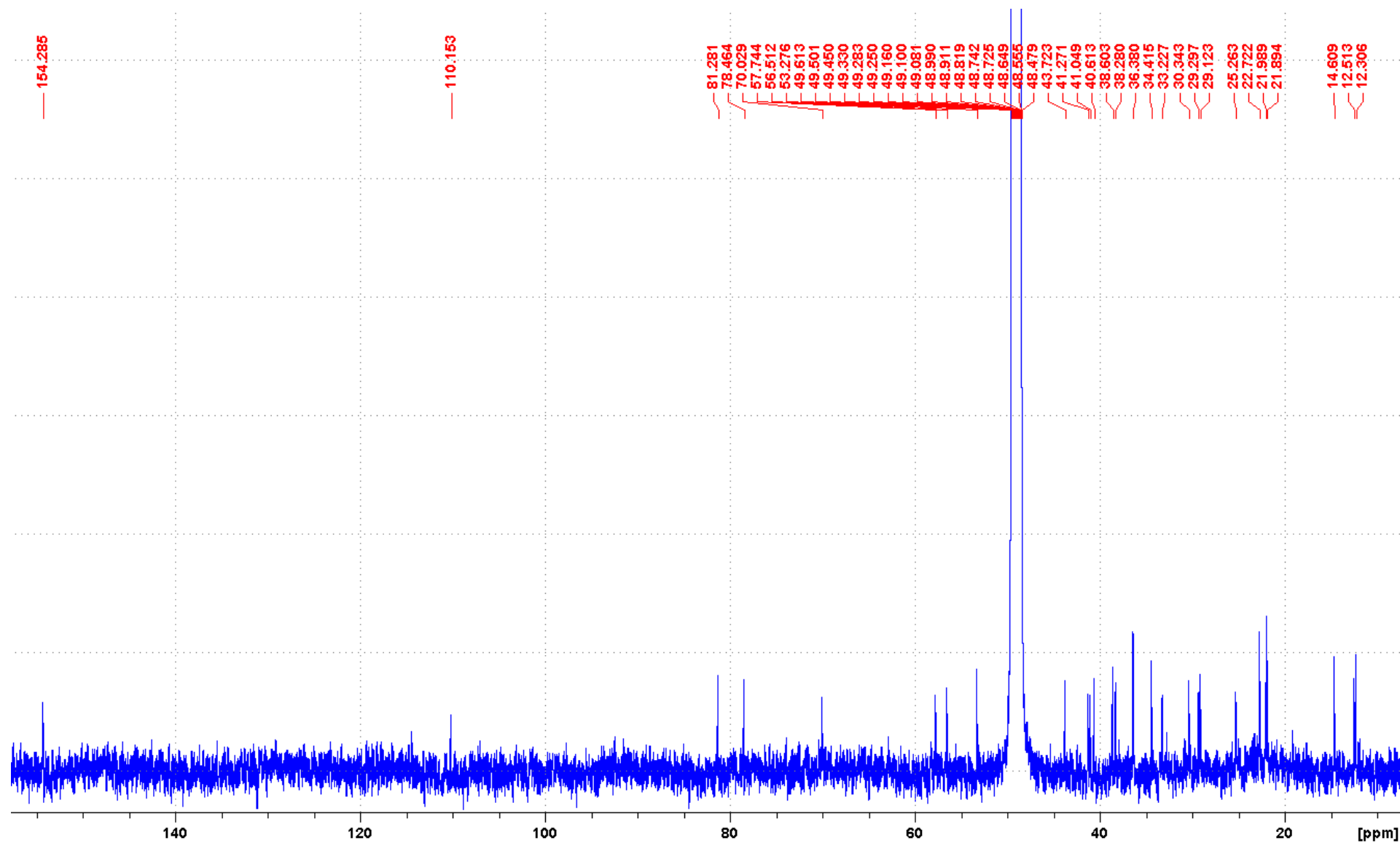


Figure S25. ^1H - ^1H COSY (700.13 MHz, CD_3OD) spectrum of compound **4**.

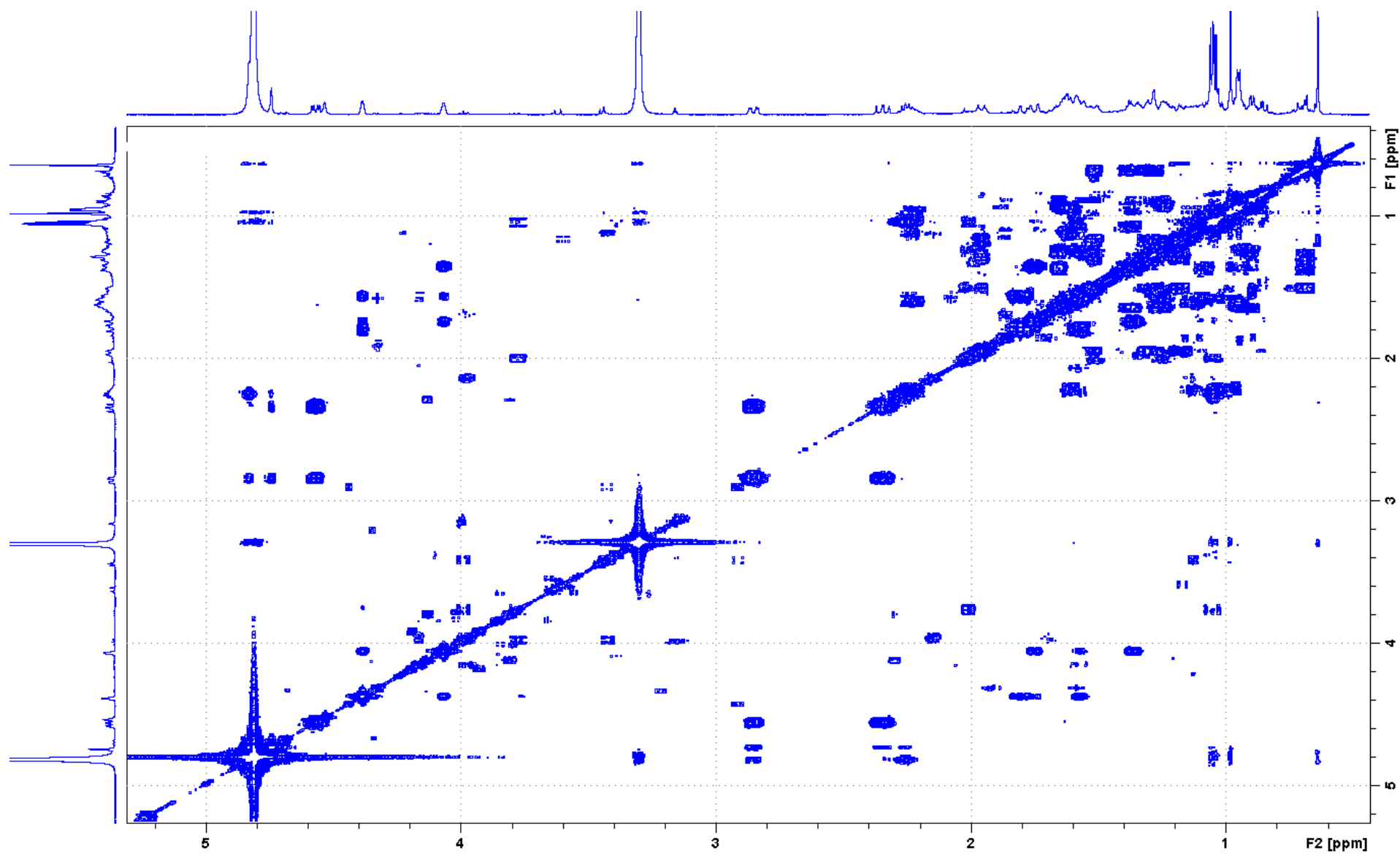


Figure S26. HSQC (700.13 MHz, CD₃OD) spectrum of compound **4**.

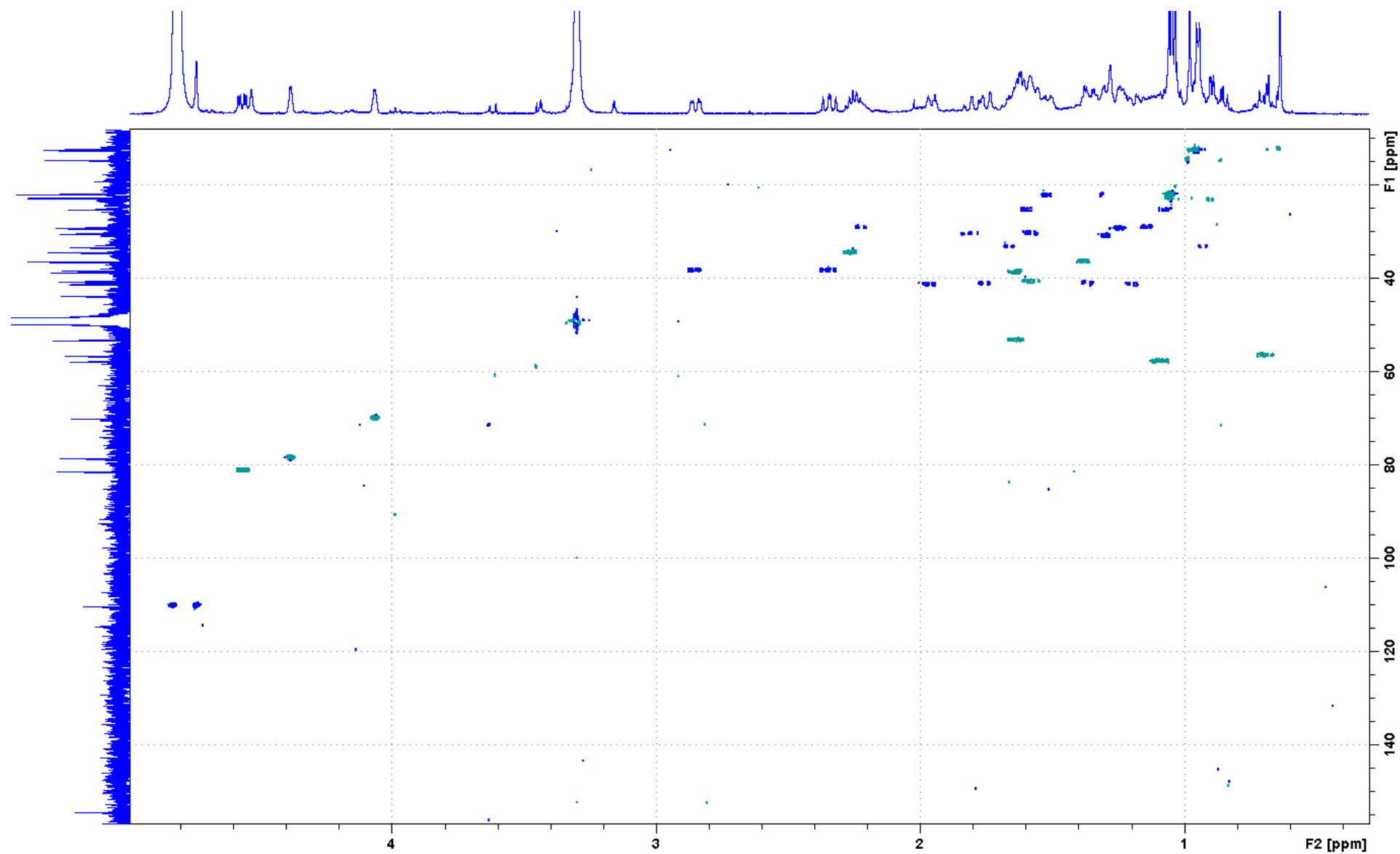


Figure S27. HMBC (700.13 MHz, CD₃OD) spectrum of compound **4**.

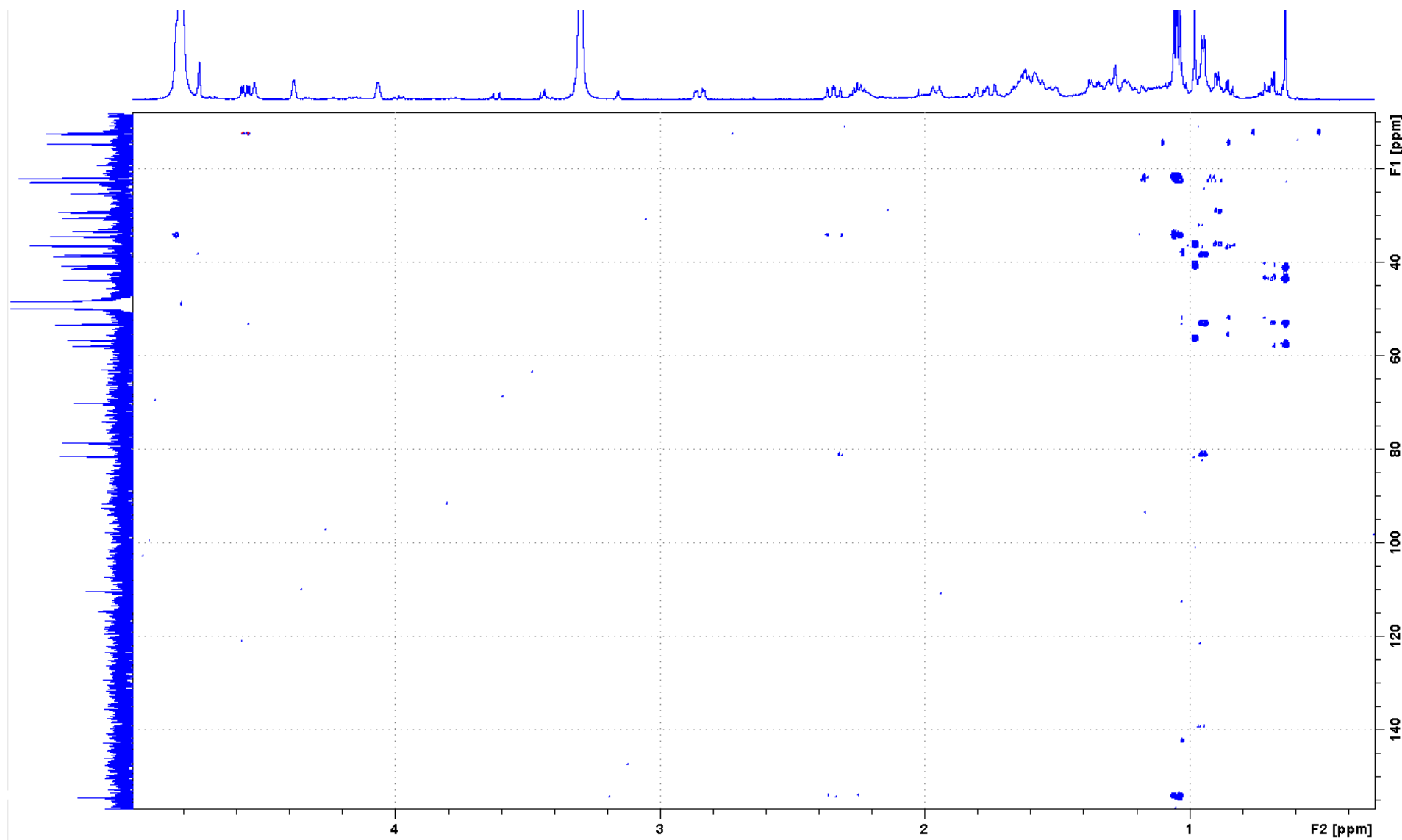


Figure S28. ROESY (700.13 MHz, CD₃OD) spectrum of compound **4**.

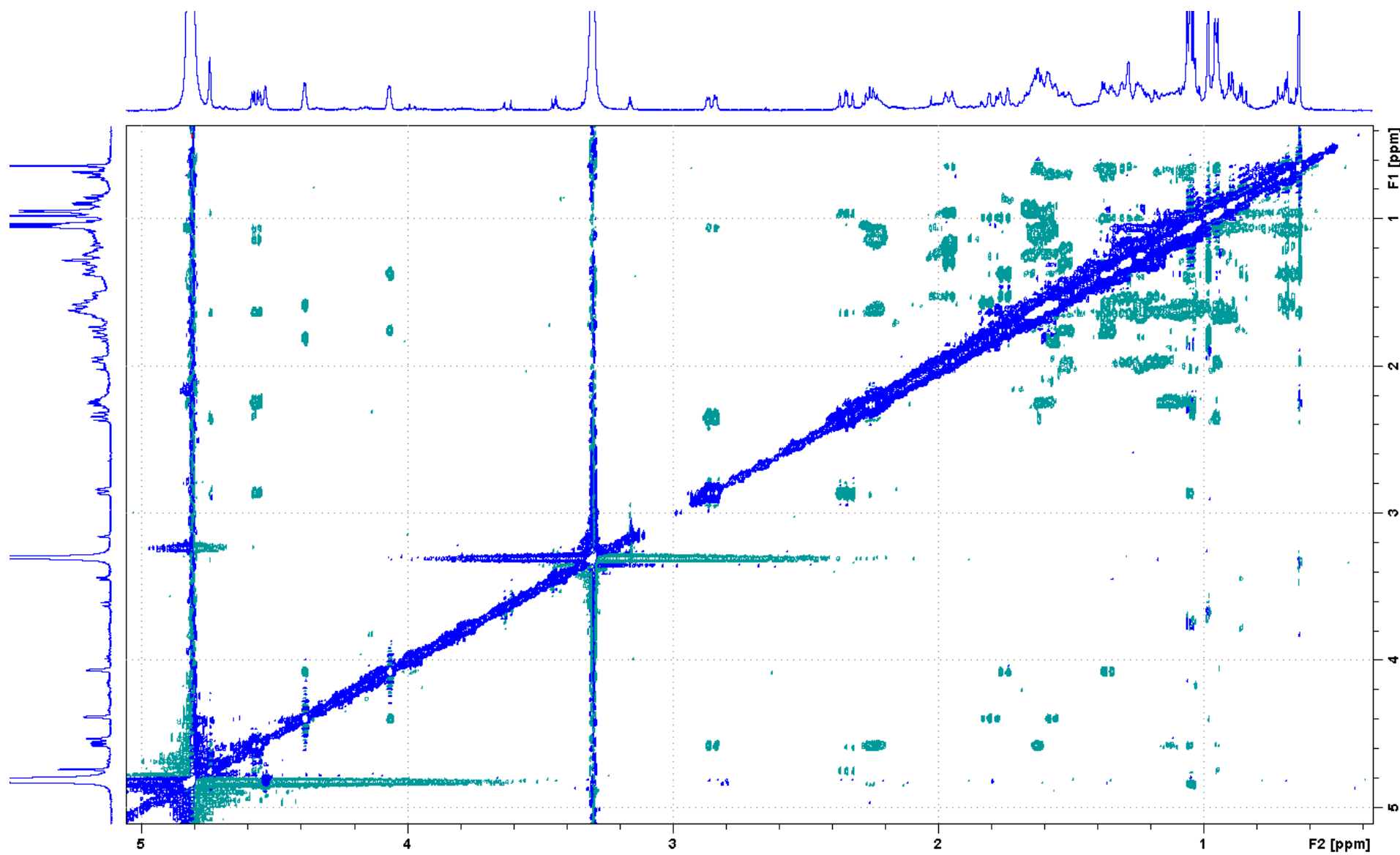


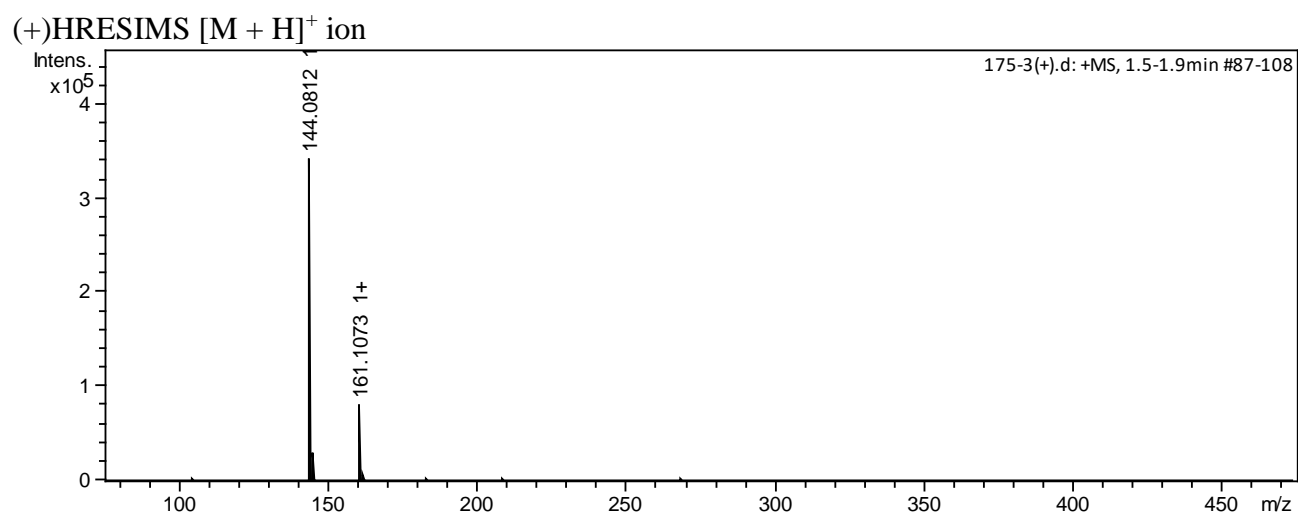
Figure S29. HRESIMS spectrum of compound **5**

Figure S30. ^1H NMR (700.13 MHz, CD_3OD) spectrum of compound **5**

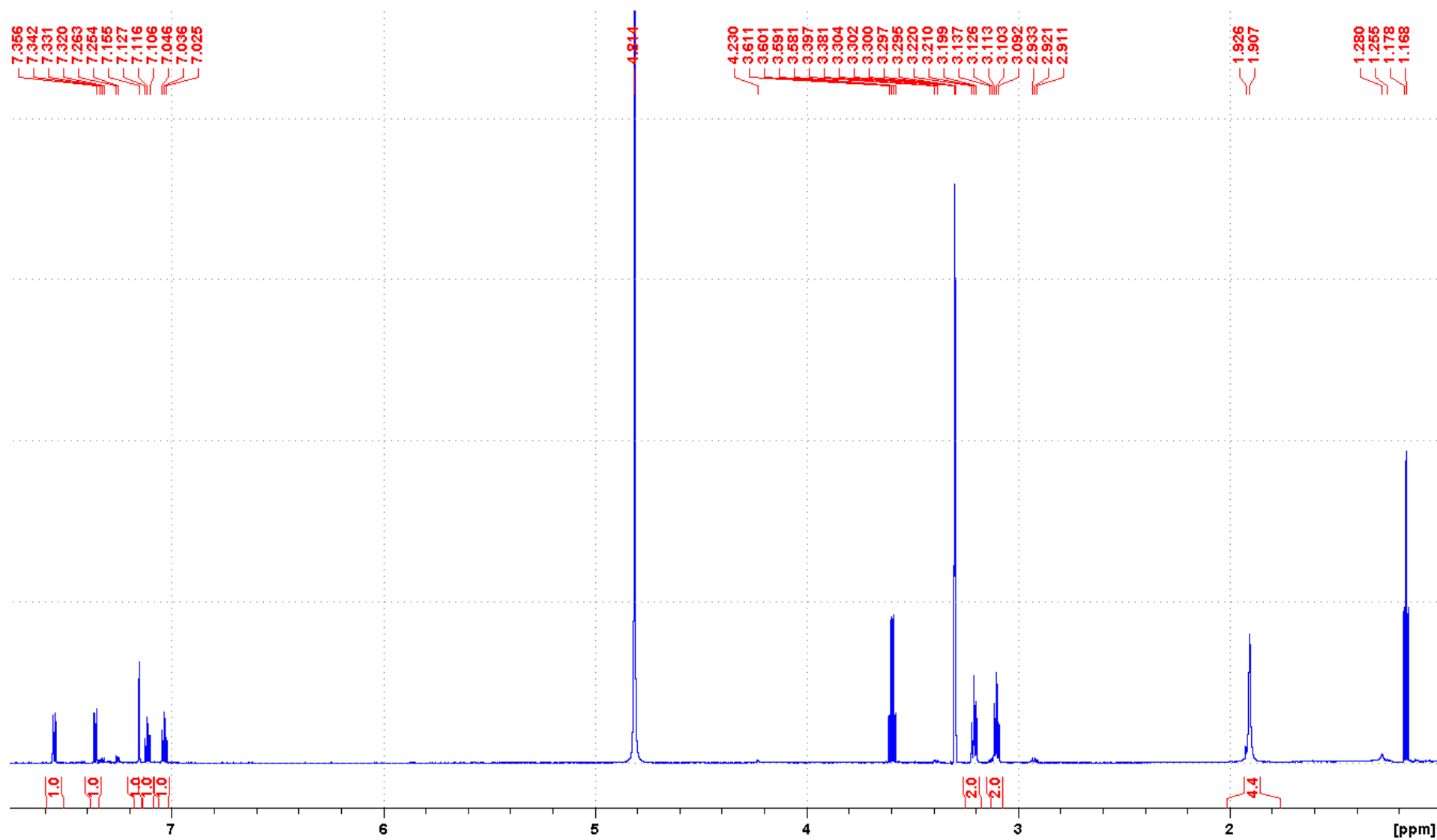


Figure S31. ^{13}C NMR (176.04 MHz, CD_3OD) spectrum of compound **5**

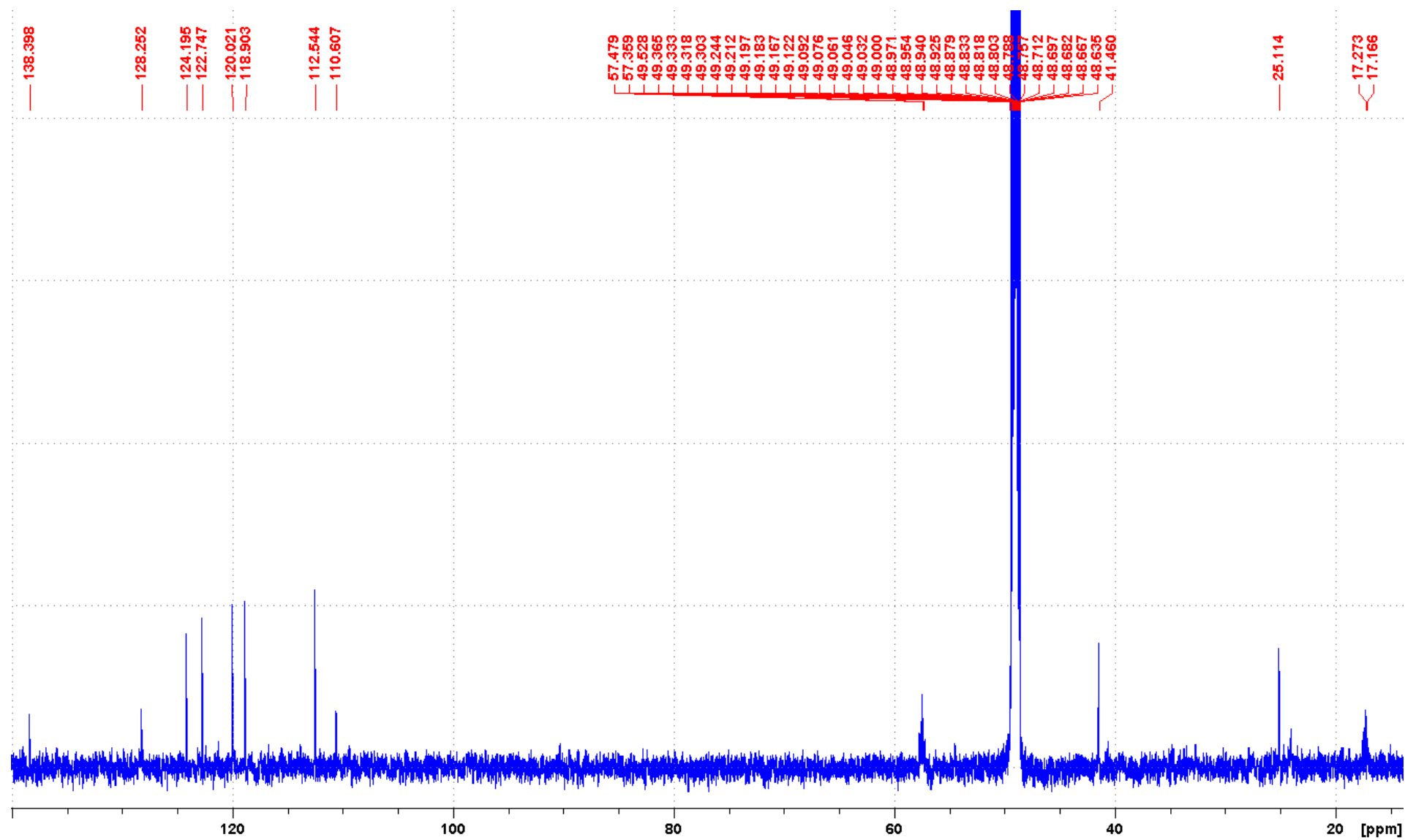


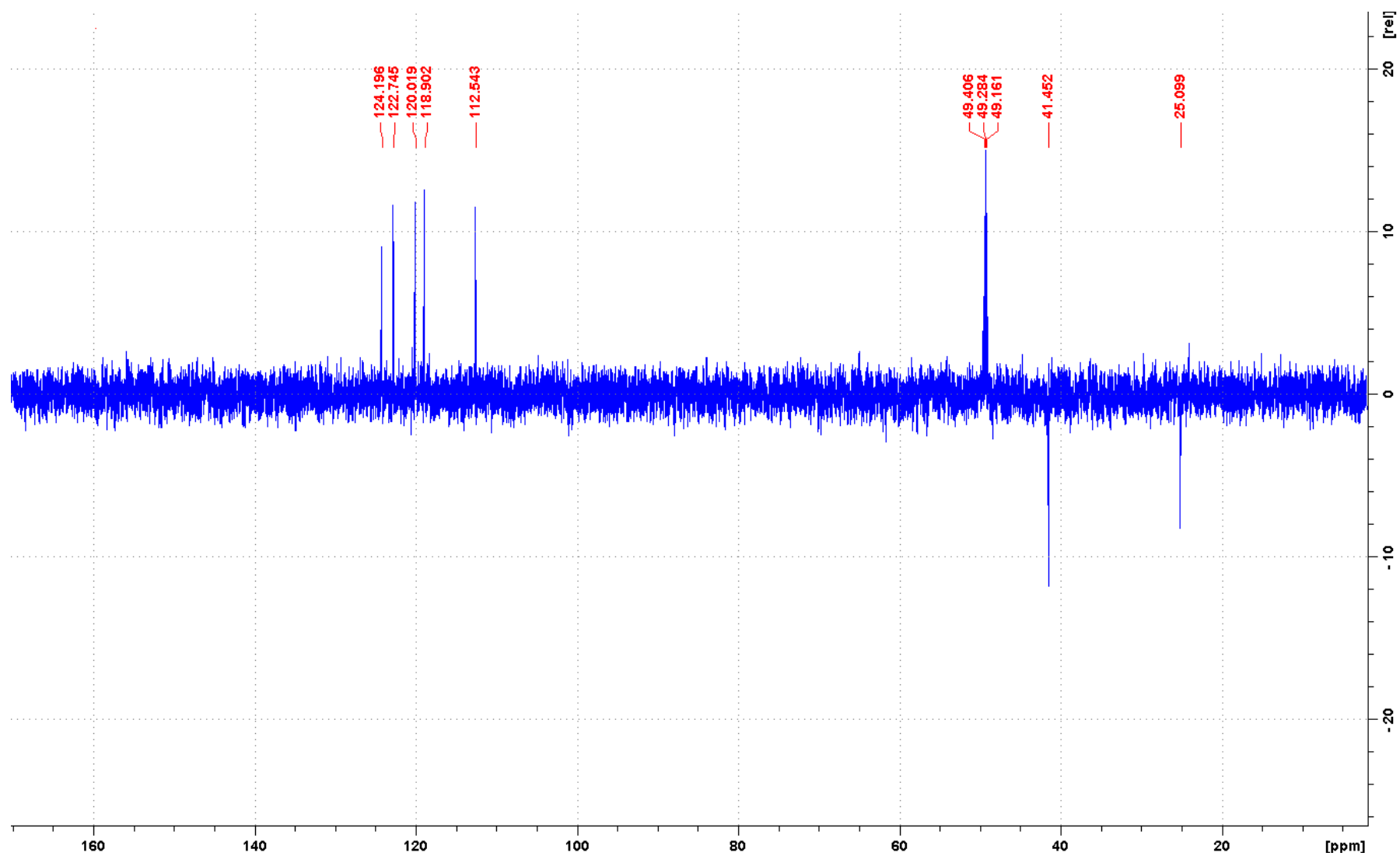
Figure S32. DEPT (176.04 MHz, CD₃OD) spectrum of compound **5**

Figure S33. ^1H - ^1H COSY (700.13 MHz, CD_3OD) spectrum of compound **5**

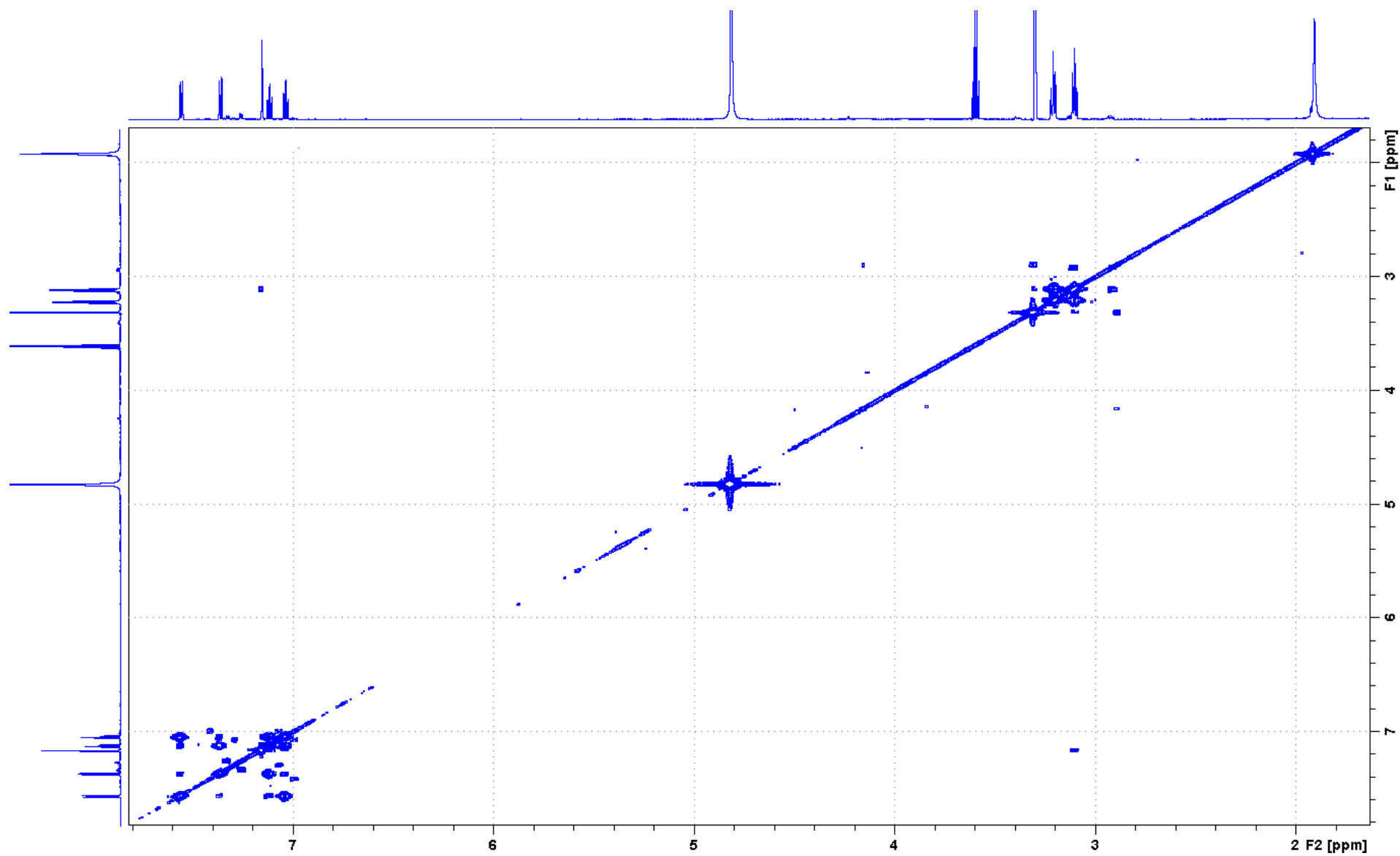


Figure S34. HSQC (700.13 MHz, CD₃OD) spectrum of compound **5**

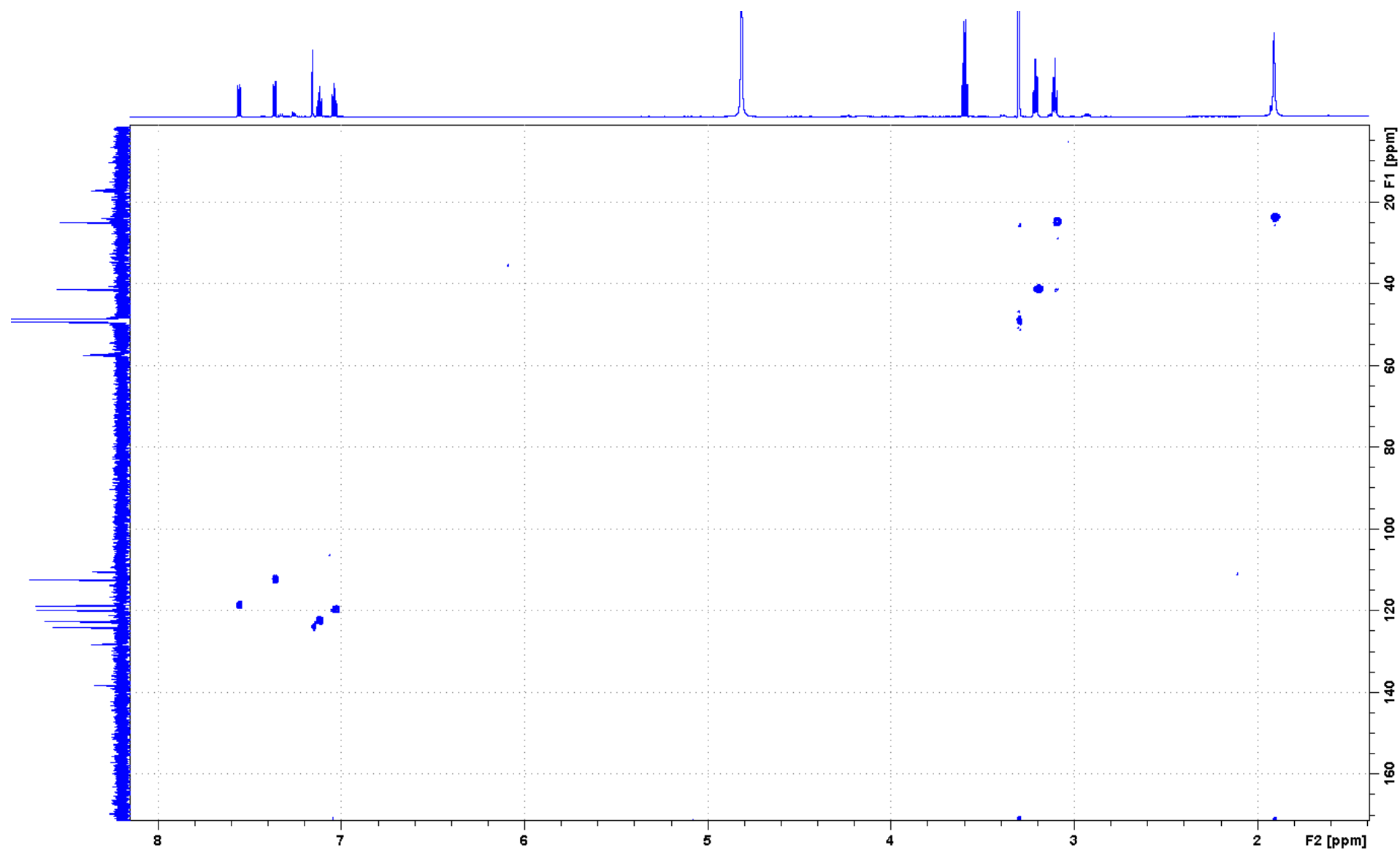


Figure S35. HMBC (700.13 MHz, CD₃OD) spectrum of compound **5**

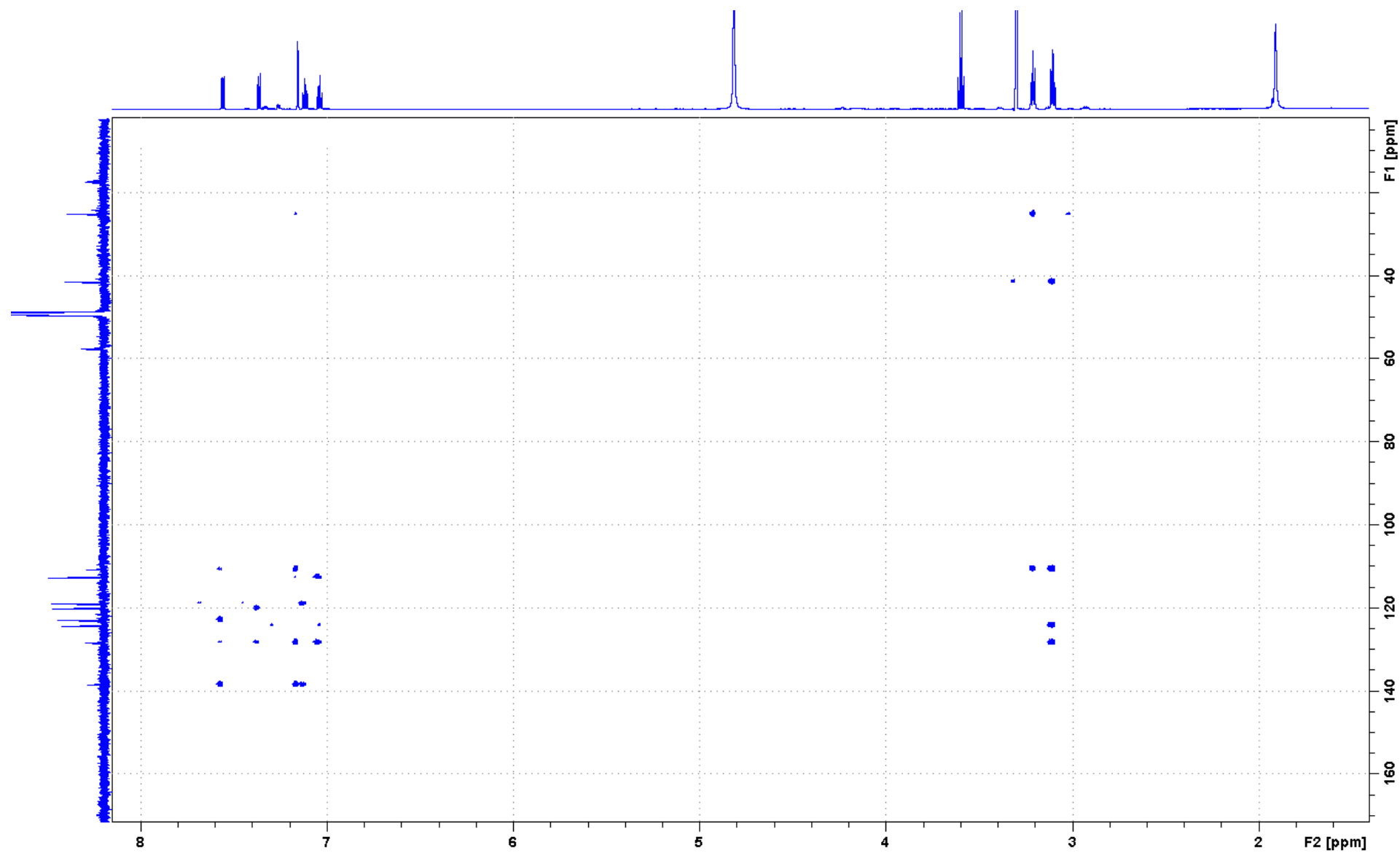


Figure S36. ROESY (700.13 MHz, CD₃OD) spectrum of compound **5**

