

Supplementary Materials

Naphthoquinone Derivatives from *Angustimassarina populi* CF-097565 Display Anti-tumour Activity in 3D Cultures of Breast Cancer cells

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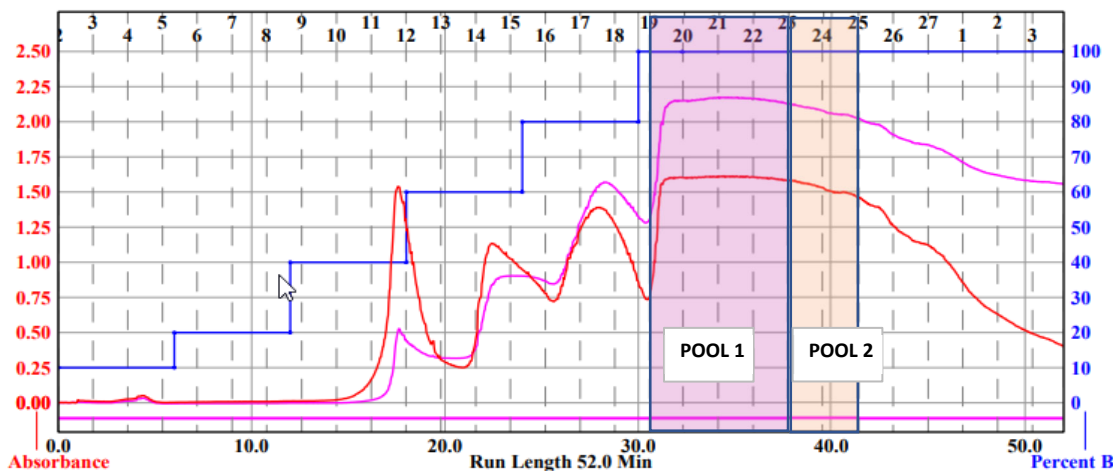


Figure S1. UV (210, red, and 280, pink trace, nm) chromatogram of preparative RP-Flash Chromatography of a 3L extract of the strain fermentation, in a stepped gradient (blue trace) of acetone in water from 10% to 100% of acetone in 52 min (6 min/step). Fractions combined according to similar bioactivity profile and chemical complexity are indicated as POOL 1 and 2.

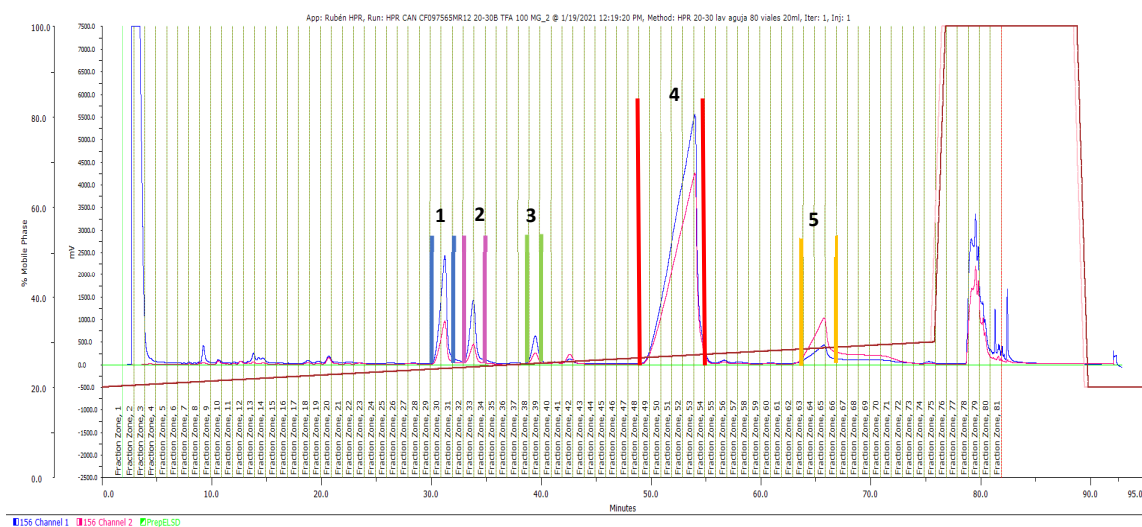


Figure S2. LC-UV (210, blue, and 280, orange trace, nm) chromatogram of POOL1 purification by preparative RP-HPLC applying a linear H₂O:CH₃CN gradient (CH₃CN: 20-30%: 1-76 min, 100%: 77-90 min). Both solvents contain 0.1% TFA. POOL2 behaved similarly.

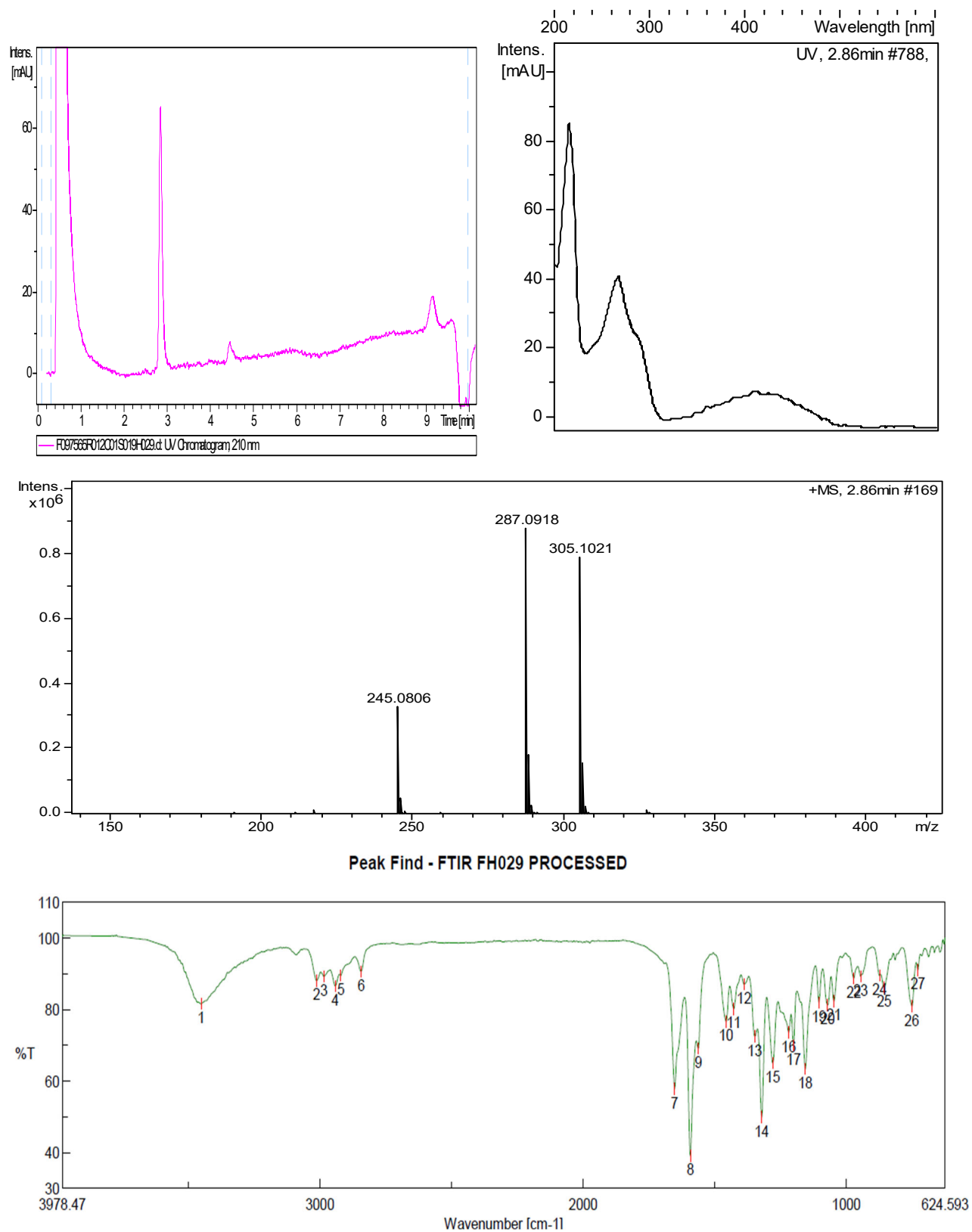


Figure S3. HPLC 210 nm trace, UV-Vis, ((+)-ESI-TOF) and IR spectra of compound **1** (purity 90% by UV).

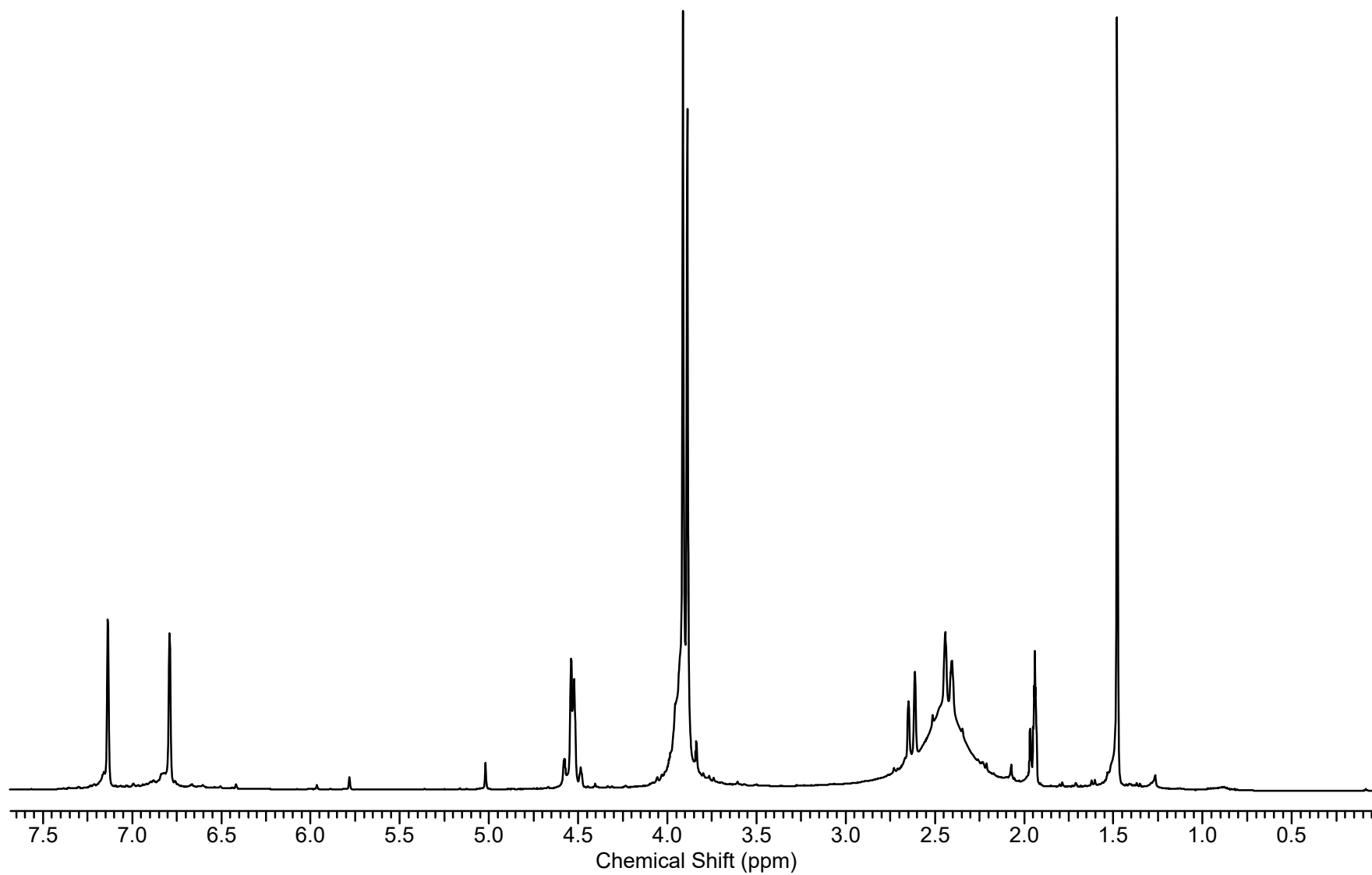


Figure S4. ^1H NMR spectrum ($\text{DMSO}-d_6$) of compound 1.

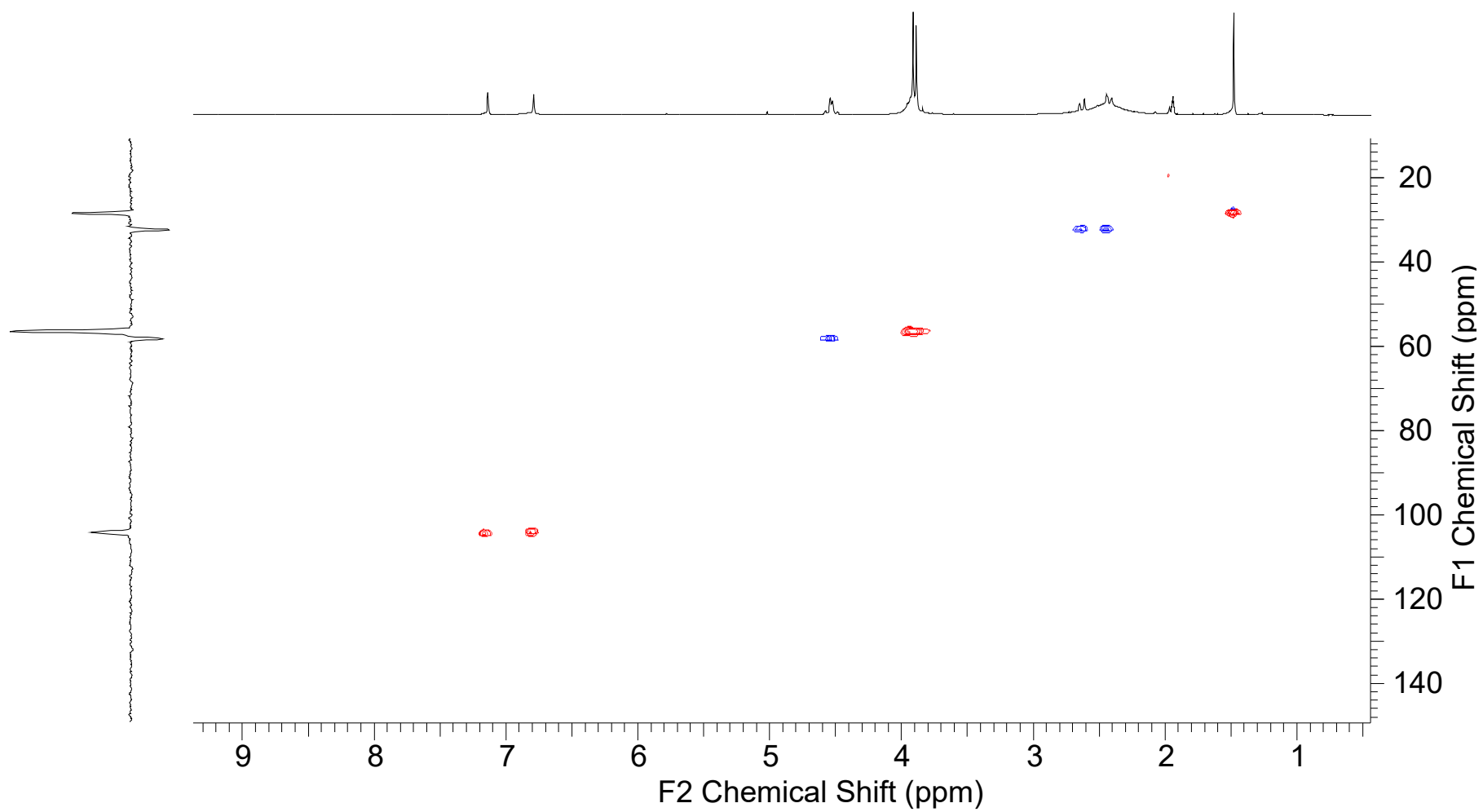


Figure S5. HSQC spectrum (DMSO- d_6) of compound 1.

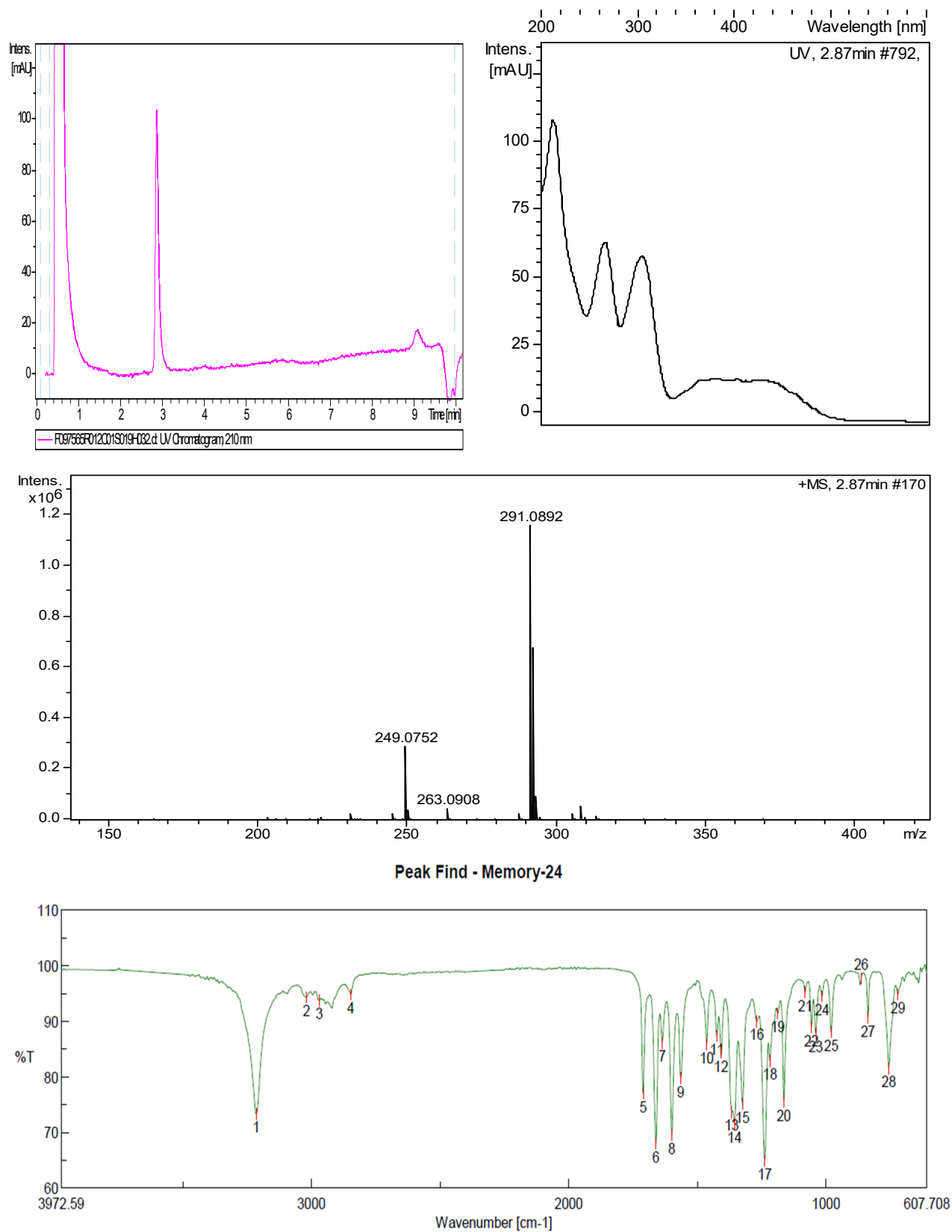


Figure S6. HPLC 210 nm trace, UV-Vis, ((+)-ESI-TOF) and IR spectra of compound **2** (purity 99% by UV).

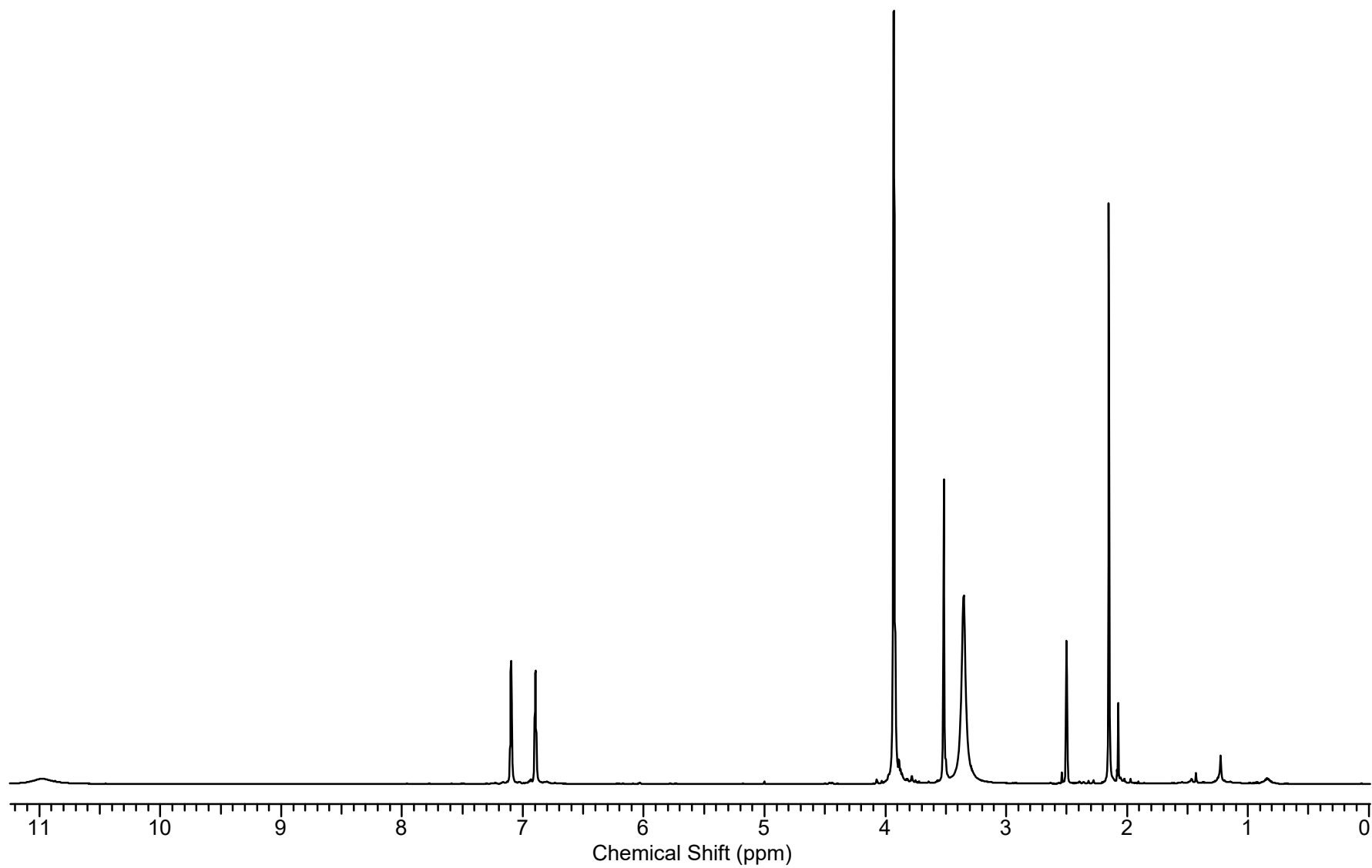


Figure S7. ^1H NMR spectrum ($\text{DMSO}-d_6$) of compound 2.

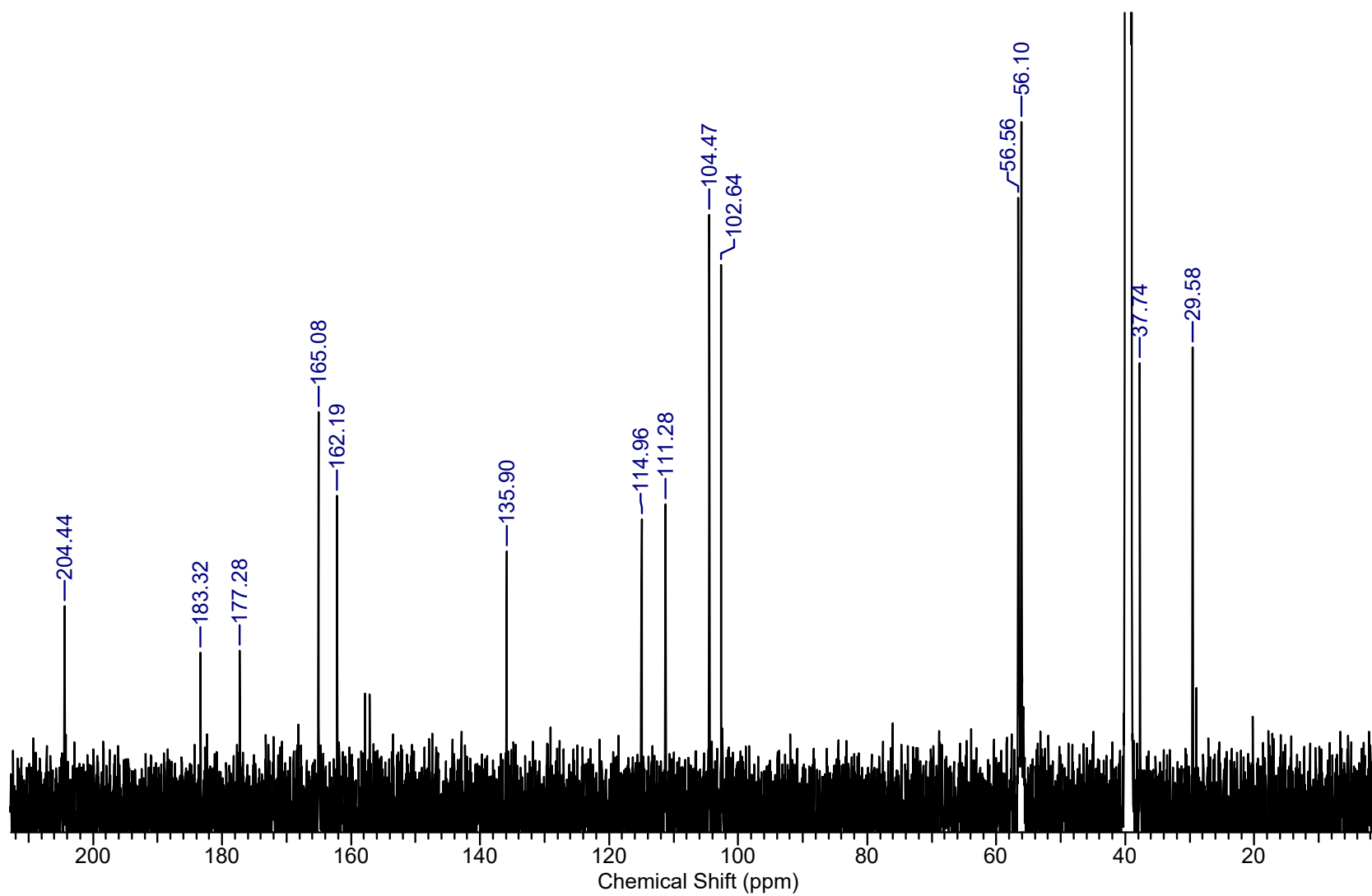


Figure S8. ¹³C NMR spectrum (DMSO-*d*₆) of compound 2.

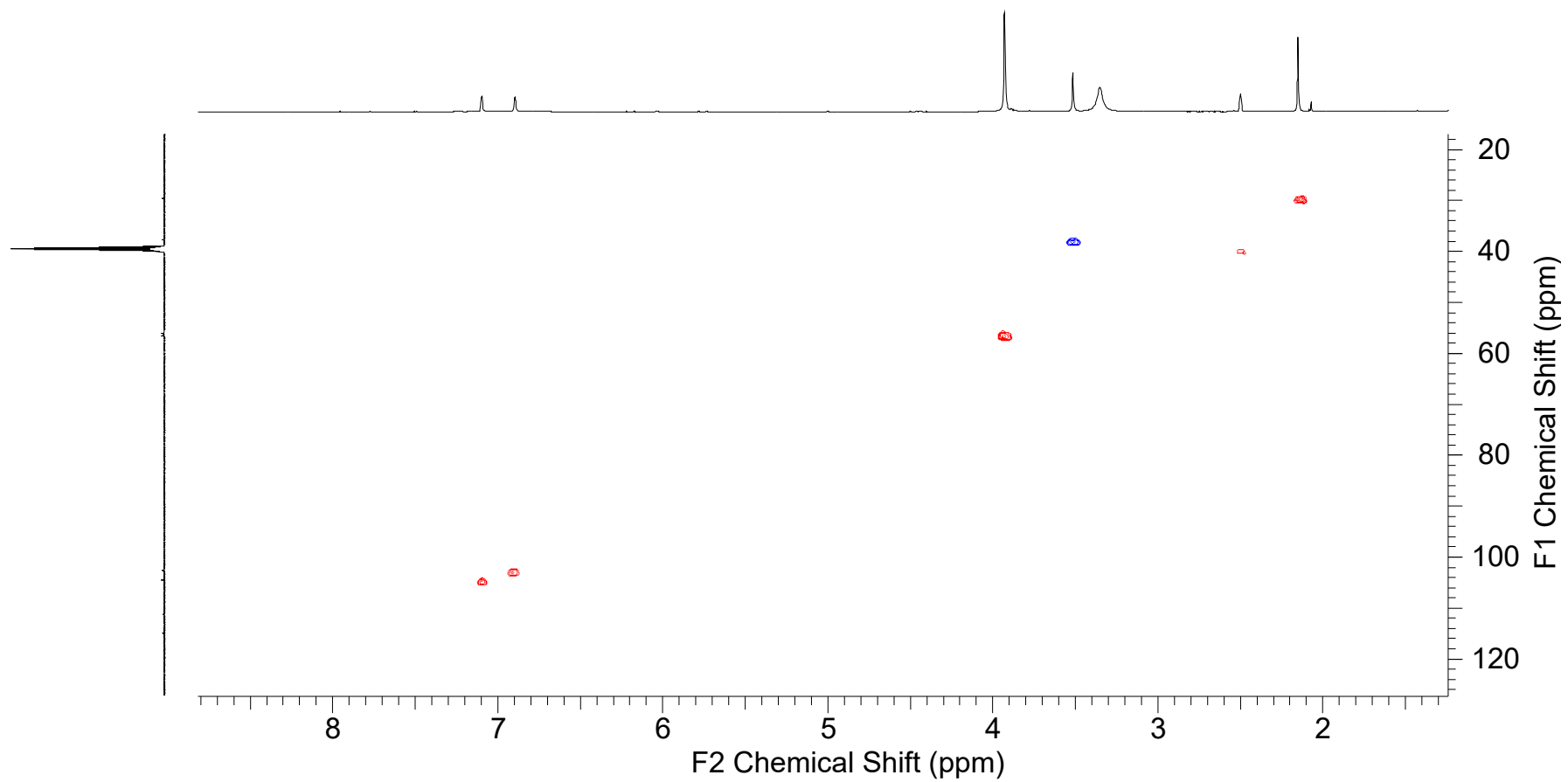


Figure S9. HSQC (DMSO-*d*₆) of compound 2.

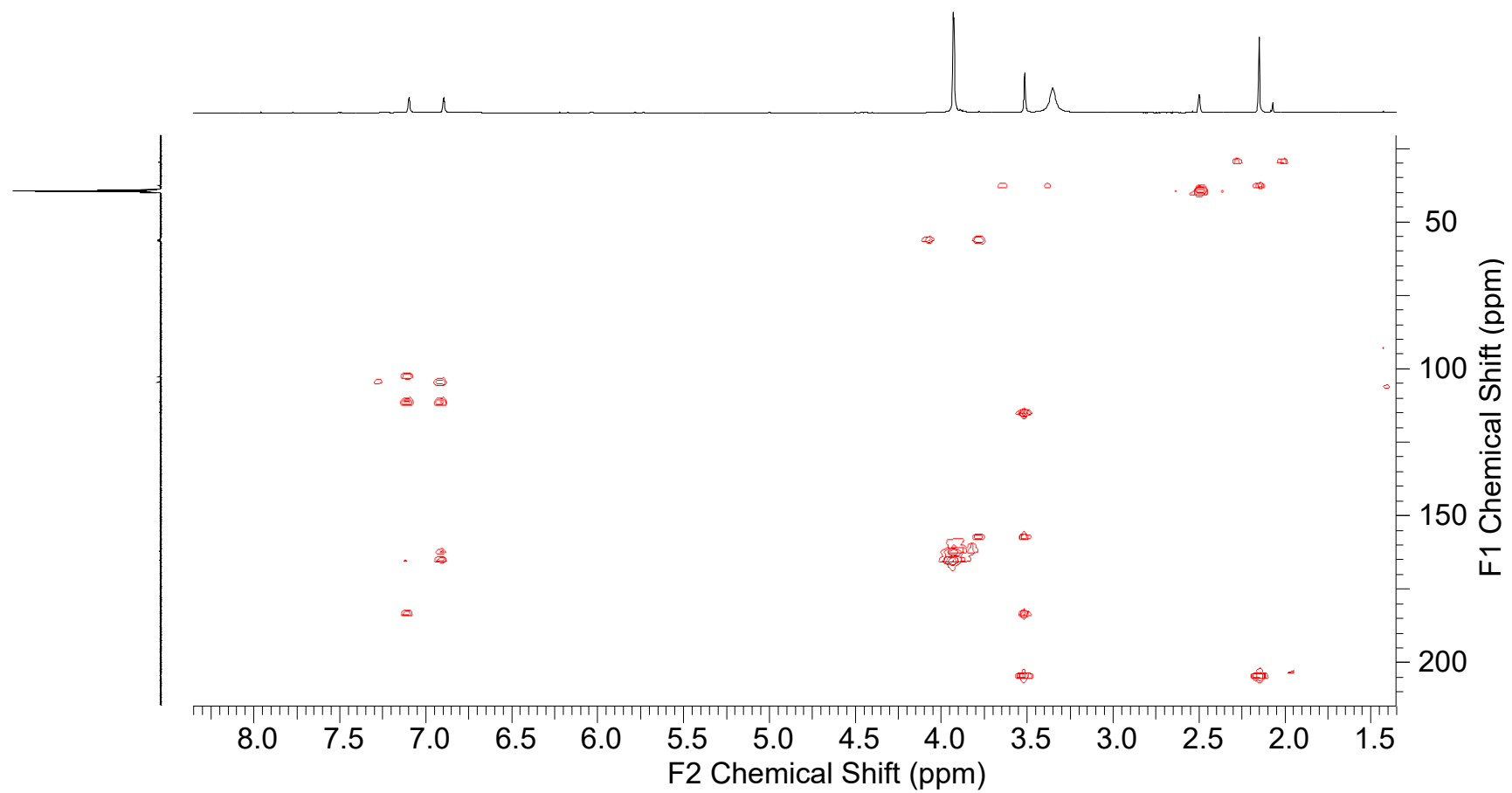


Figure S10. HMBC (DMSO- d_6) of compound 2.

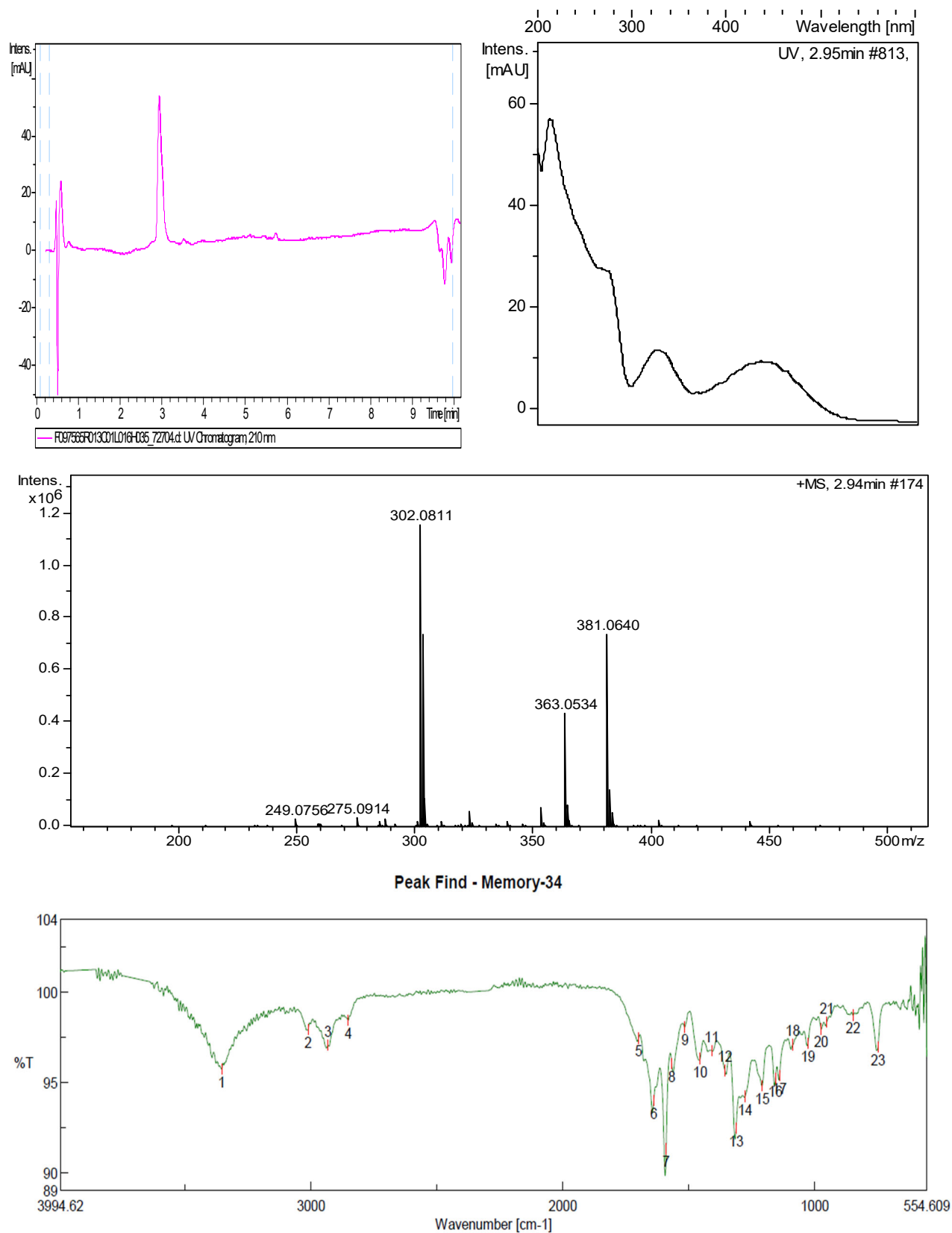


Figure S11. HPLC 210 nm trace, UV-Vis, ((+)-ESI-TOF) and IR spectra of compound **3** (purity 95% by UV).

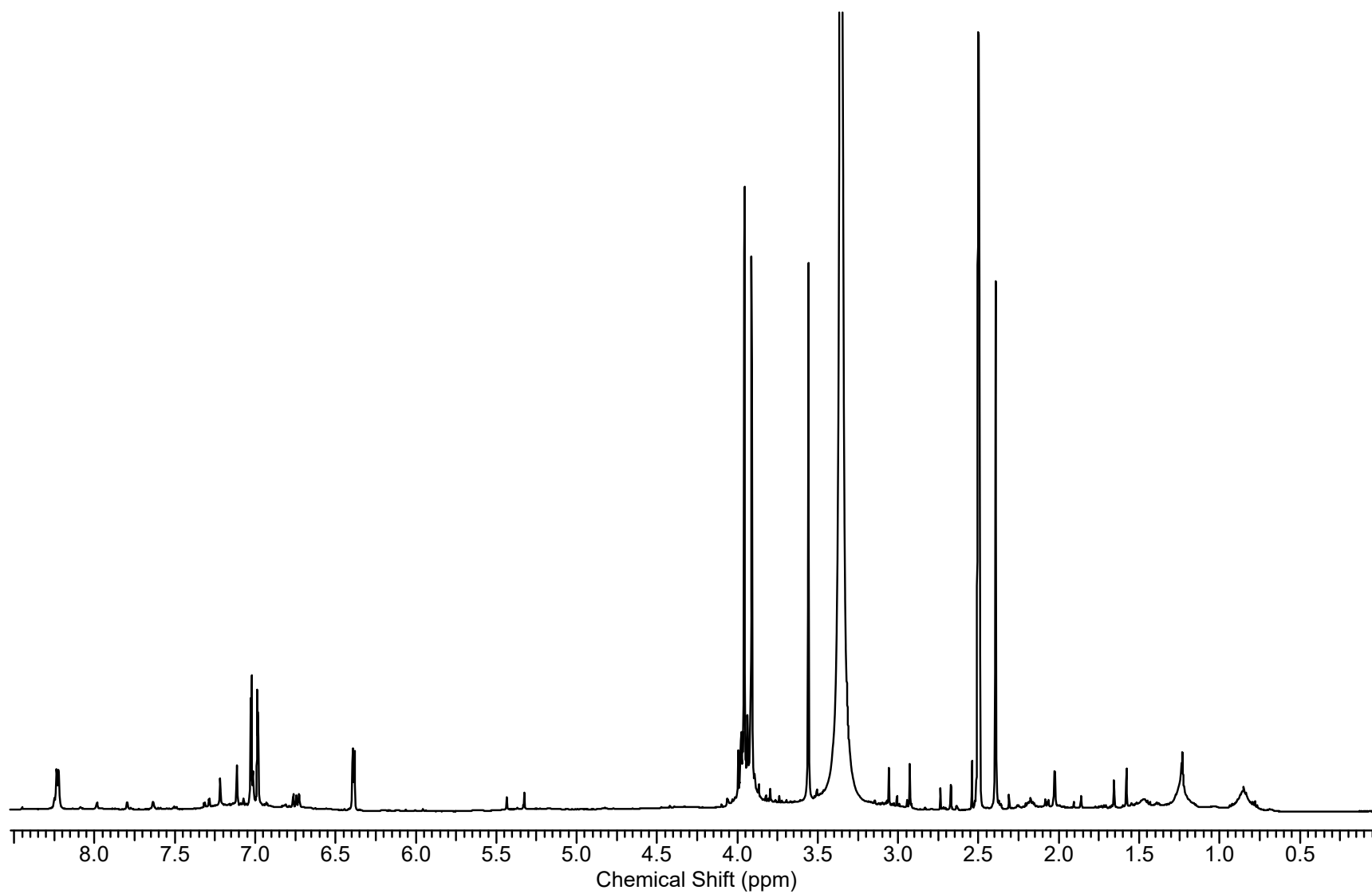


Figure S12. ^1H NMR spectrum ($\text{DMSO-}d_6$) of compound 3.

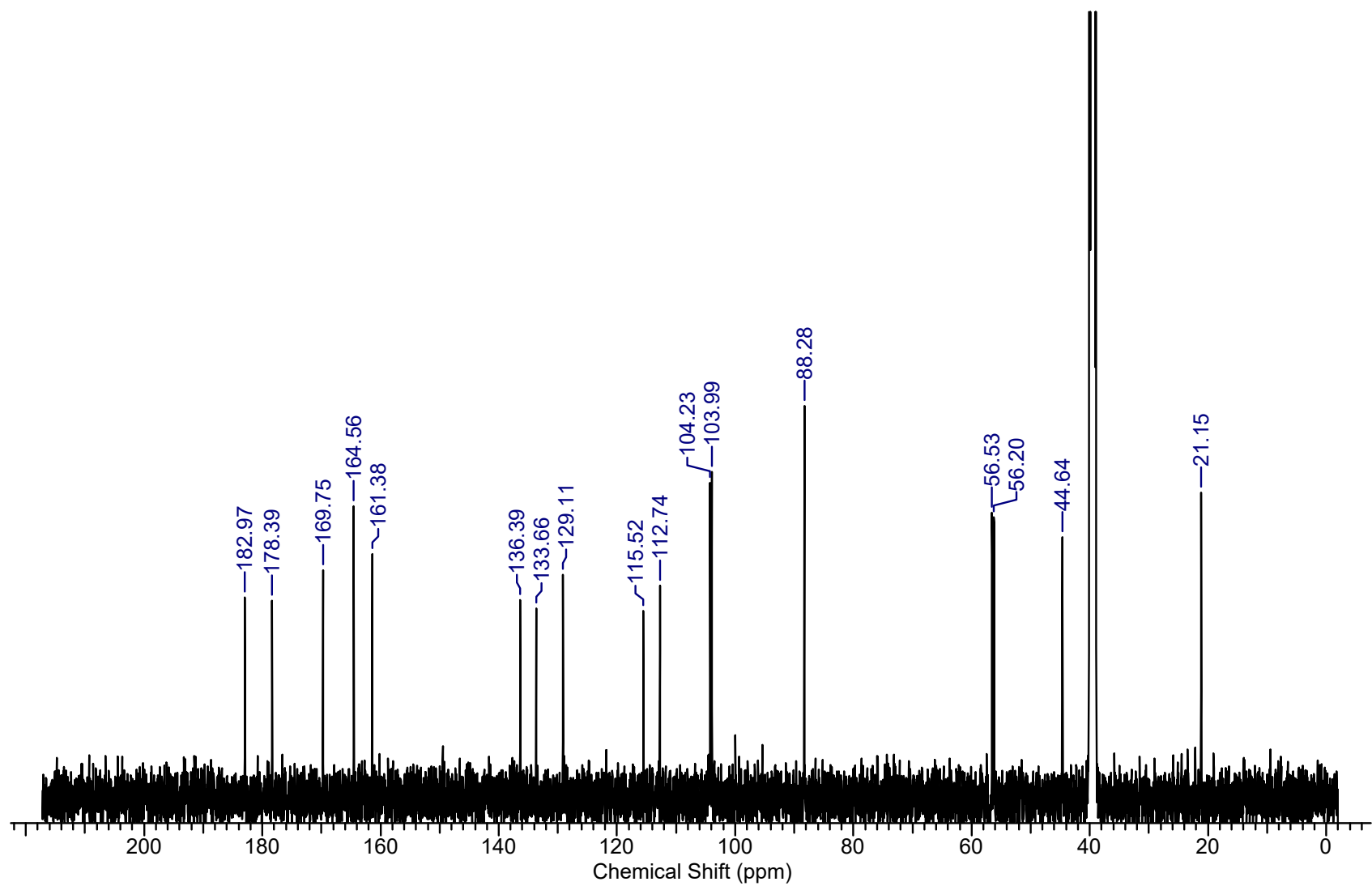


Figure S13. ^{13}C NMR spectrum ($\text{DMSO}-d_6$) of compound 3.

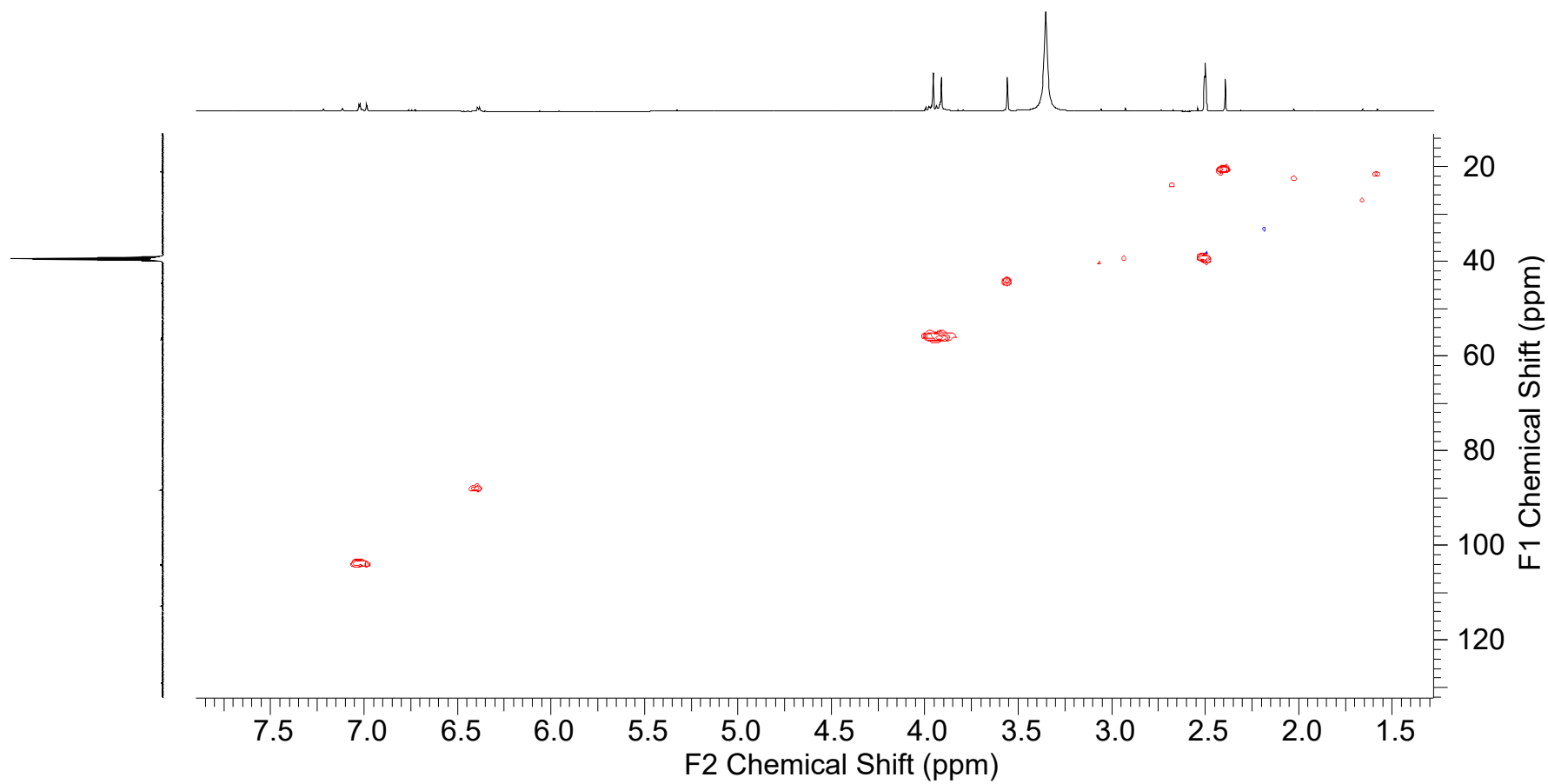


Figure S14. HSQC (DMSO- d_6) of compound 3.

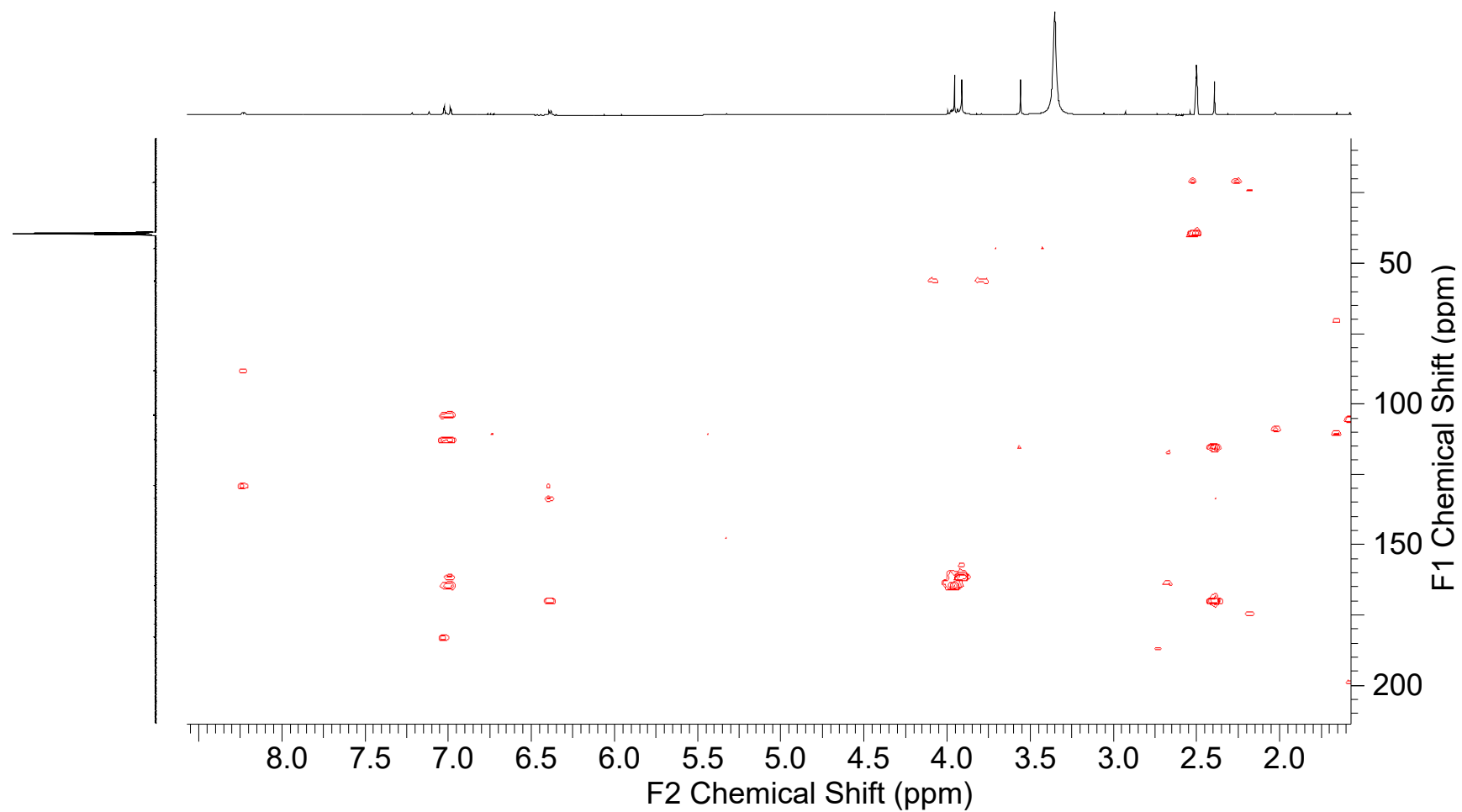


Figure S15. HMBC (DMSO-*d*₆) of compound 3

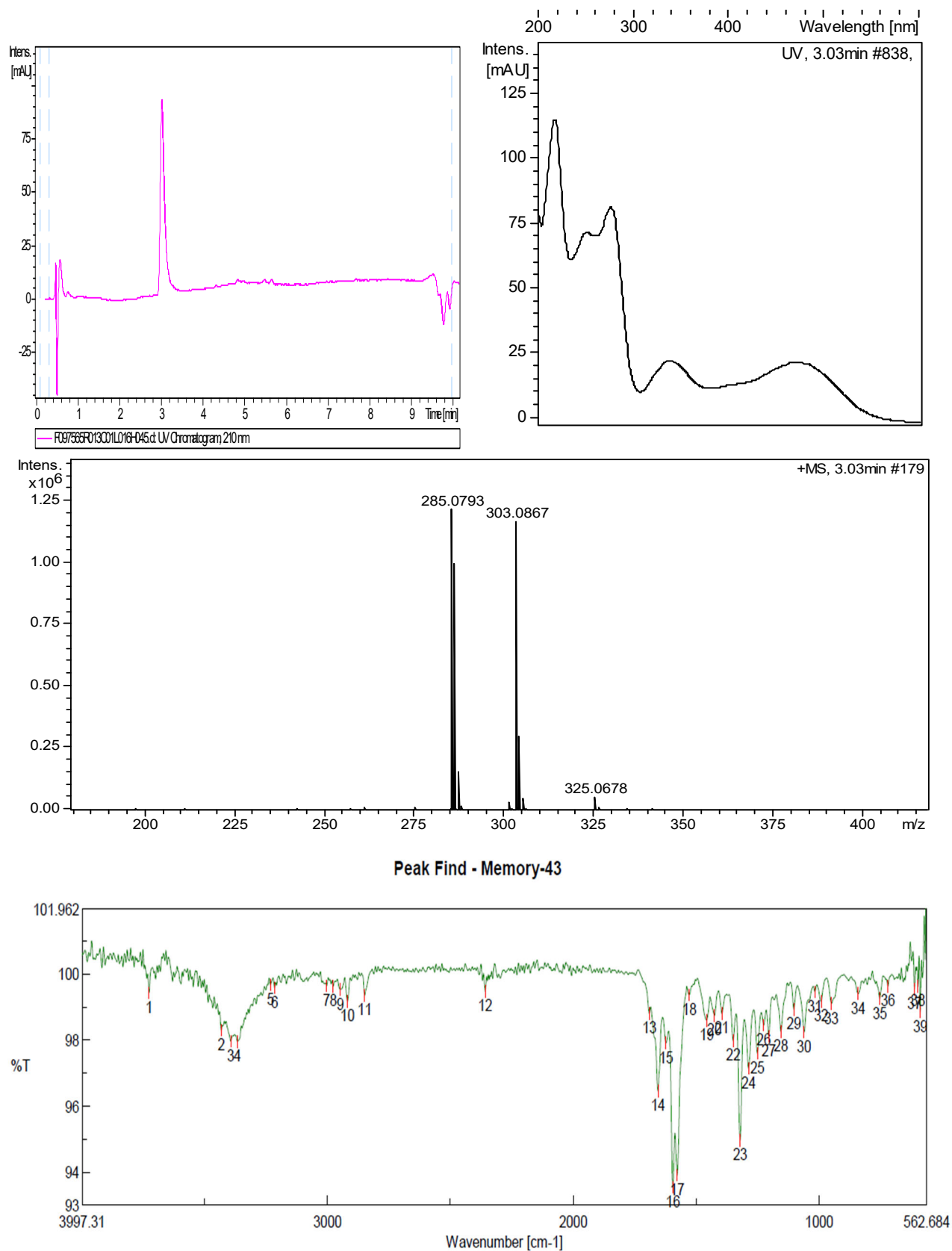


Figure S16. HPLC 210 nm trace, UV-Vis, ((+)-ESI-TOF) and IR spectra of compound **4** (purity 94% by UV).

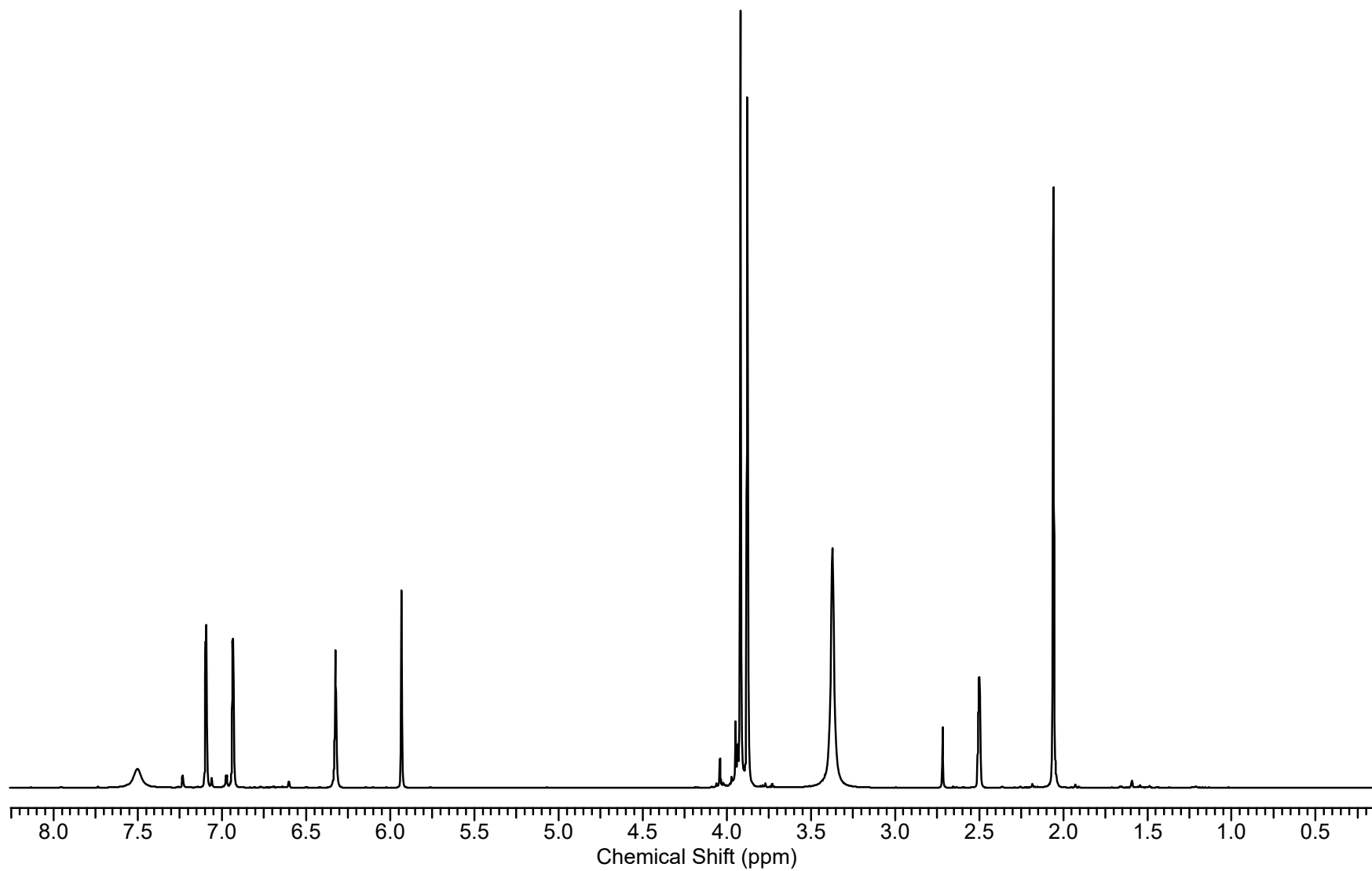


Figure S17. ^1H NMR spectrum ($\text{DMSO-}d_6$) of compound 4.

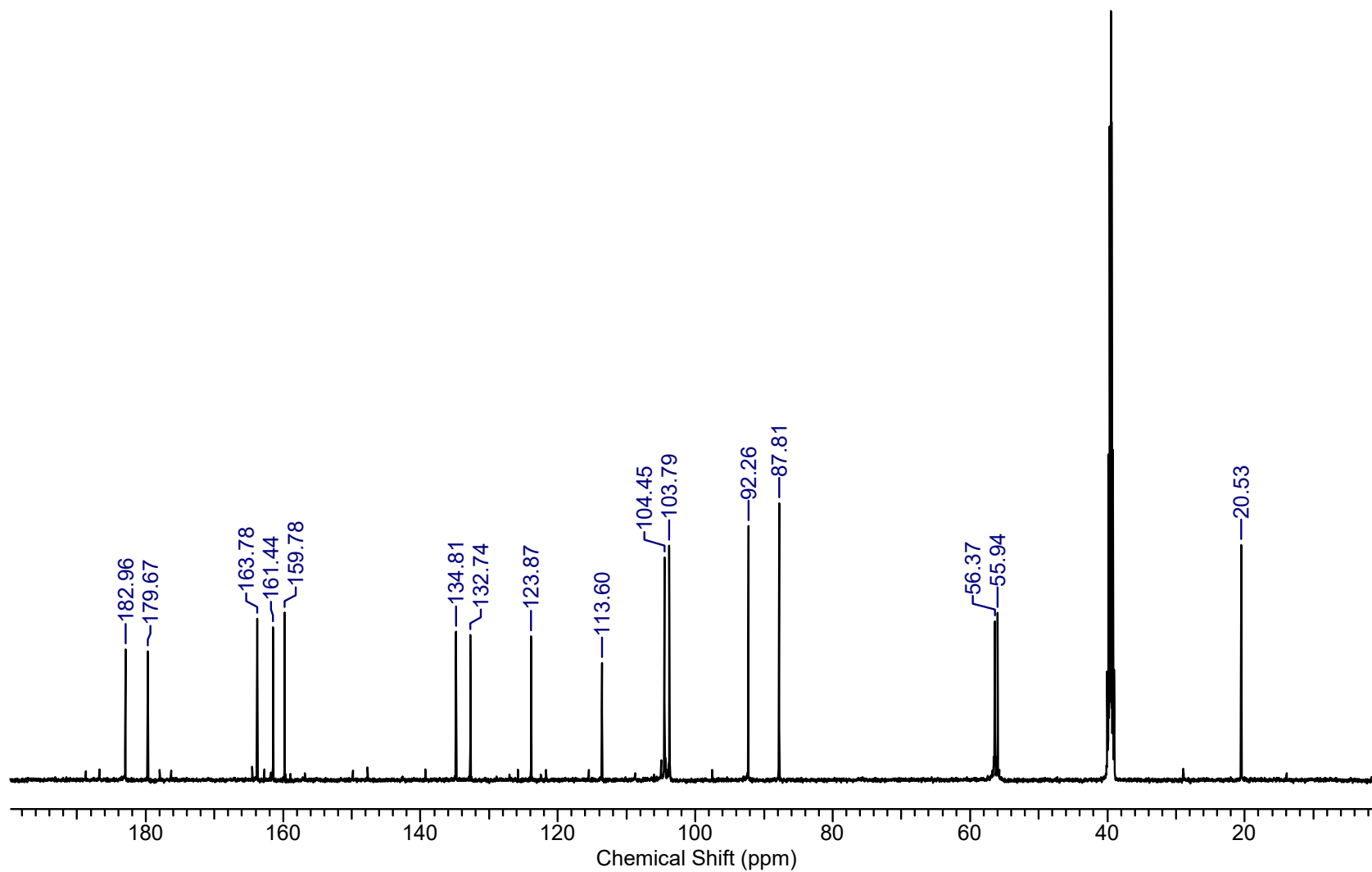


Figure S18. ¹³C NMR spectrum (DMSO-*d*₆) of compound 4.

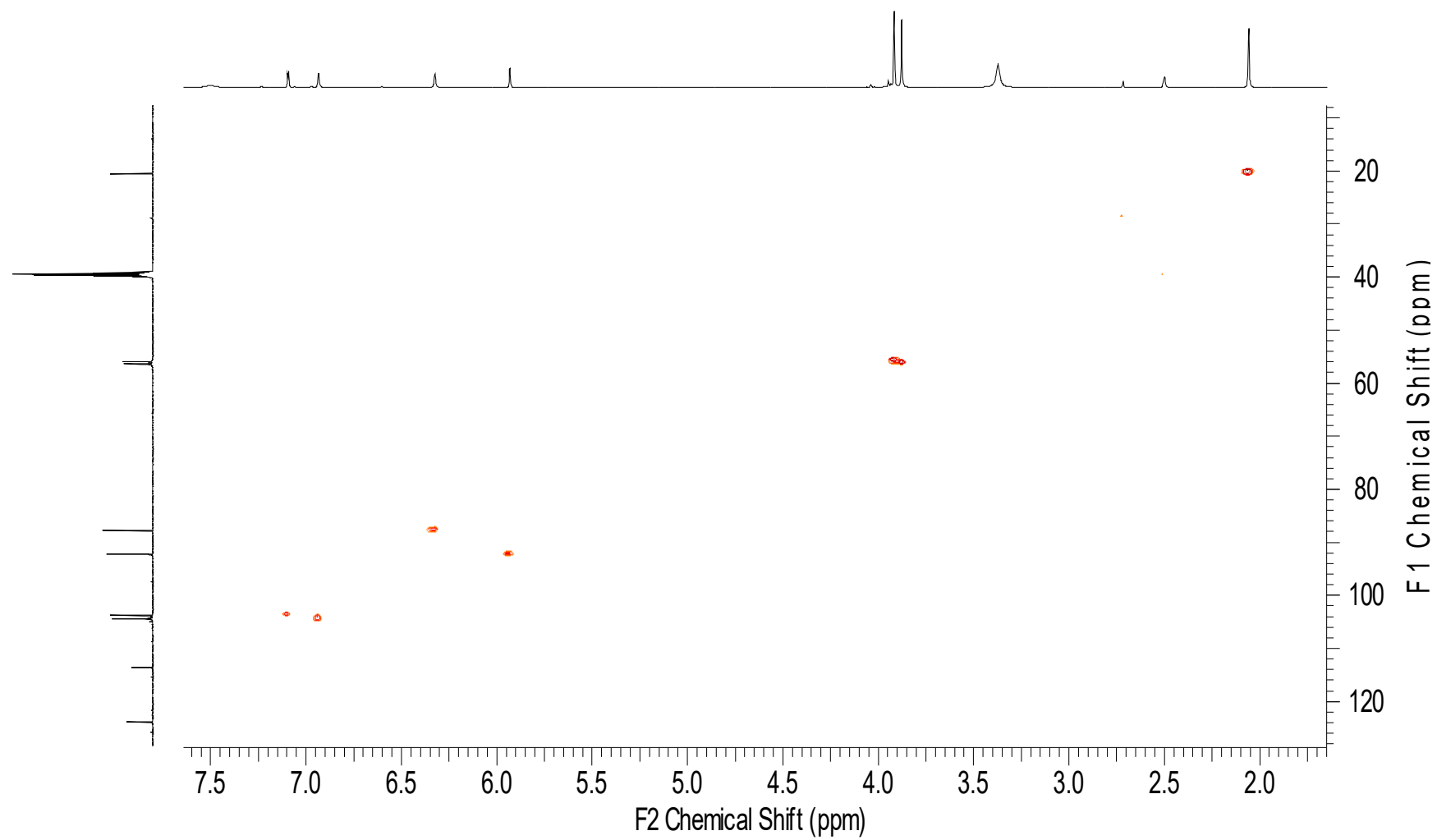


Figure S19. HSQC (DMSO- d_6) of compound 4.

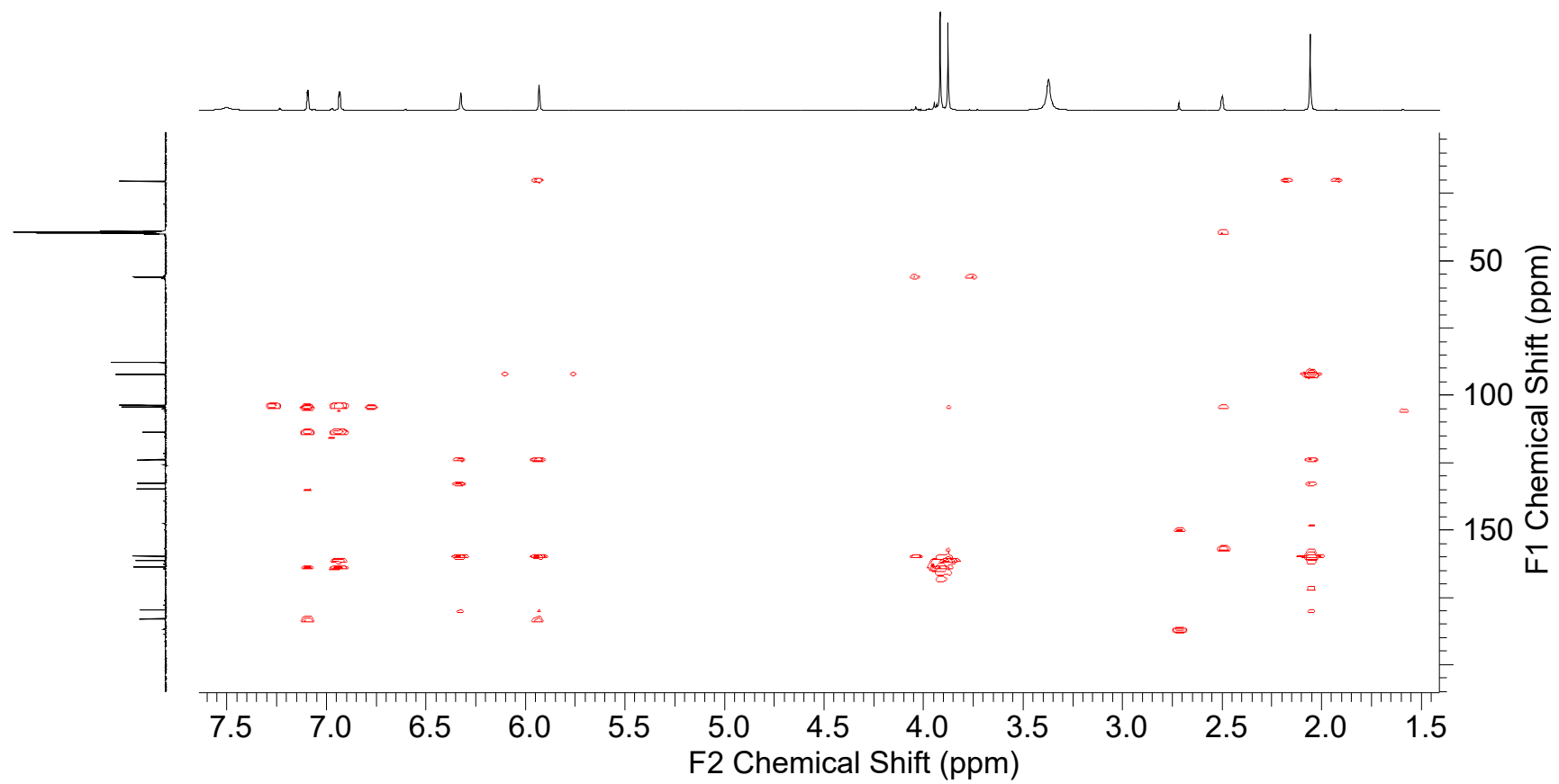


Figure S20. HMBC (DMSO- d_6) of compound 4.

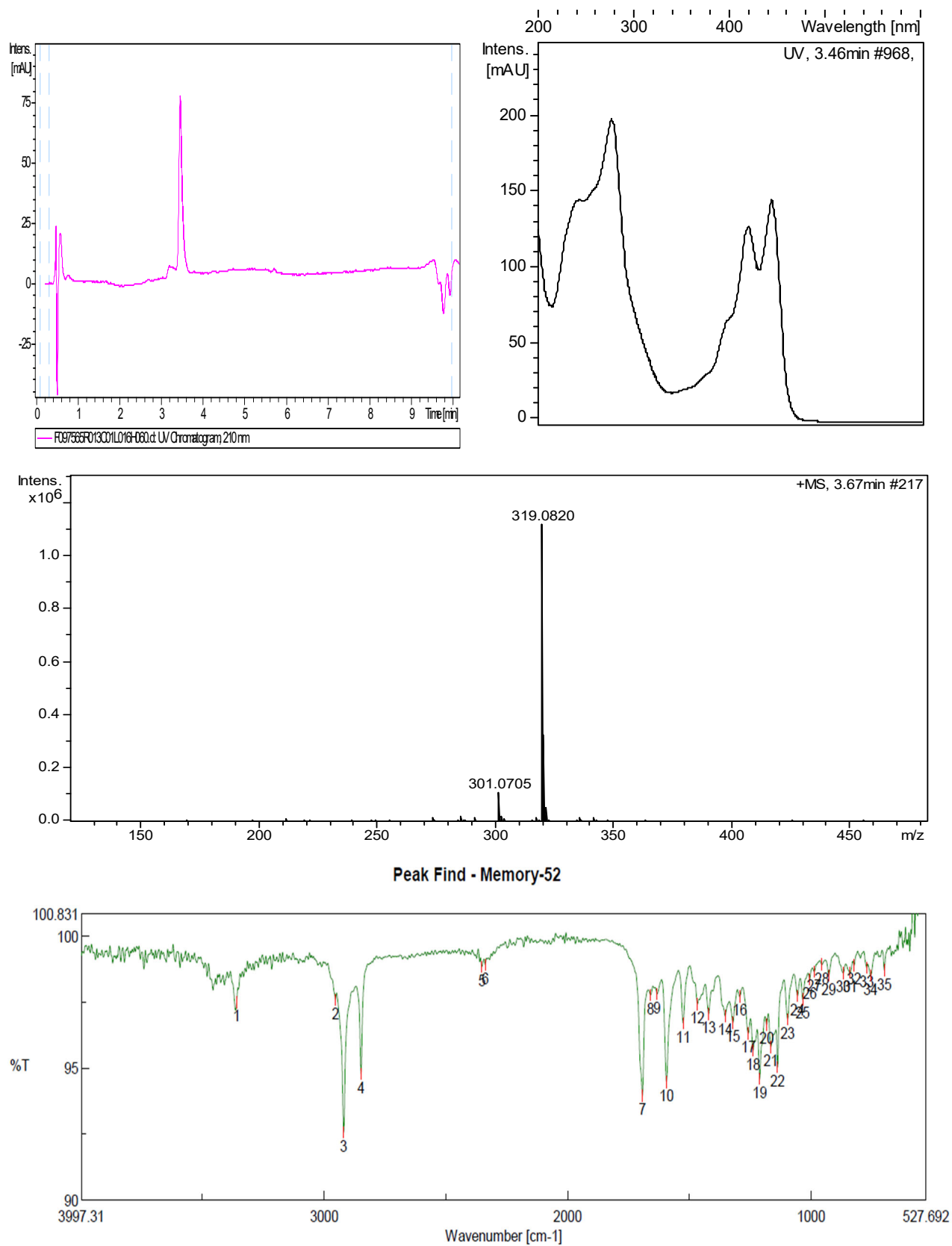


Figure S21. HPLC 210 nm trace, UV-Vis, ((+)-ESI-TOF) and IR spectra of compound 5 (purity 89% by UV).

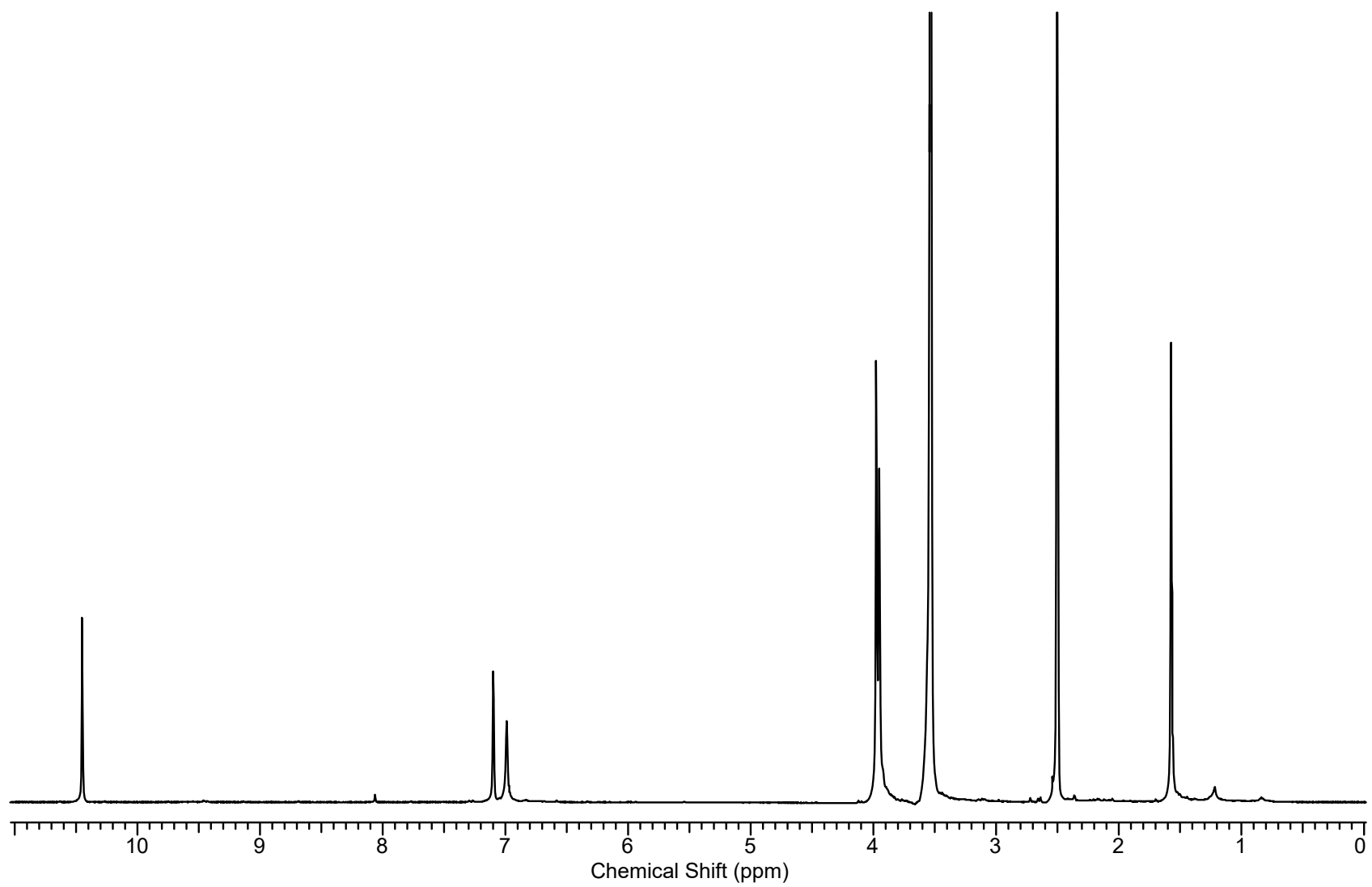


Figure S22. ^1H NMR spectrum ($\text{DMSO-}d_6$) of compound 5.

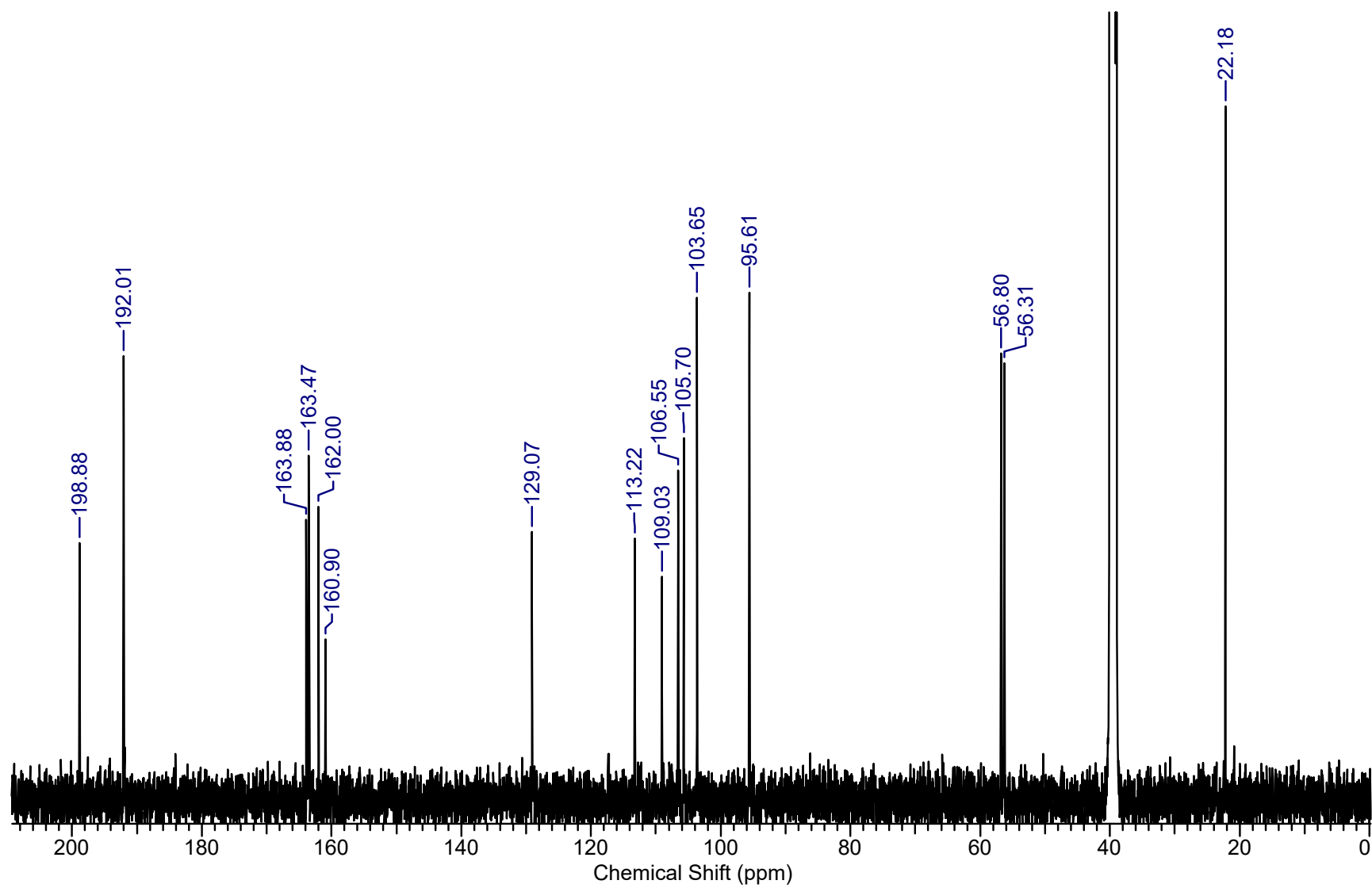


Figure S23. ¹³C NMR spectrum (DMSO-*d*₆) of compound 5.

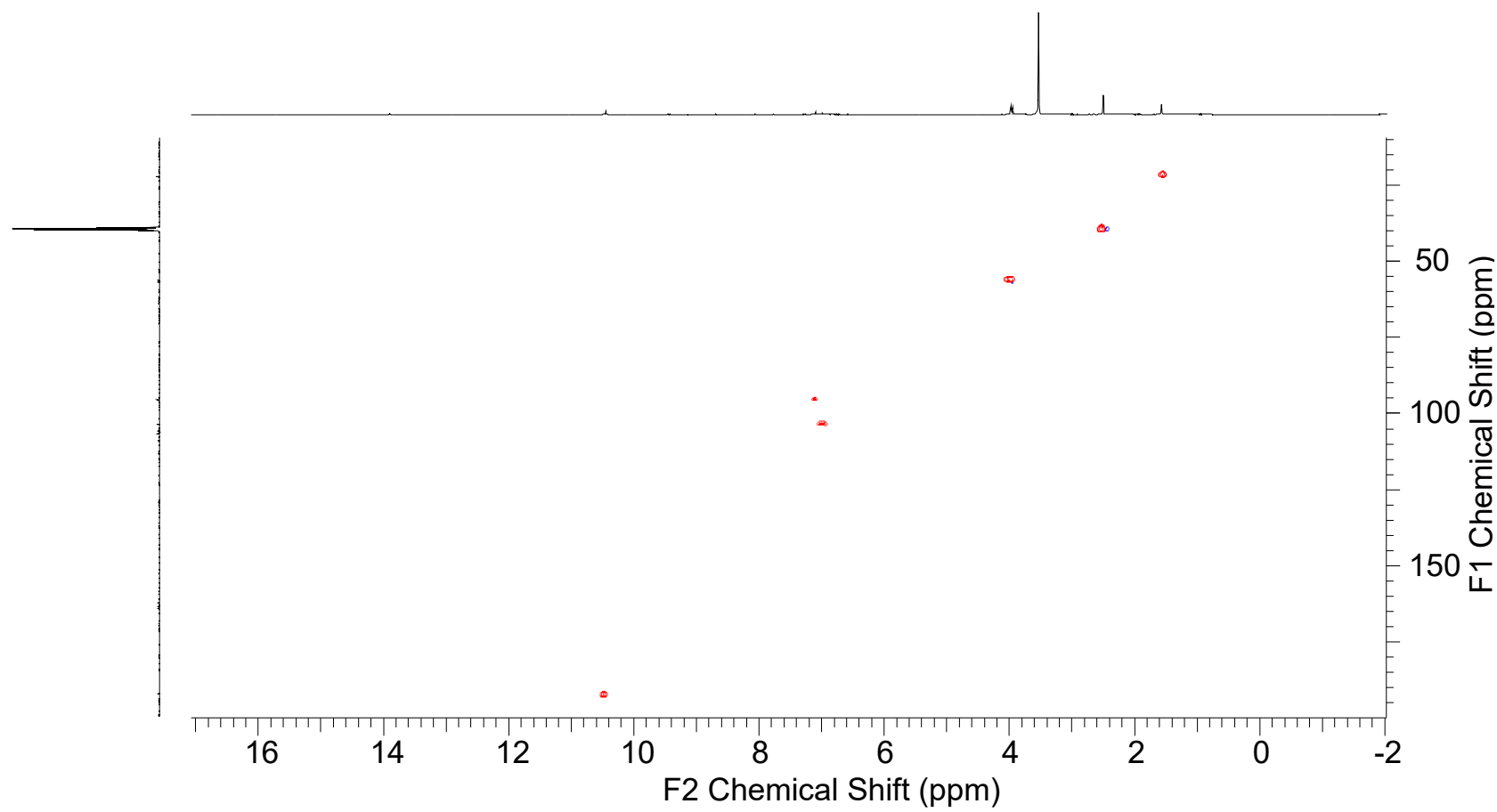


Figure S24. HSQC (DMSO-*d*₆) of compound 5.

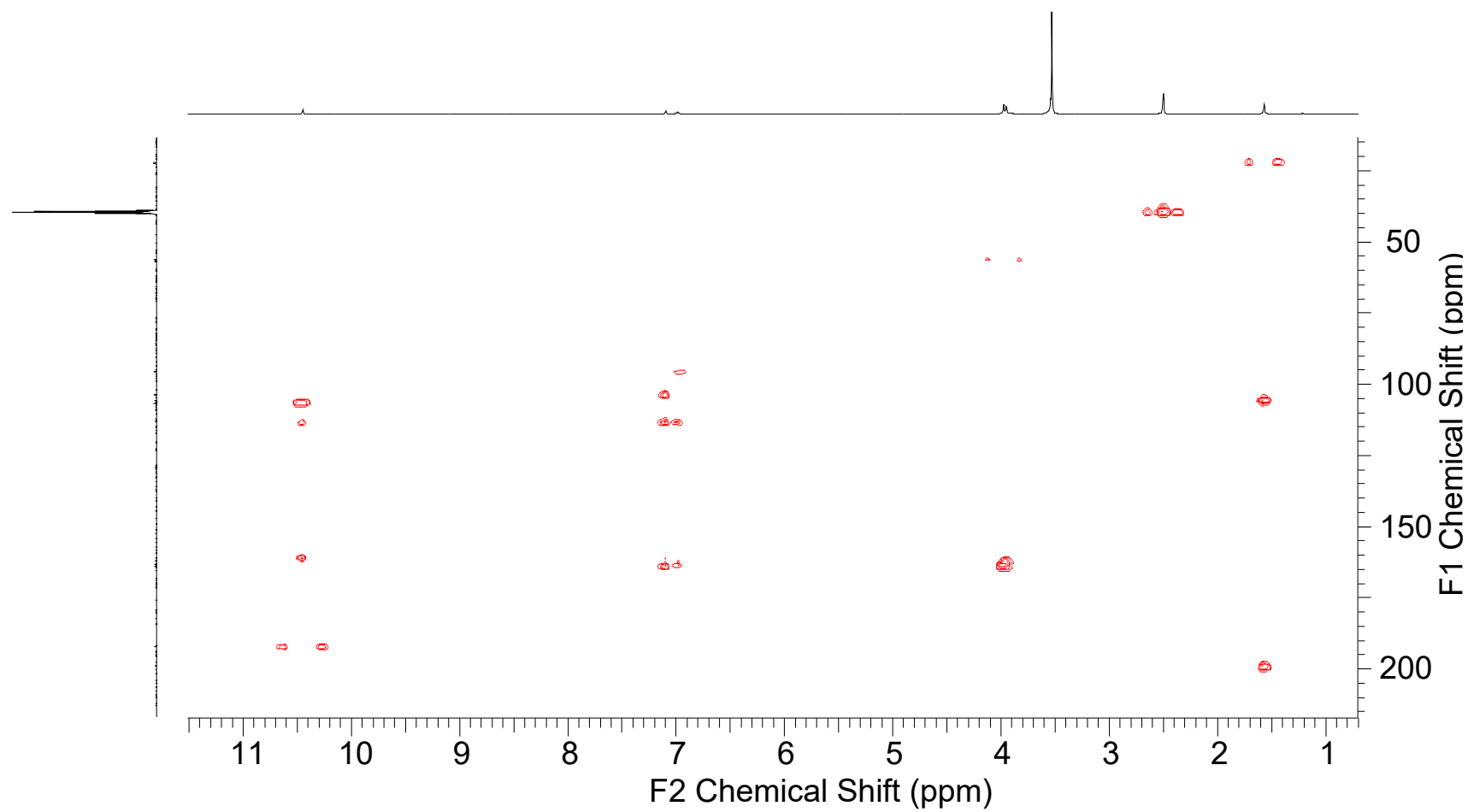


Figure S25. HMBC (DMSO- d_6) of compound 5.