Supplementary Information

Figure S1. ^{1}H NMR spectrum of hyaluromycin (**1**) (500 MHz, DMSO-d_6).

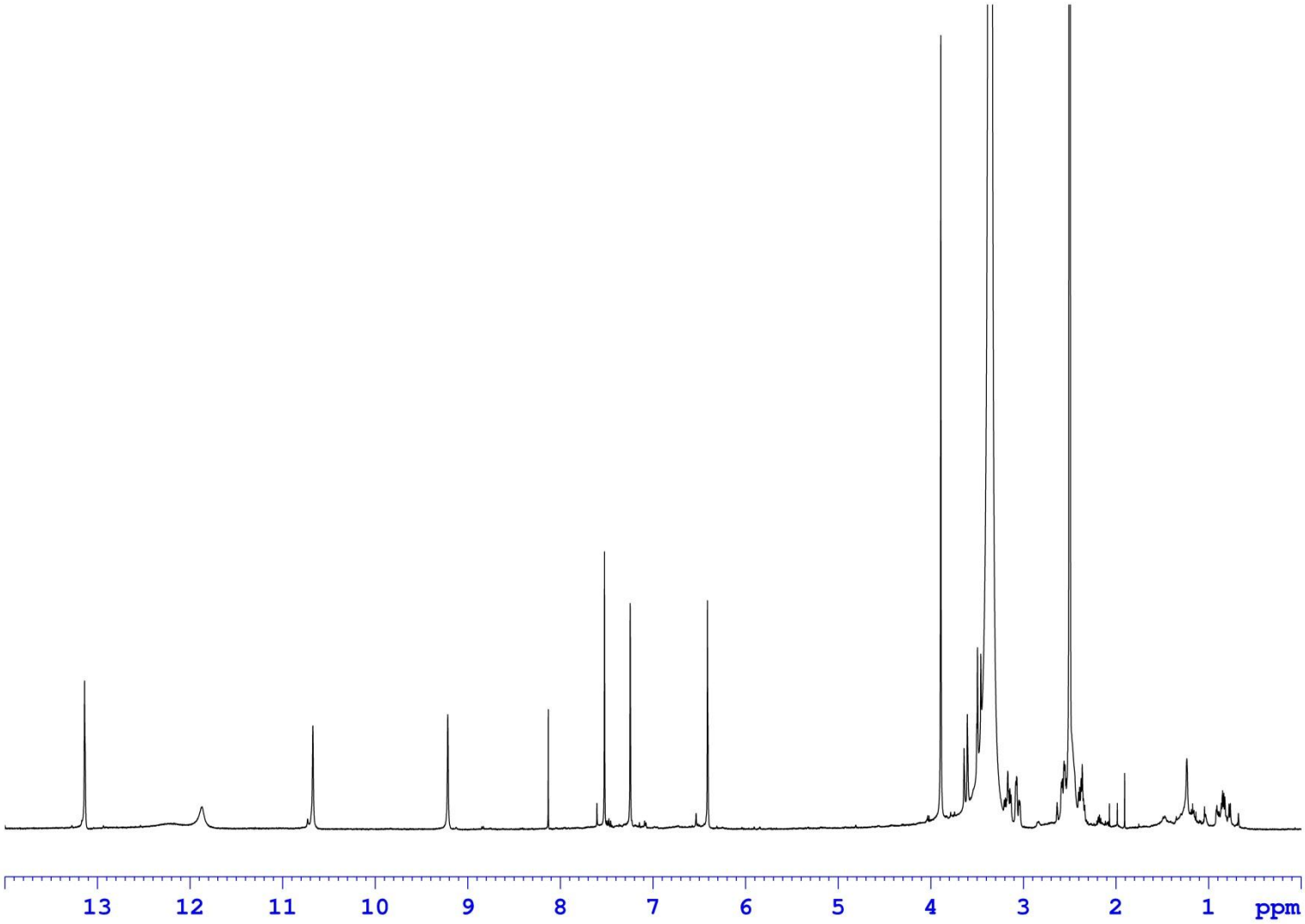

Figure S2. ^{13}C NMR spectrum of **1** (125 MHz, DMSO-d_6).

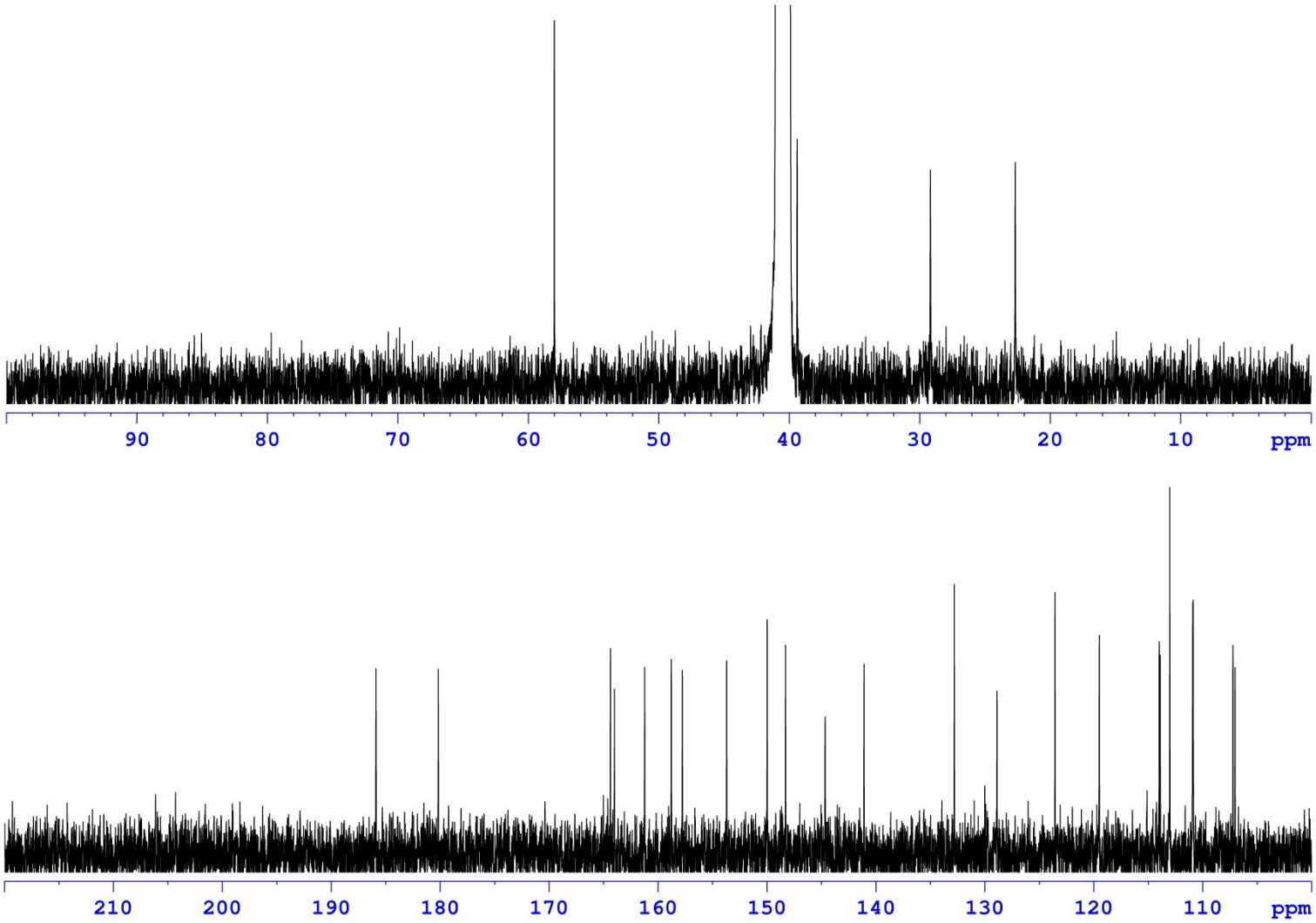

Figure S3. ^{1}H-^{1}H COSY spectrum of **1** (500 MHz, DMSO-d_6).

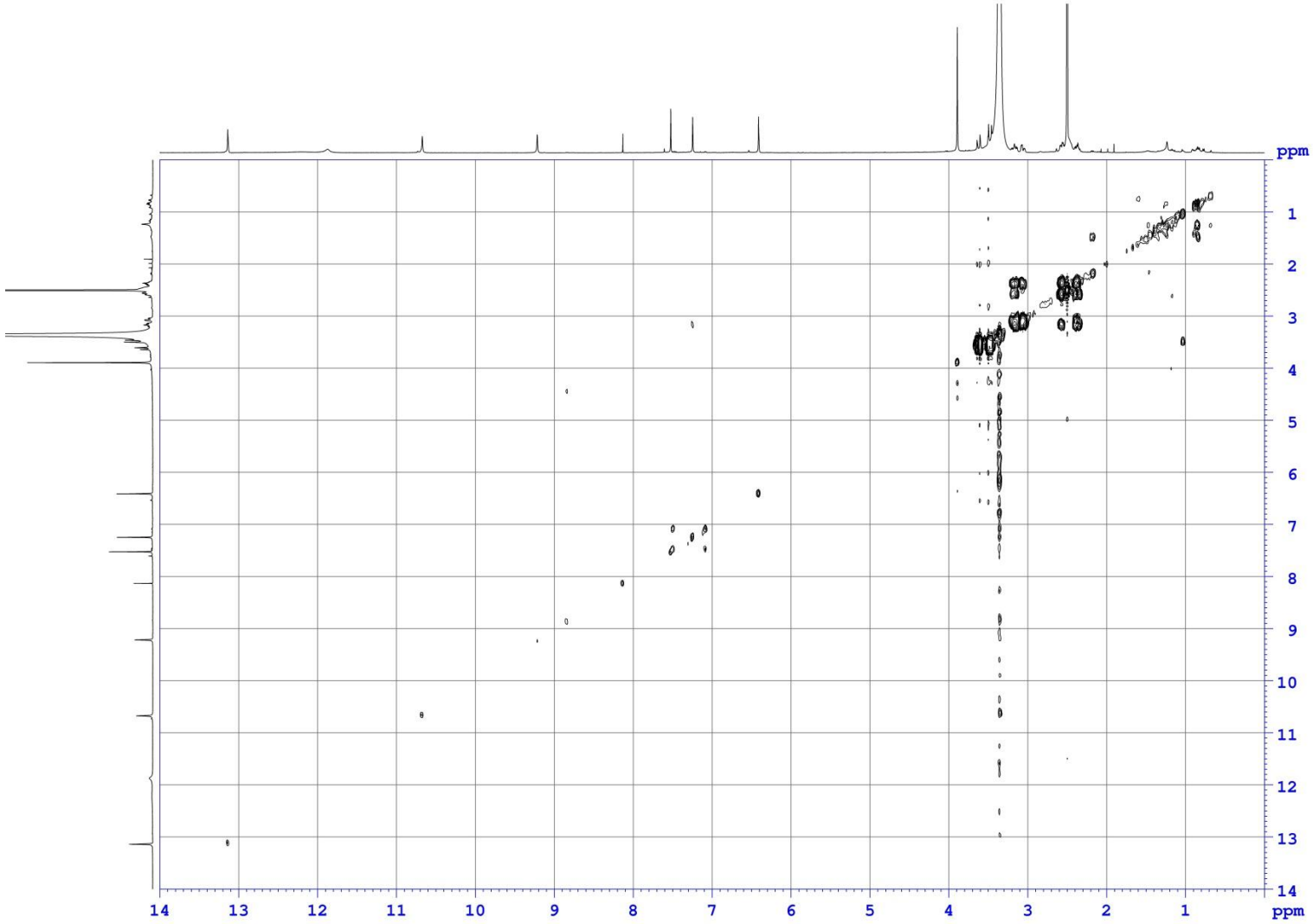

Figure S4. HSQC spectrum of **1** (500 MHz, DMSO-d_6).

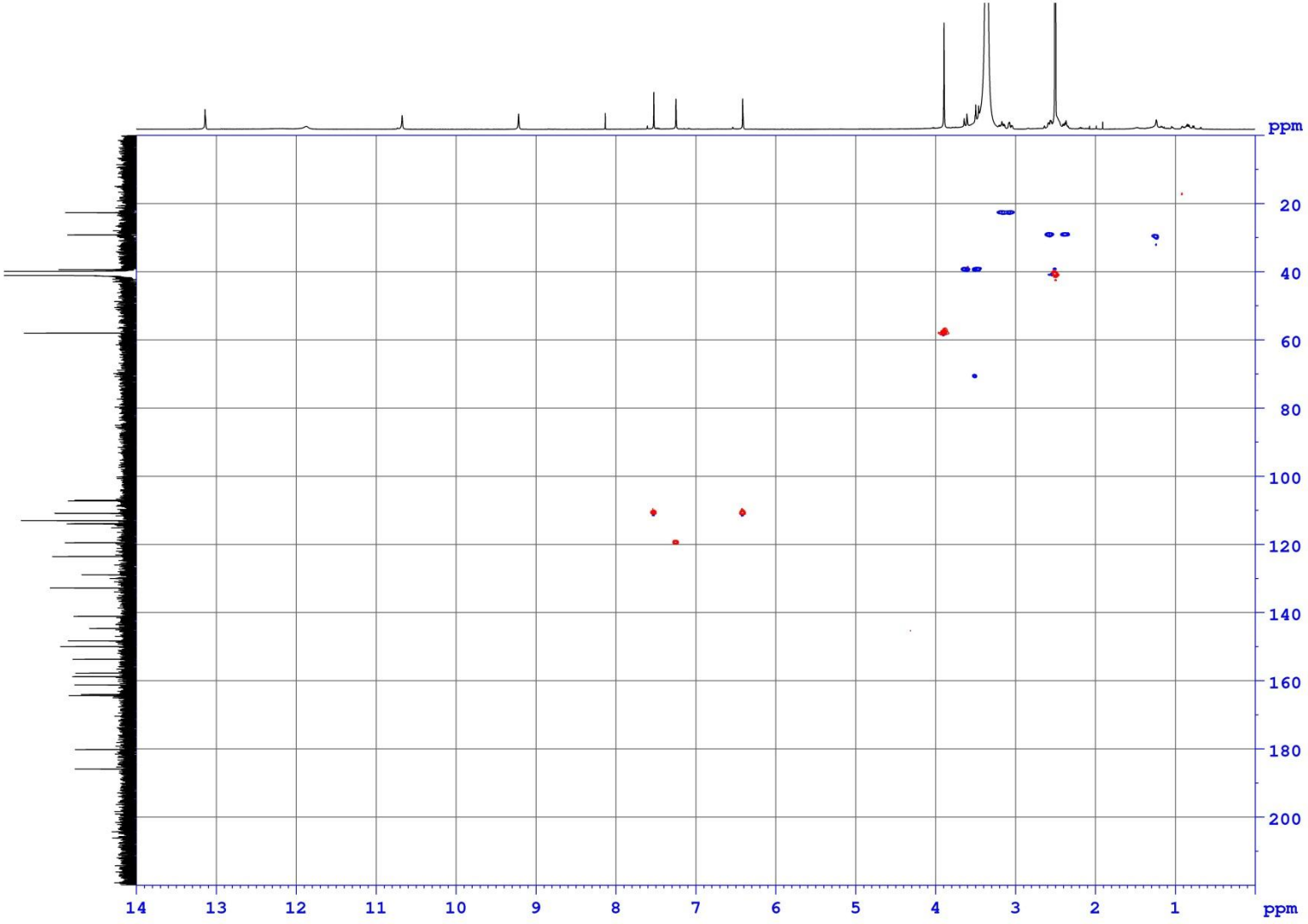

Figure S5. HMBC spectrum of **1** (500 MHz, DMSO-d_6).

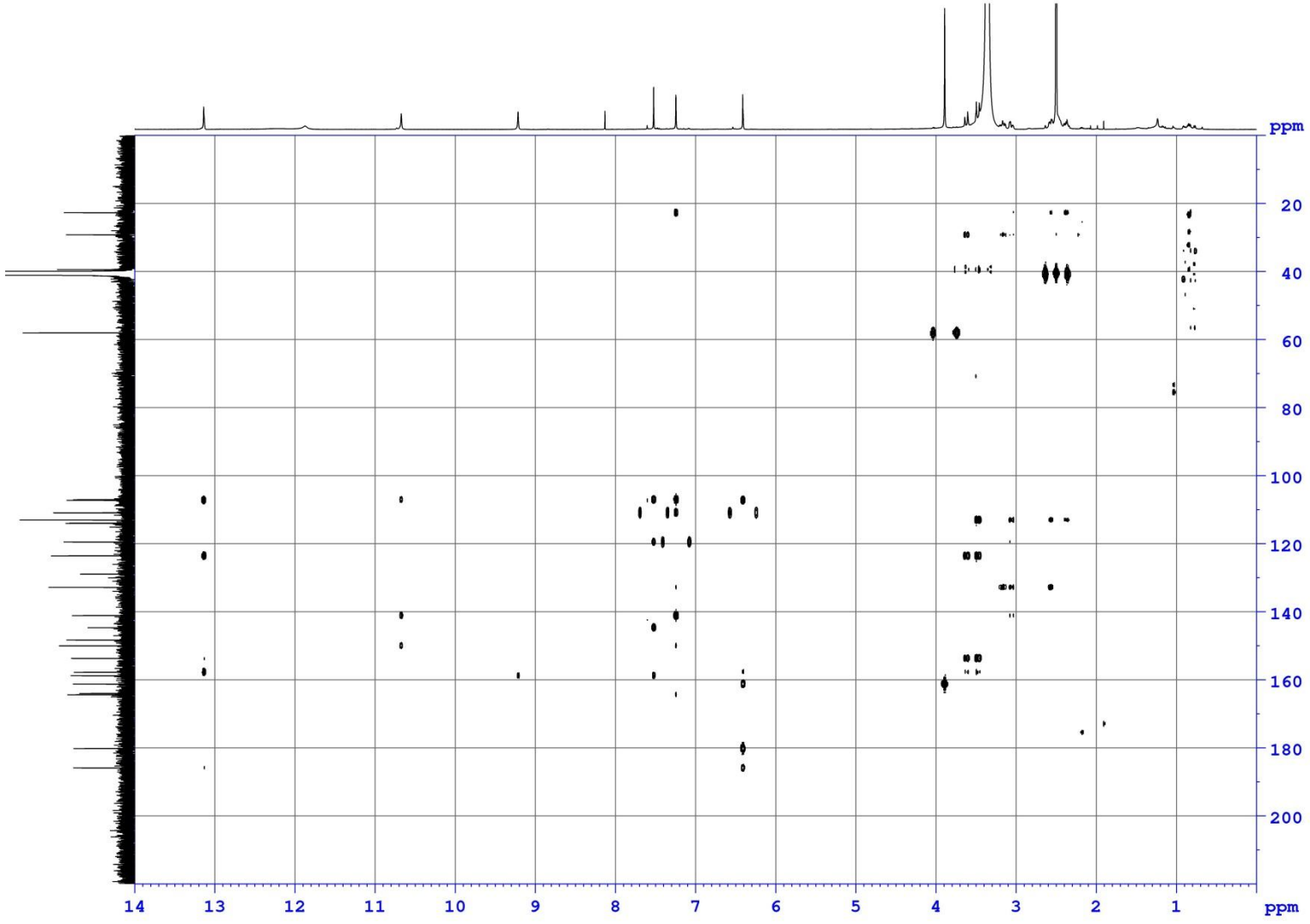

Figure S6. ^{1}H NMR of 3″-*O*-methyl hyaluromycin (**2**) (500 MHz, DMSO-d_6).

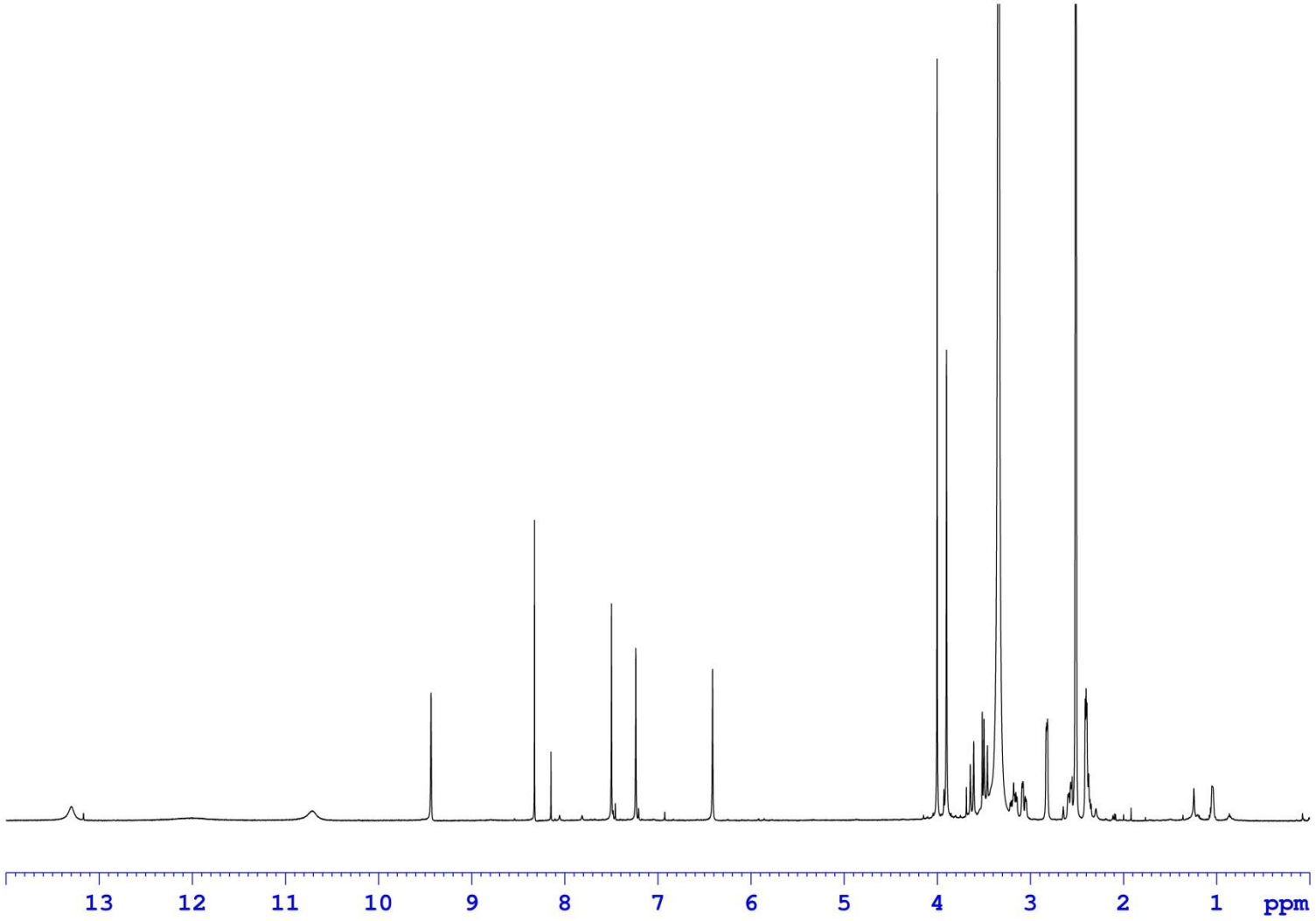

Figure S7. ^{13}C NMR spectrum of **2** (125 MHz, DMSO-d_6).

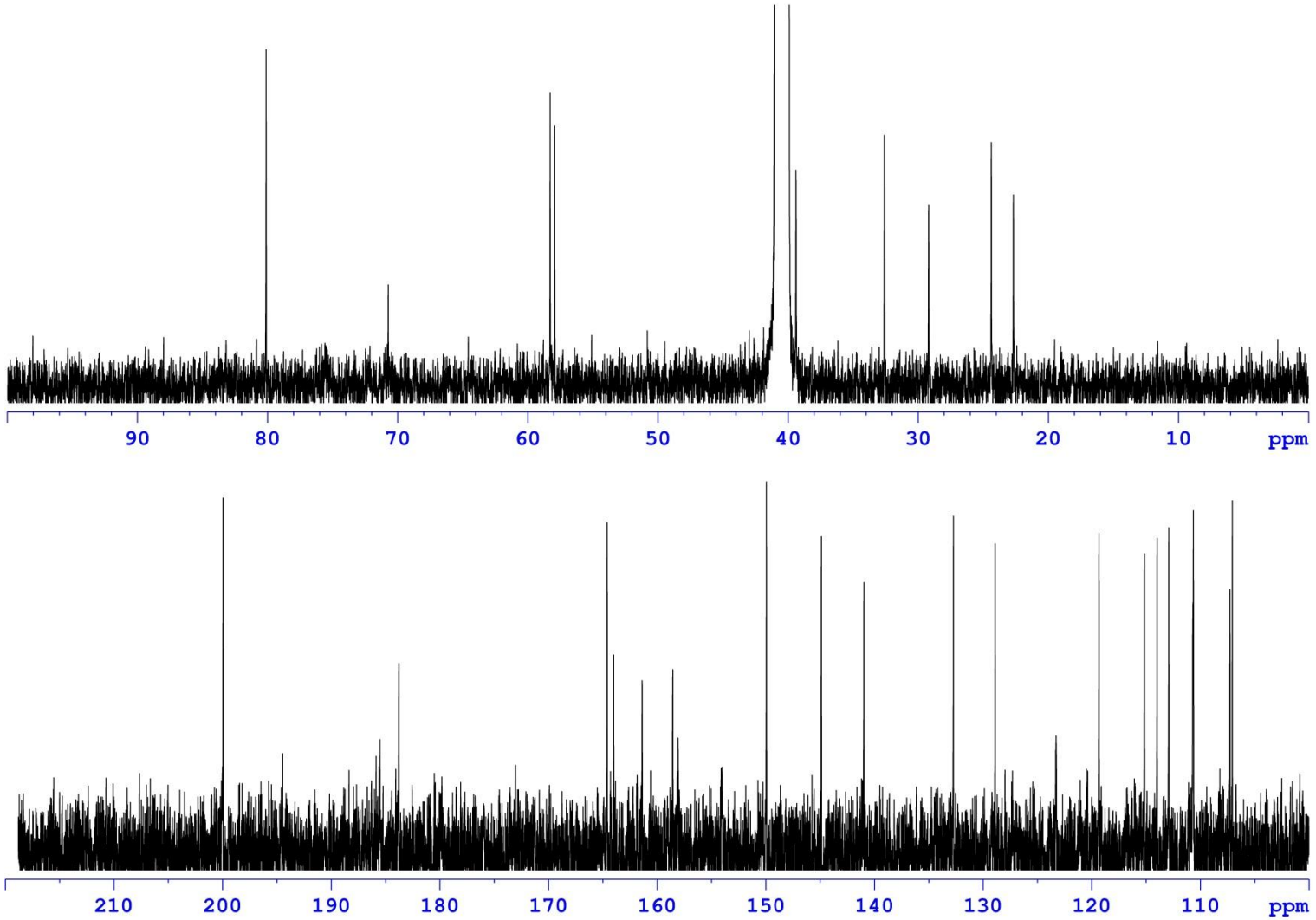

Figure S8. HSQC spectrum of **2** (500 MHz, DMSO-d_6).

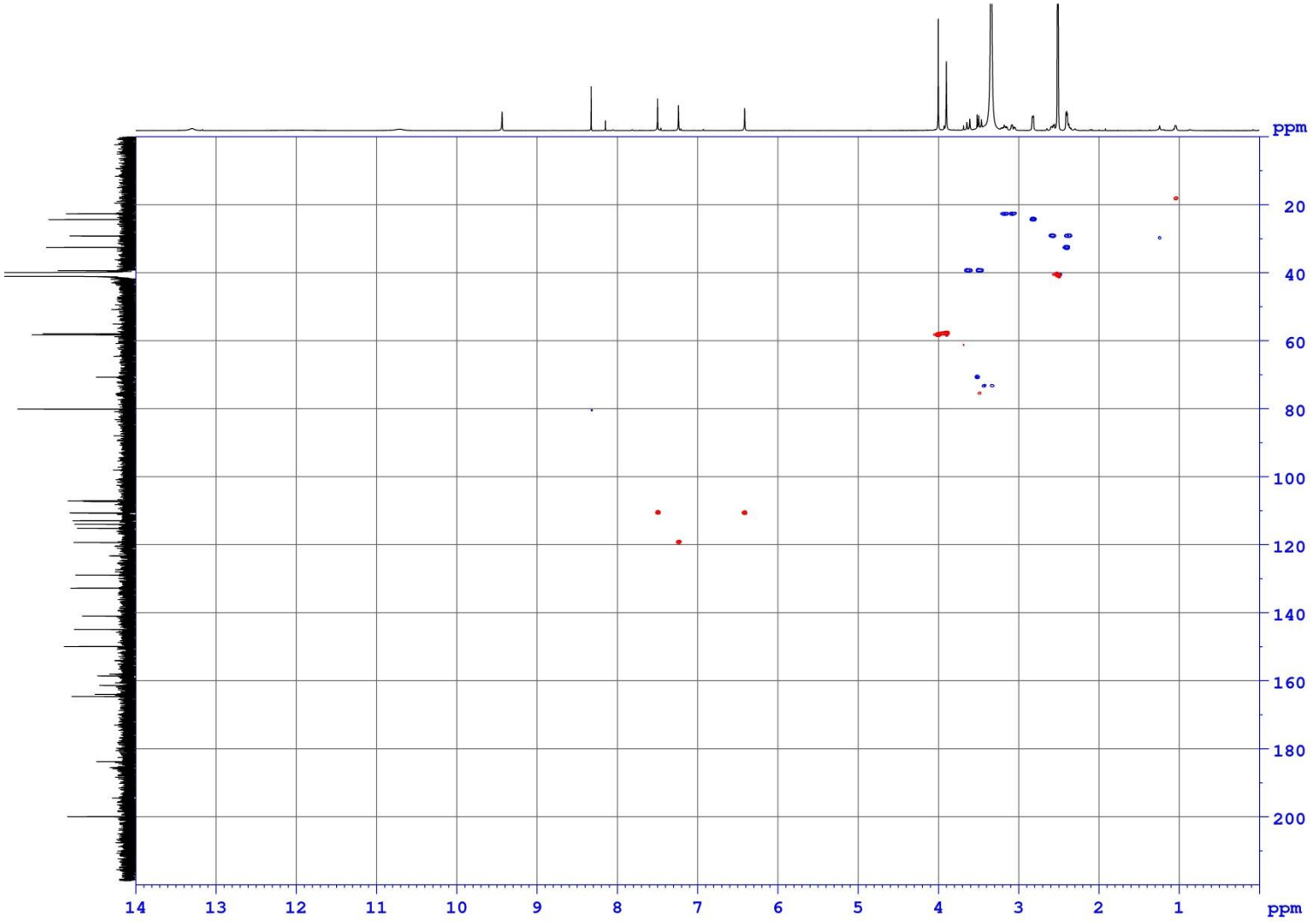

Figure S9. HMBC spectrum of **2** (500 MHz, DMSO-d_6).

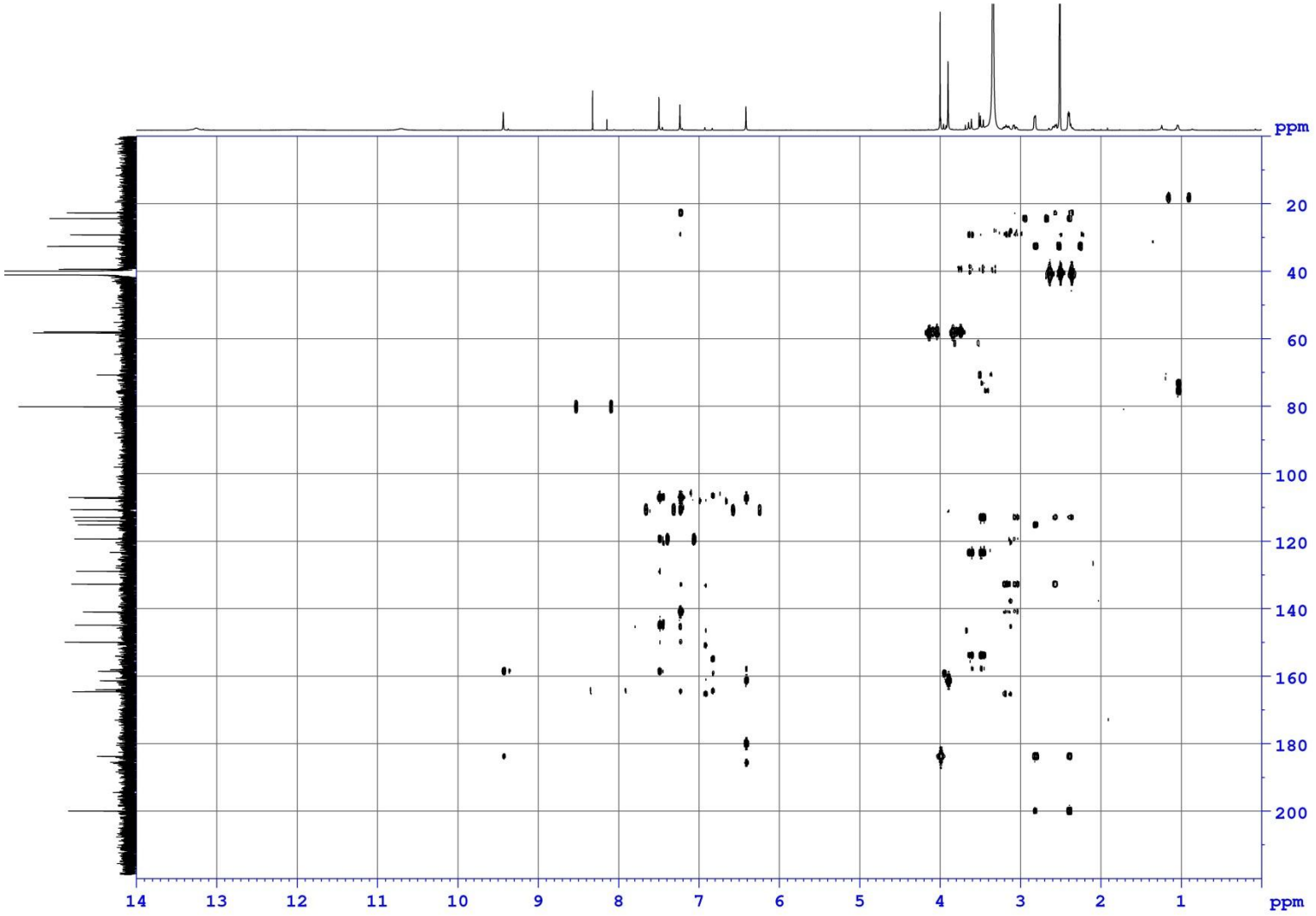

Figure S10. ^{13}C NMR spectrum of [1,2-$^{13}C_2$]acetate-labeled **1** (500 MHz, DMSO-d_6).

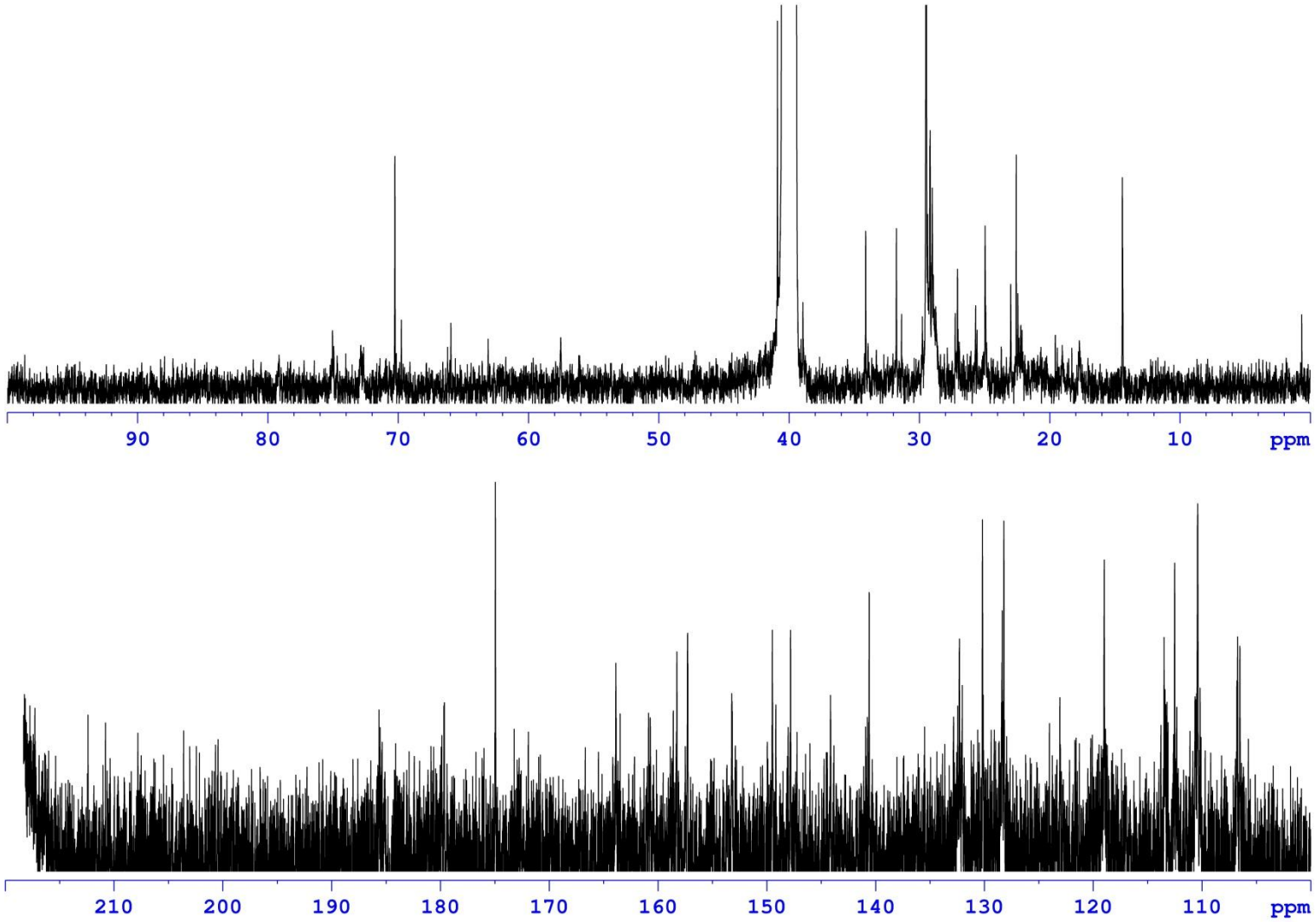

Figure S11. ^{1}H NMR of [1,2-$^{13}C_2$]acetate-labeled **2** (500 MHz, DMSO-d_6).

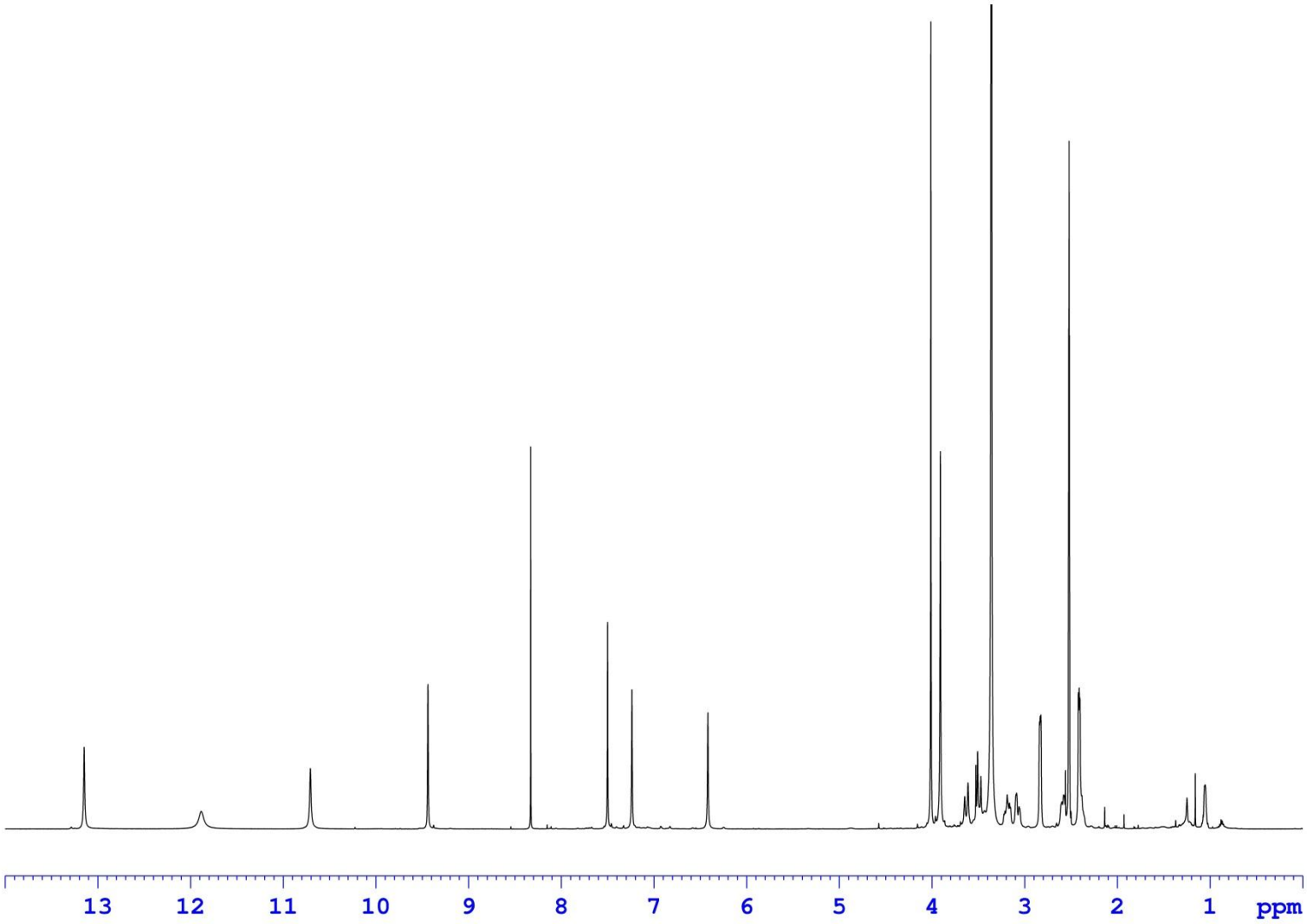

Figure S12. ^{13}C NMR spectrum of [1,2-$^{13}C_2$]acetate-labeled **2** (125 MHz, DMSO-d_6).

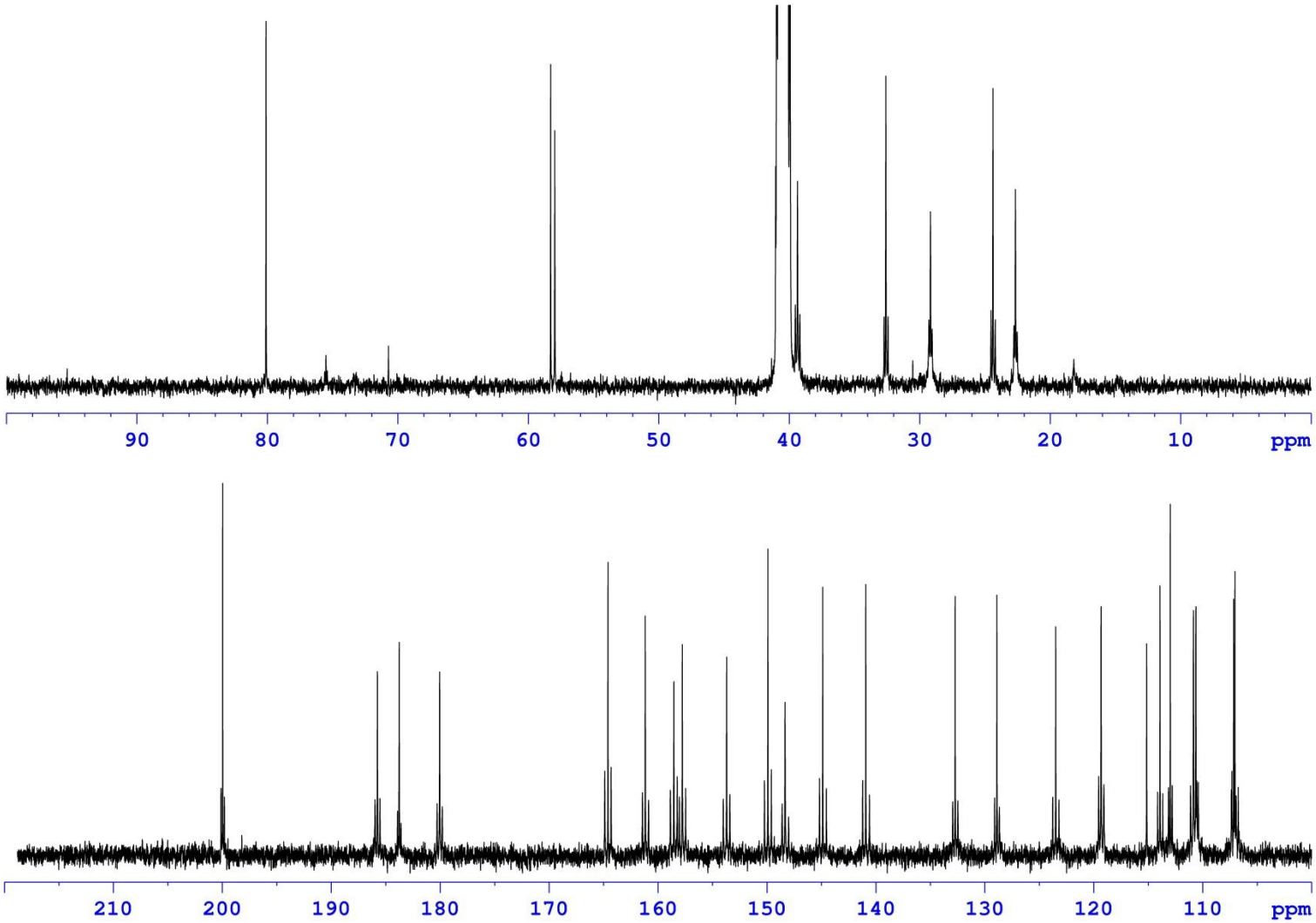

Figure S13. ^{1}H-^{1}H COSY spectrum of [1,2-$^{13}C_2$]acetate-labeled **2** (500 MHz, DMSO-d_6).

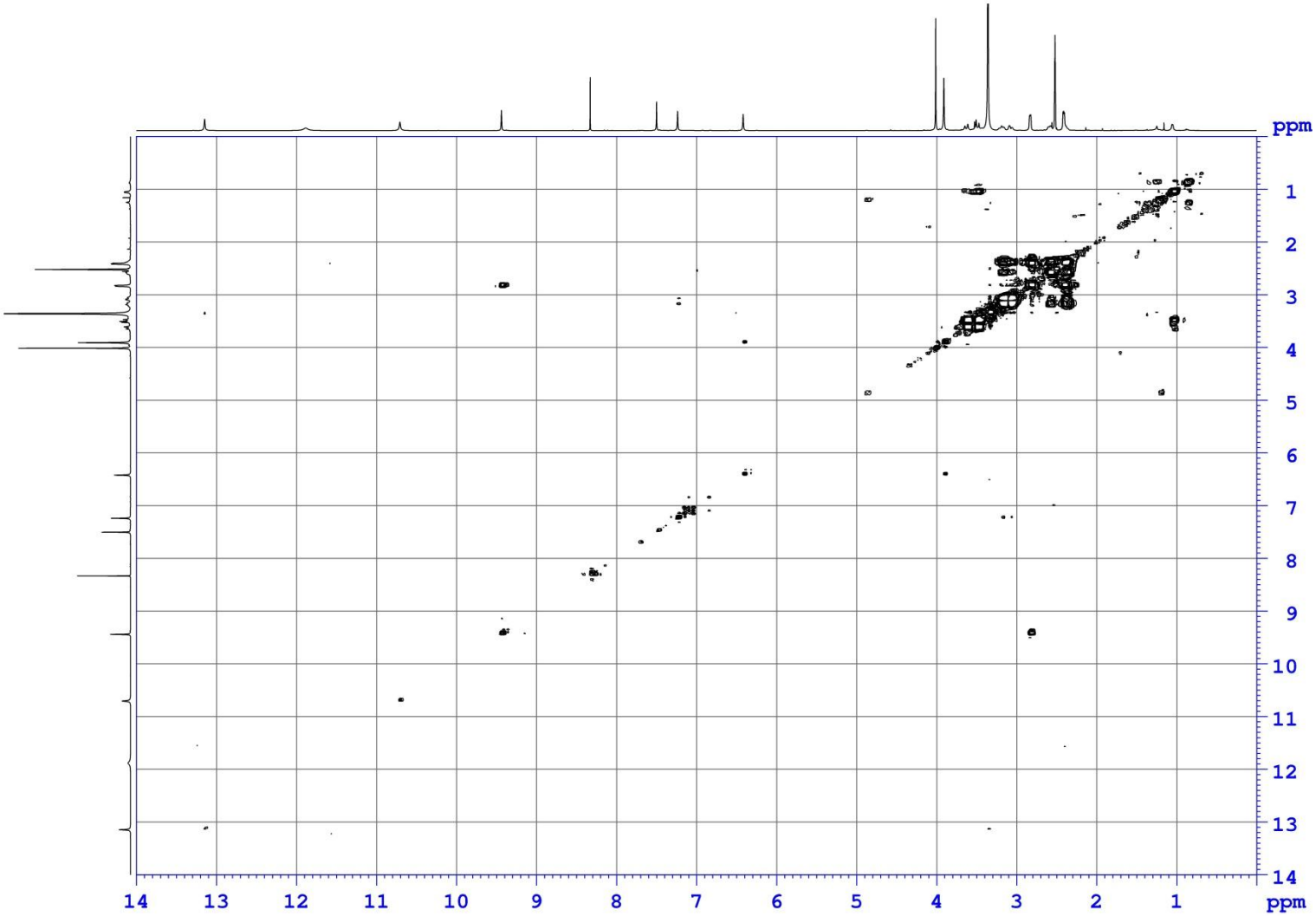

Figure S14. HSQC spectrum of [1,2-$^{13}C_2$]acetate-labeled **2** (500 MHz, DMSO-d_6).

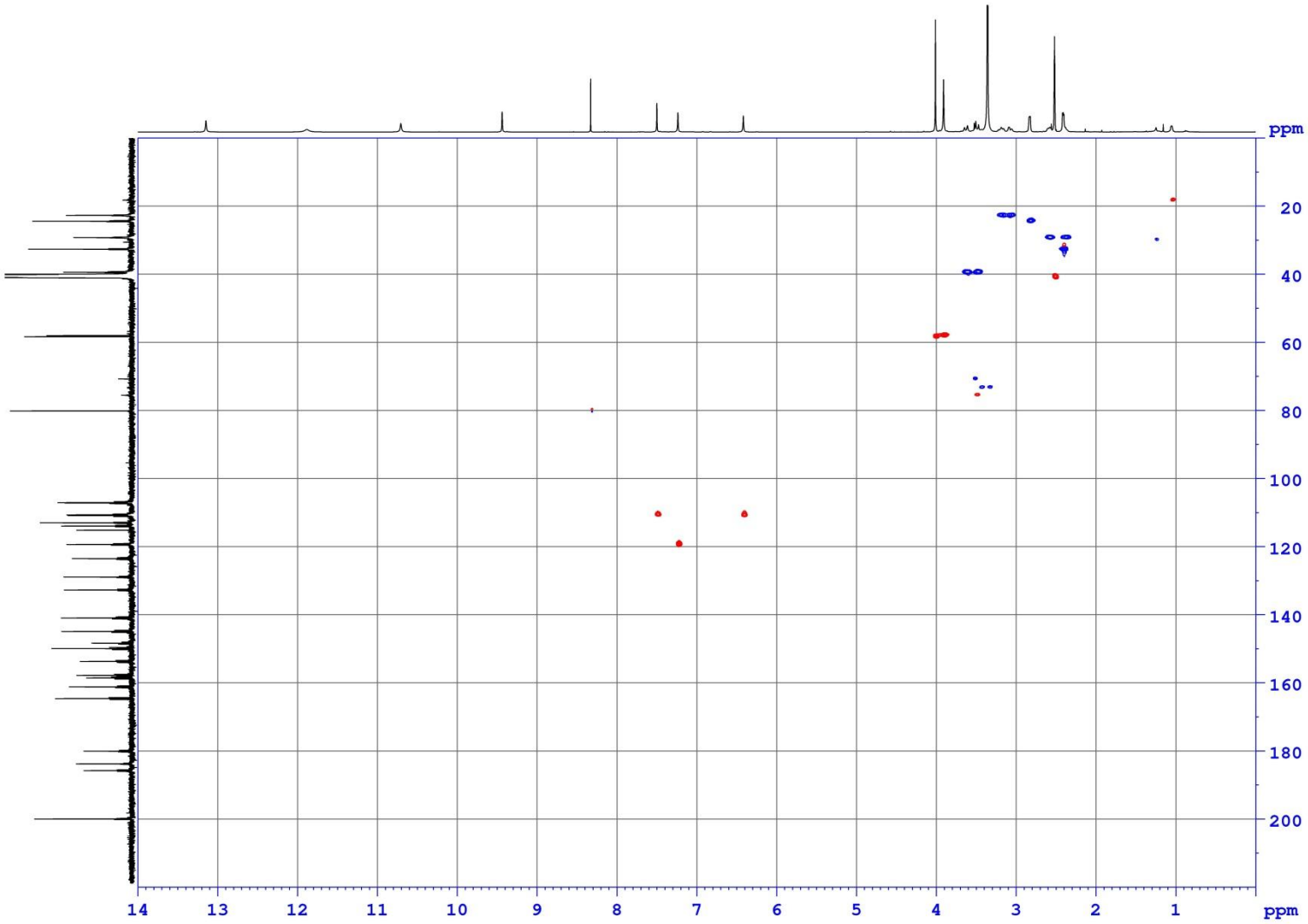

Figure S15. HMBC spectrum of [1,2-$^{13}C_2$]acetate-labeled **2** (500 MHz, DMSO-d_6).

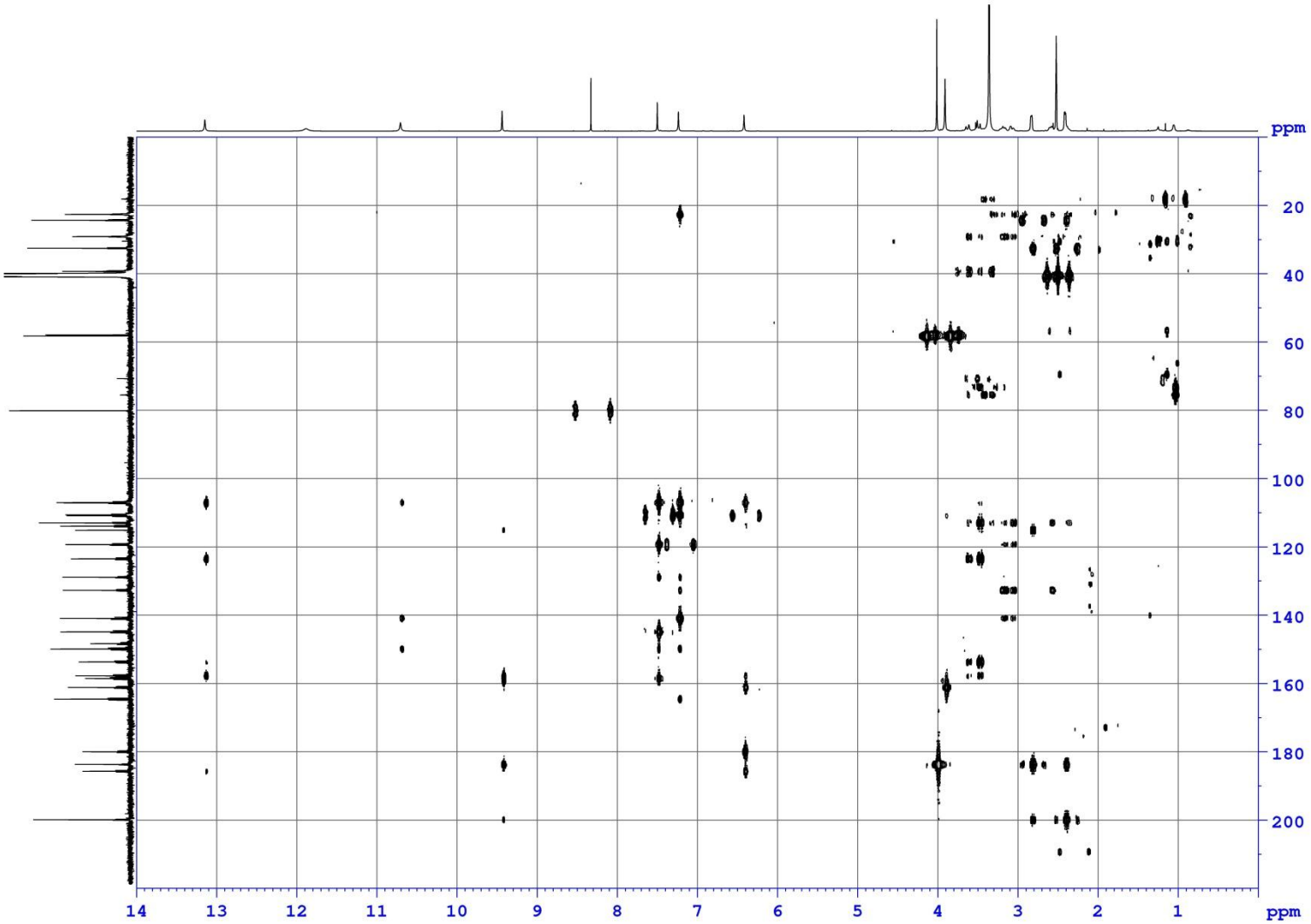

Figure S16. INADEQUATE spectrum of [1,2-$^{13}C_2$]acetate-labeled **2** (125 MHz, DMSO-d_6, $^1J_{C\text{-}C}$ = 50 Hz).

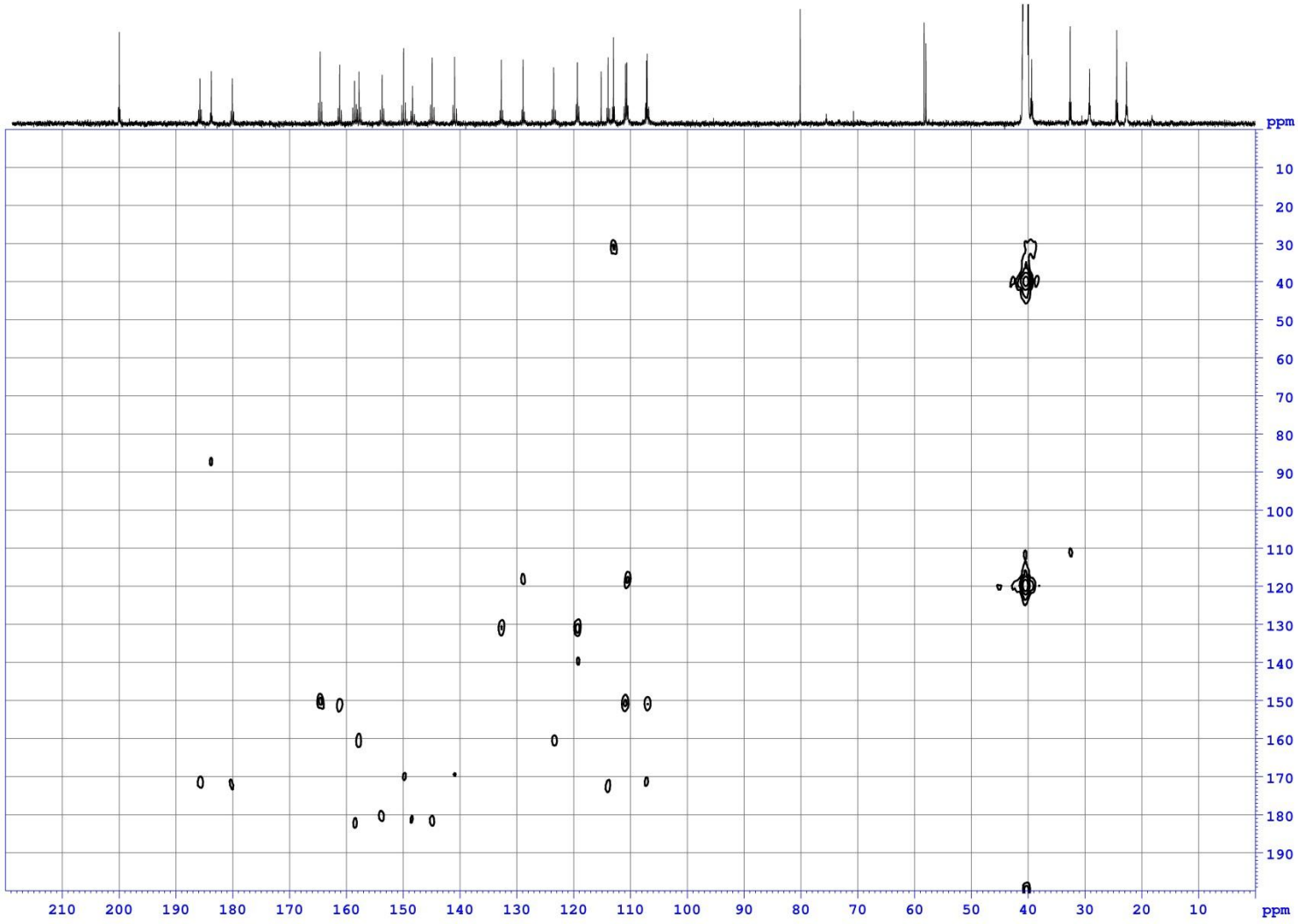

Figure S17. INADEQUATE spectrum of [1,2-$^{13}C_2$]acetate-labeled **2** (125 MHz, DMSO-d_6, $^1J_{C\text{-}C}$ = 35 Hz).