

Electronic Supplementary Information (ESI)

Reactions of triosmium and triruthenium clusters with 2-ethynylpyridine: new modes for alkyne C–C bond coupling and C–H bond activation

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Figures S1 to S16 contain IR and ¹H NMR spectra of the new compounds (2, 3, 5 and 6).

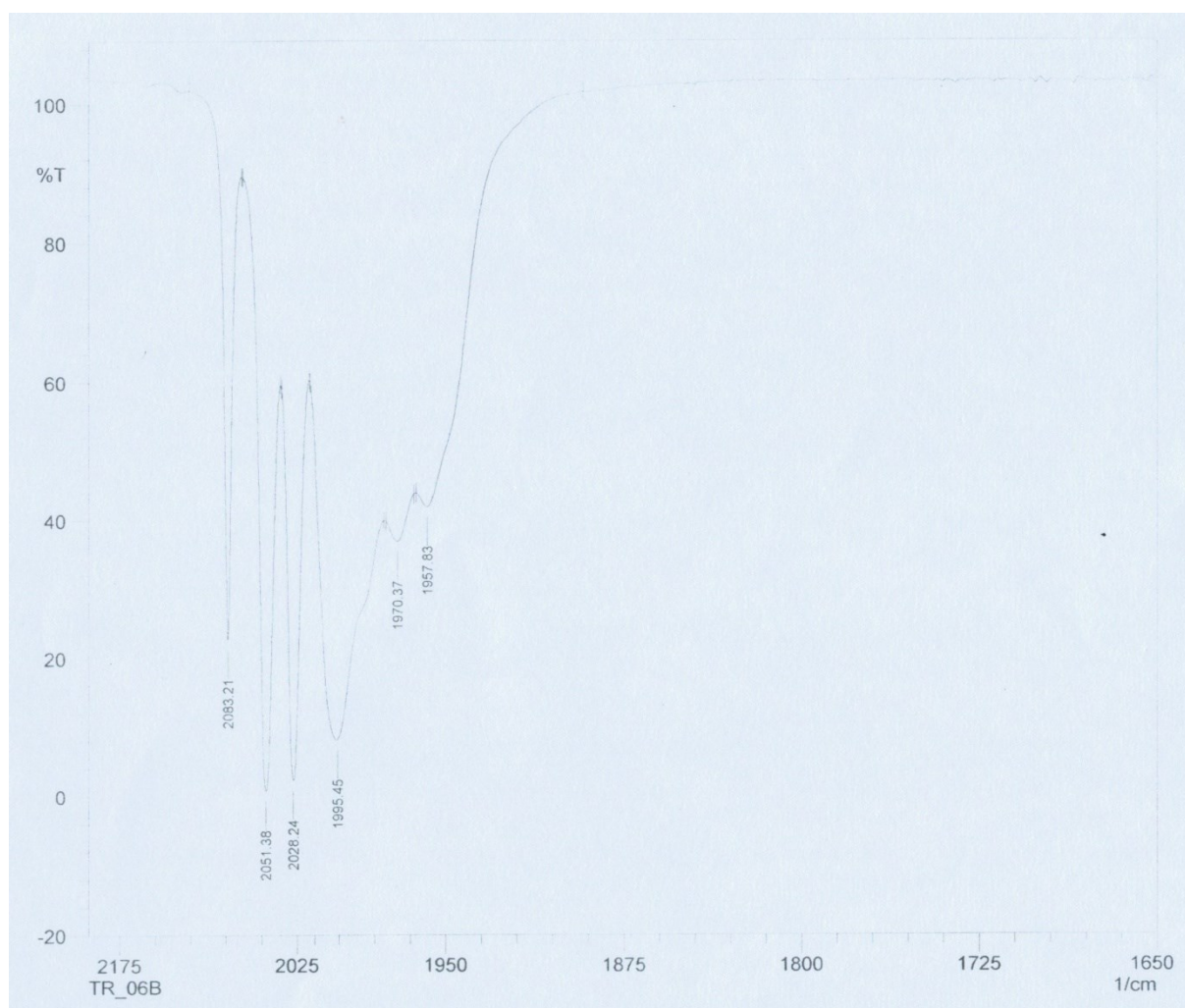


Figure S1. IR spectrum of $[\text{HOs}_3(\text{CO})_9(\mu_3\text{-C}_5\text{H}_4\text{NC}=\text{CH}_2)]$ (**2**) in CH_2Cl_2 .

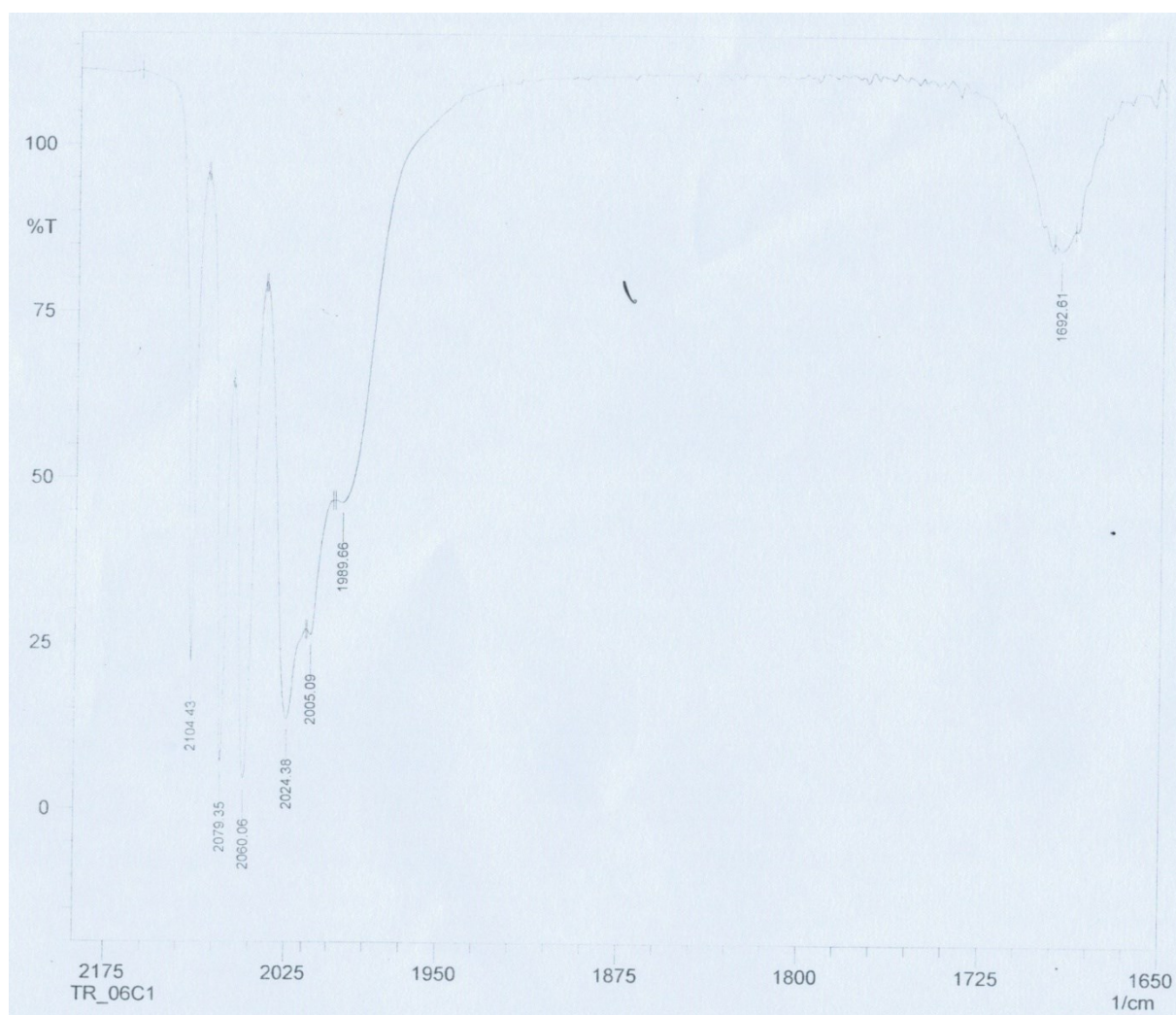


Figure S2. IR spectrum of $[\text{HOs}_3(\text{CO})_9(\mu_3\text{-C}_5\text{H}_4\text{NC=CHCO}_2)]$ (**3**) in CH_2Cl_2 .

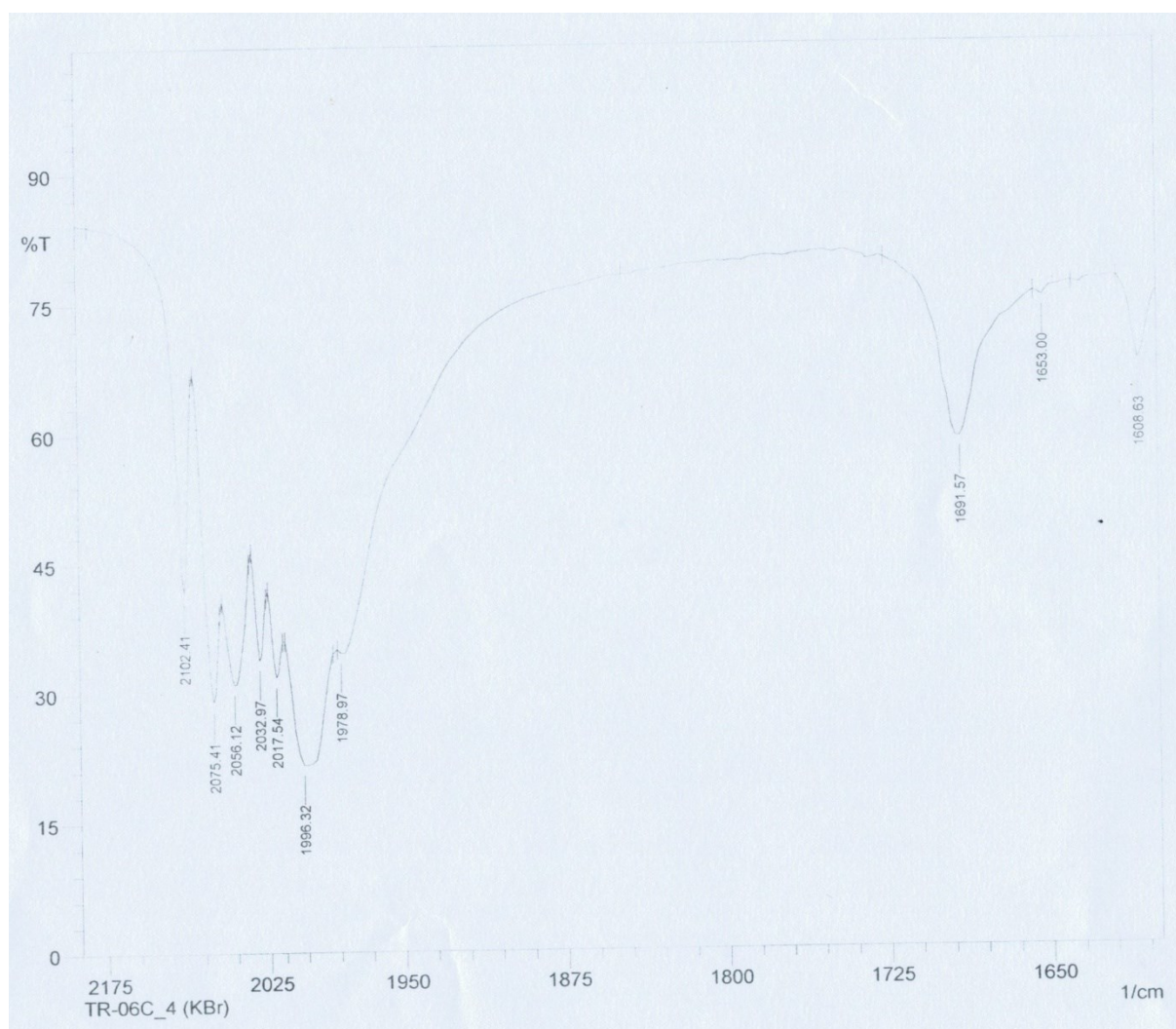


Figure S3. IR spectrum of $[\text{HOs}_3(\text{CO})_9(\mu_3\text{-C}_5\text{H}_4\text{NC=CHCO}_2)]$ (**3**) in KBr.

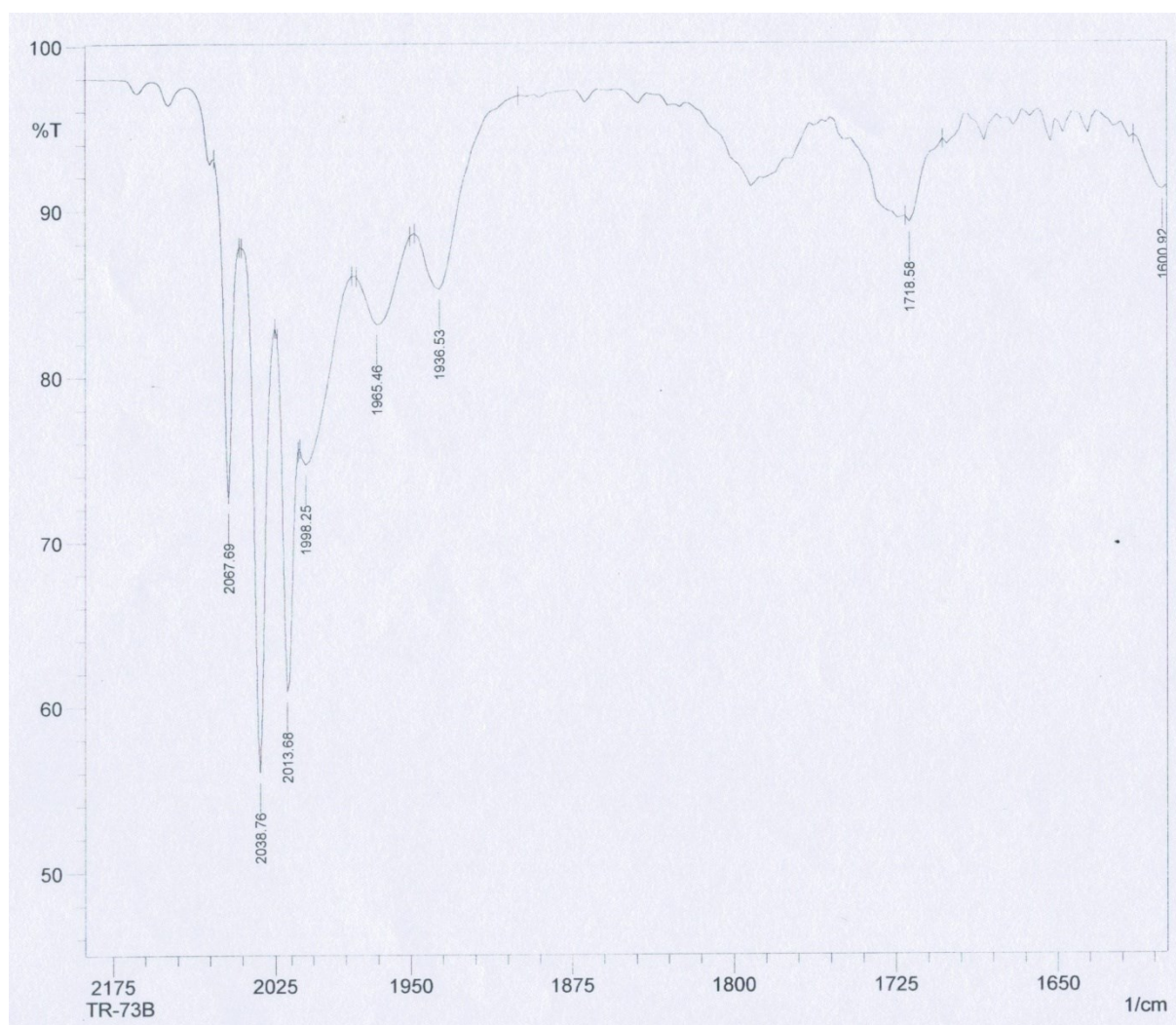


Figure S4. IR spectrum of $[\text{Ru}_3(\text{CO})_7(\mu\text{-CO})\{\mu_3\text{-C}_5\text{H}_4\text{NC}=\text{CHC}(\text{C}_5\text{H}_4\text{N})=\text{CH}\}]$ (**5**) in CH_2Cl_2 .

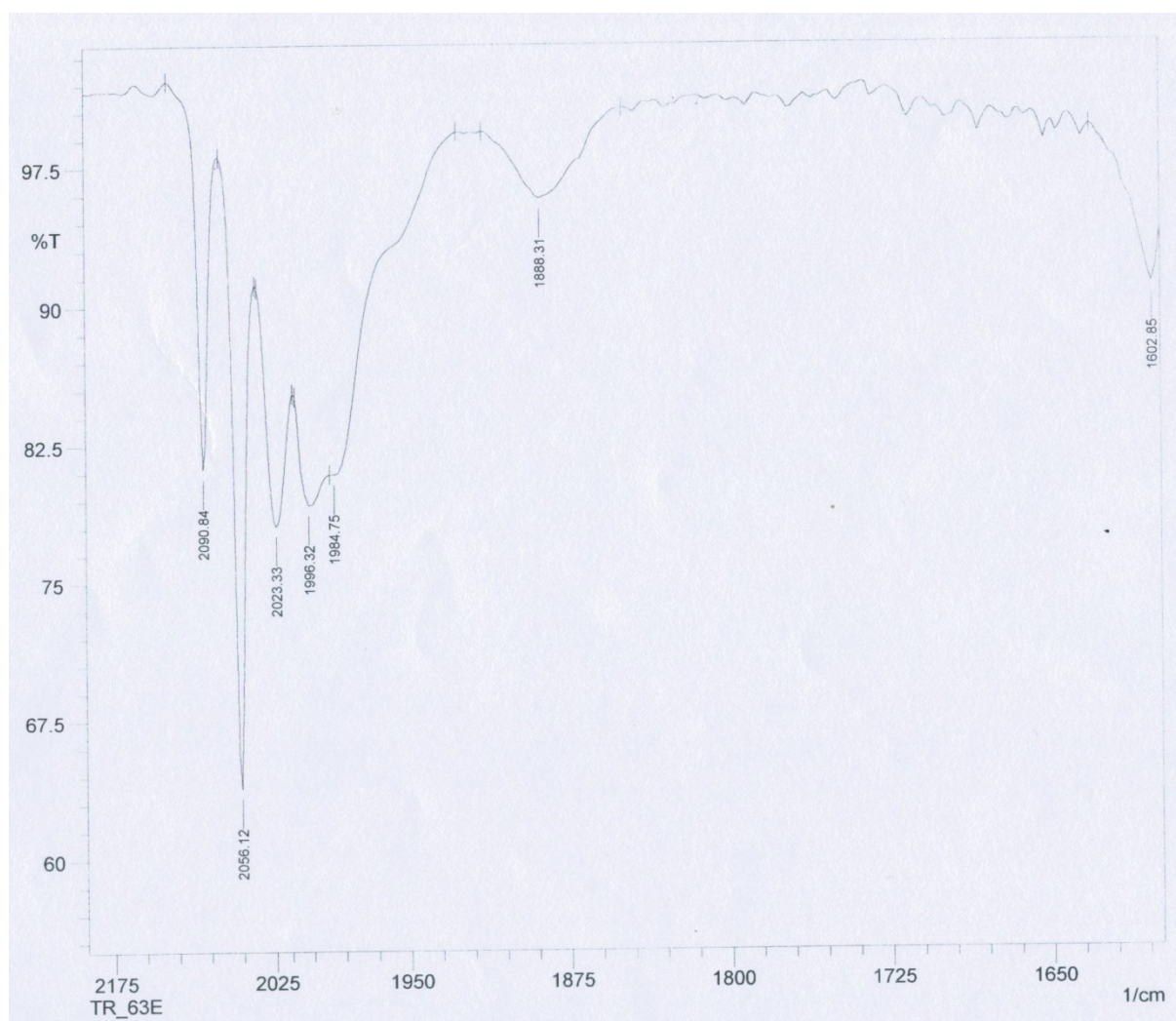


Figure S5. IR spectrum of $[\text{Ru}_3(\text{CO})_7(\mu\text{-CO})\{\mu_3\text{-C}_5\text{H}_4\text{NCCHC}(\text{C}_5\text{H}_4\text{N})\text{CHCHC}(\text{C}_5\text{H}_4\text{N})\}]$ (**6**) in CH_2Cl_2 .

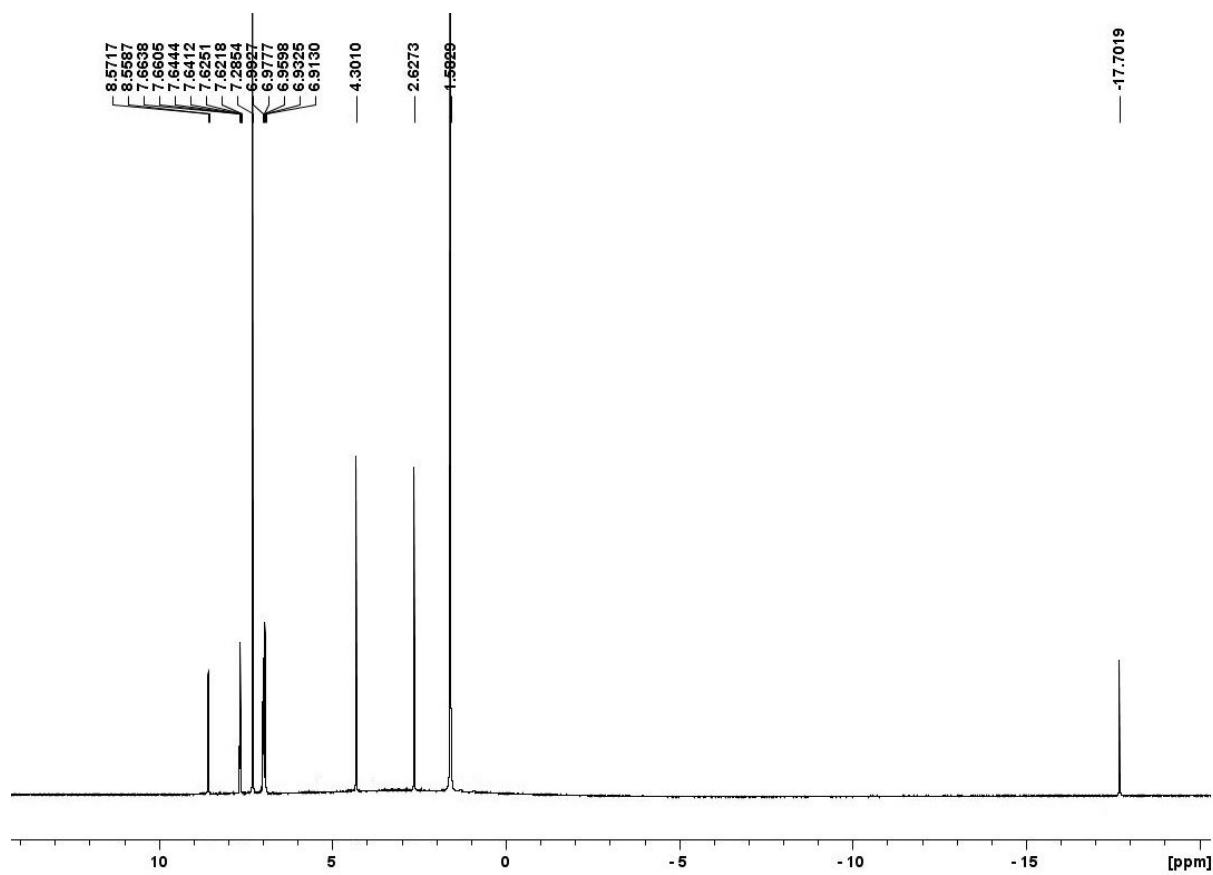


Figure S6. ^1H NMR spectrum of $[\text{HOs}_3(\text{CO})_9(\mu_3\text{-C}_5\text{H}_4\text{NC}=\text{CH}_2)]$ (**2**) in CDCl_3 at 400 MHz. The spectrum also shows residual proton signal of CDCl_3 (at δ 7.28) and H_2O (at δ 1.50).

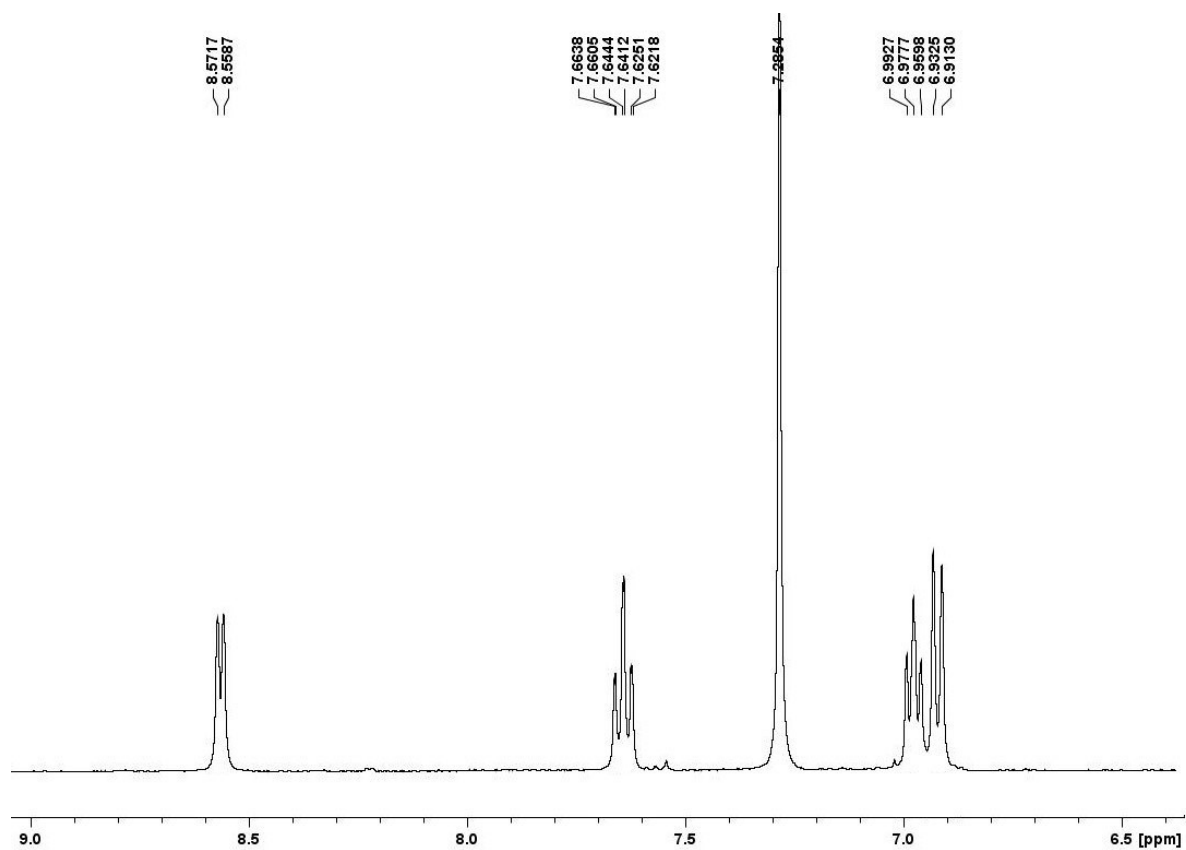


Figure S7. Aromatic region of the ^1H NMR spectrum of $[\text{HOs}_3(\text{CO})_9(\mu_3\text{-C}_5\text{H}_4\text{NC}=\text{CH}_2)]$ (**2**) in CDCl_3 at 400 MHz. The spectrum also shows residual proton signal of CDCl_3 (at δ 7.28).

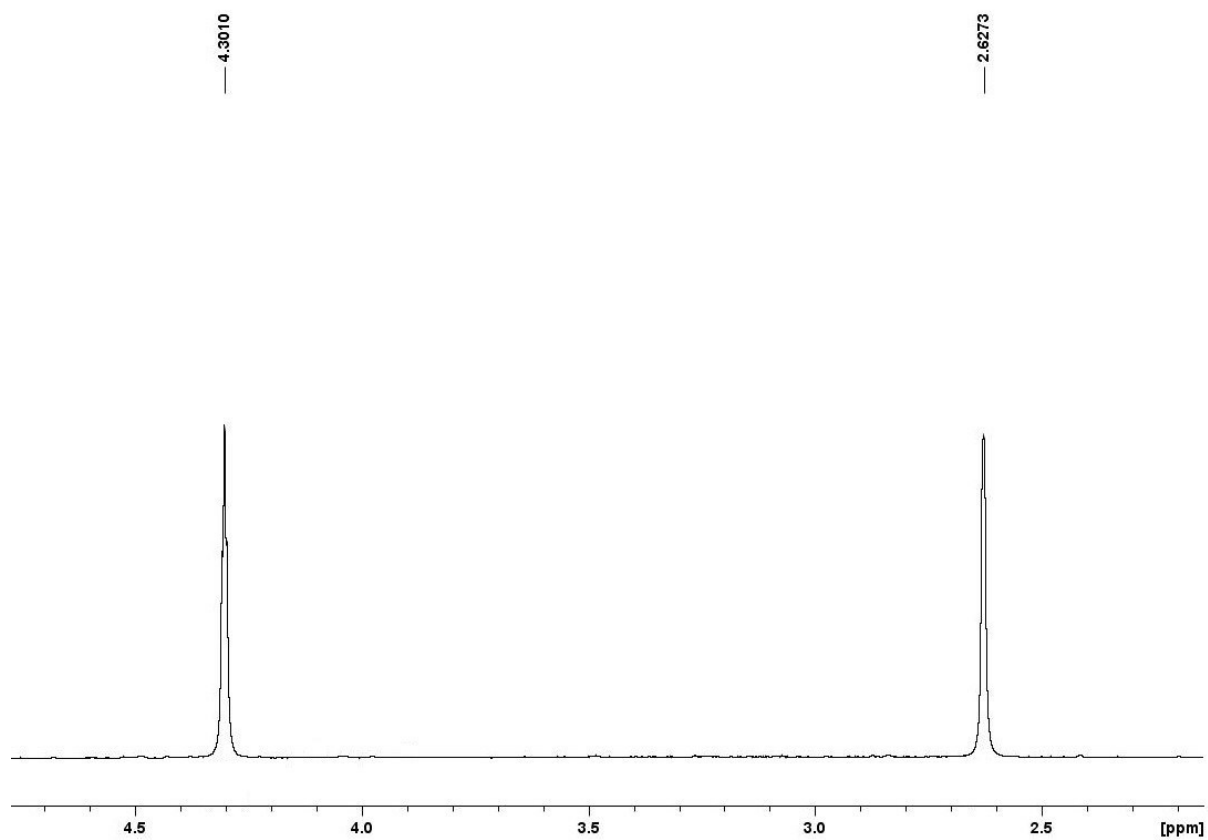


Figure S8. Aliphatic region of the ^1H NMR spectrum of $[\text{HOs}_3(\text{CO})_9(\mu_3\text{-C}_5\text{H}_4\text{NC}=\text{CH}_2)]$ (2) in CDCl_3 at 400 MHz.

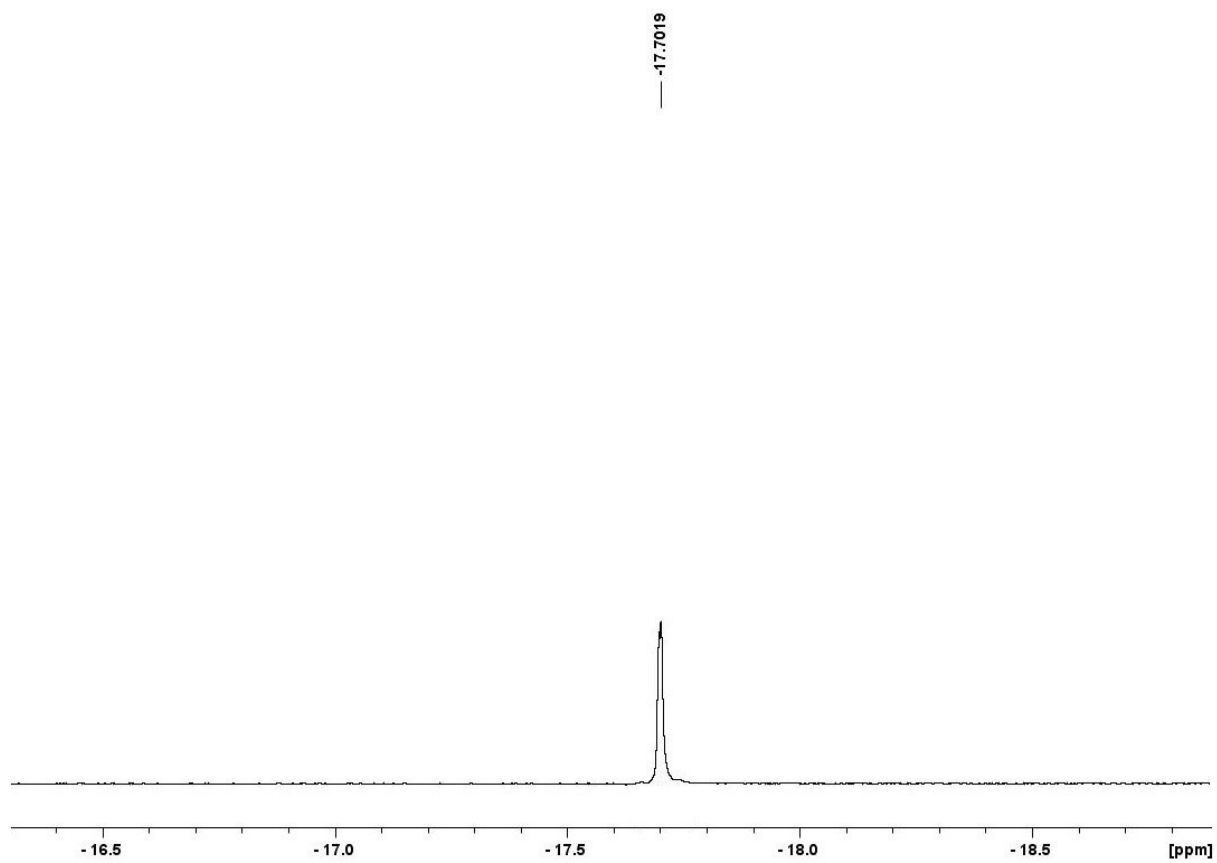


Figure S9. Hydride region of the ^1H NMR spectrum of $[\text{Os}_3(\text{CO})_9(\mu_3\text{-C}_5\text{H}_4\text{NC}=\text{CH}_2)]$ (**2**) in CDCl_3 at 400 MHz.

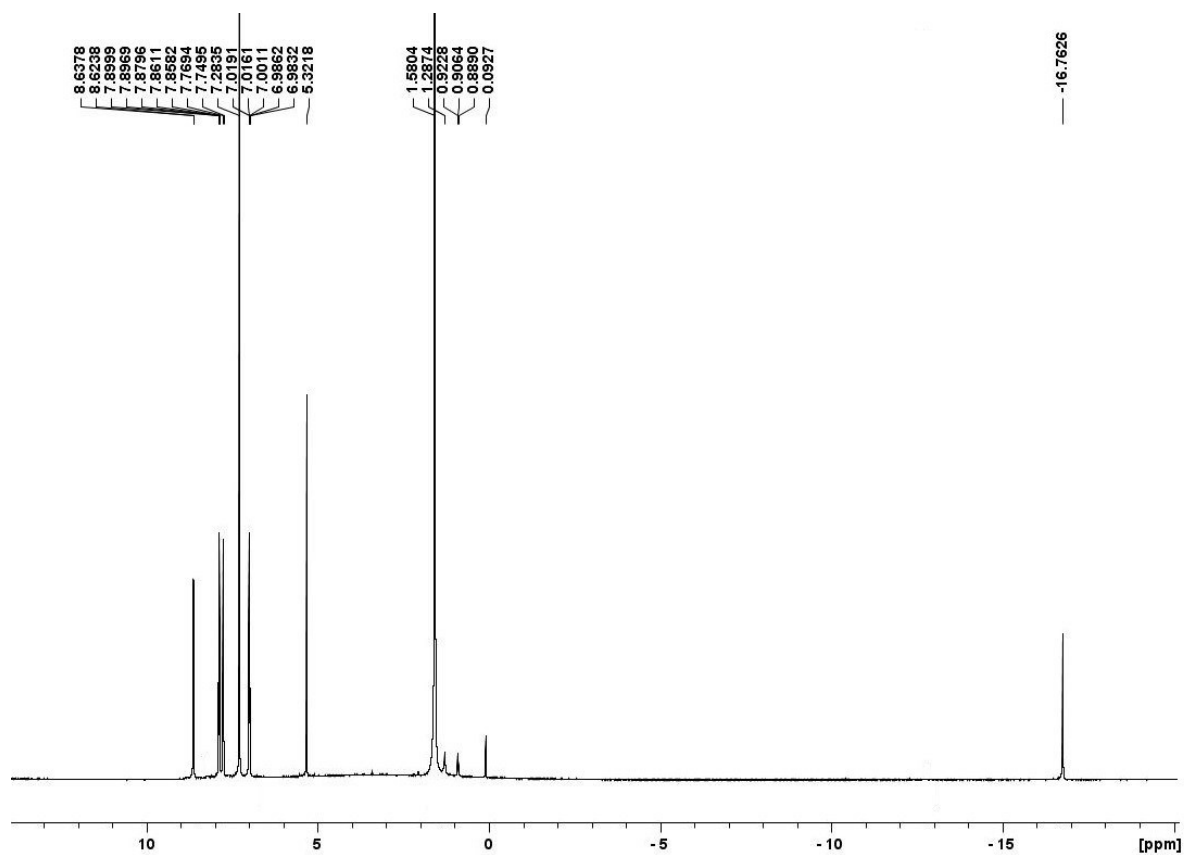


Figure S10. ^1H NMR spectrum of $[\text{HOs}_3(\text{CO})_9(\mu_3\text{-C}_5\text{H}_4\text{NC=CHCO}_2)]$ (**3**) in CDCl_3 at 400 MHz. The spectrum also shows residual proton signal of CDCl_3 (at δ 7.28) and H_2O (at δ 1.58). The compound was recrystallized from *n*-hexane/ CH_2Cl_2 for purification during workup whose signals are also present in the spectrum (CH_2Cl_2 at δ 5.32 and *n*-hexane at δ 1.28 and 0.90).

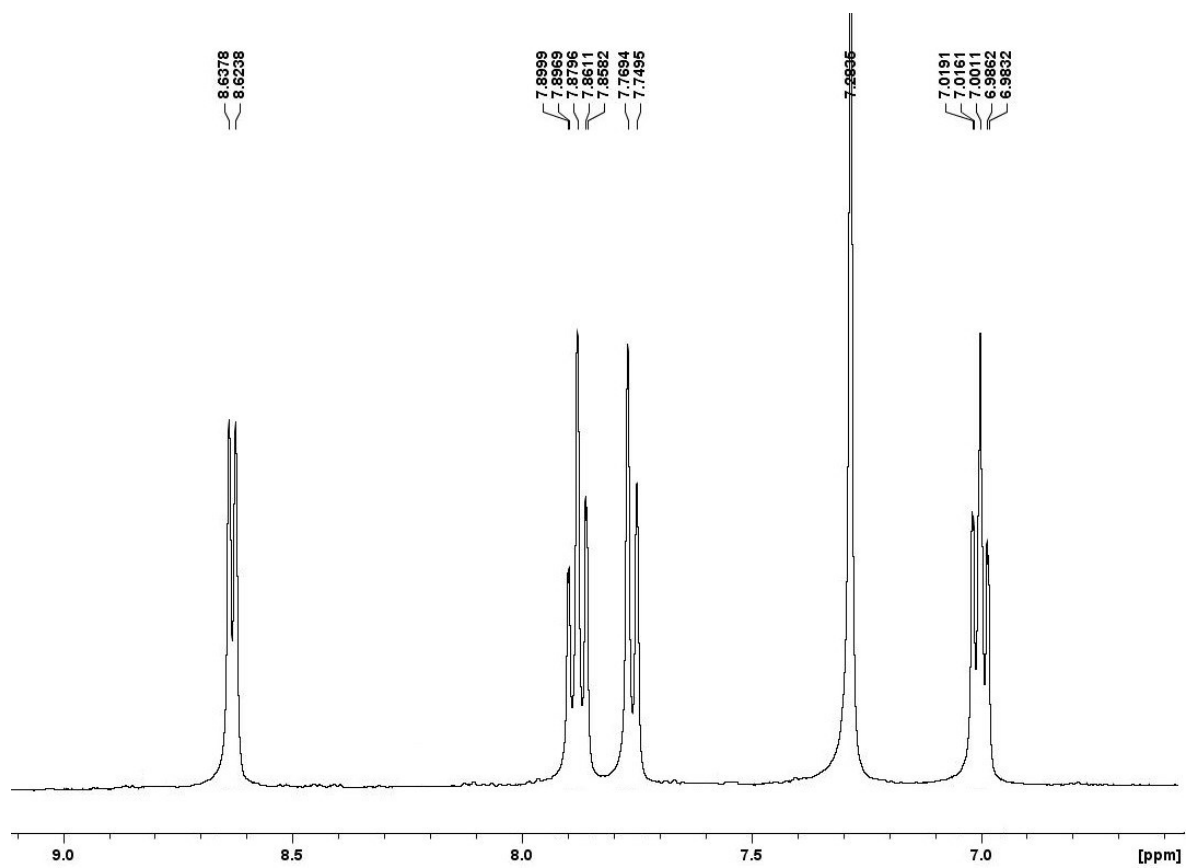


Figure S11. Aromatic region of the ^1H NMR spectrum of $[\text{HO}_3(\text{CO})_9(\mu_3\text{-C}_5\text{H}_4\text{NC}=\text{CHCO}_2)]$ (**3**) in CDCl_3 at 400 MHz. The spectrum also shows residual proton signal of CDCl_3 (at δ 7.28).

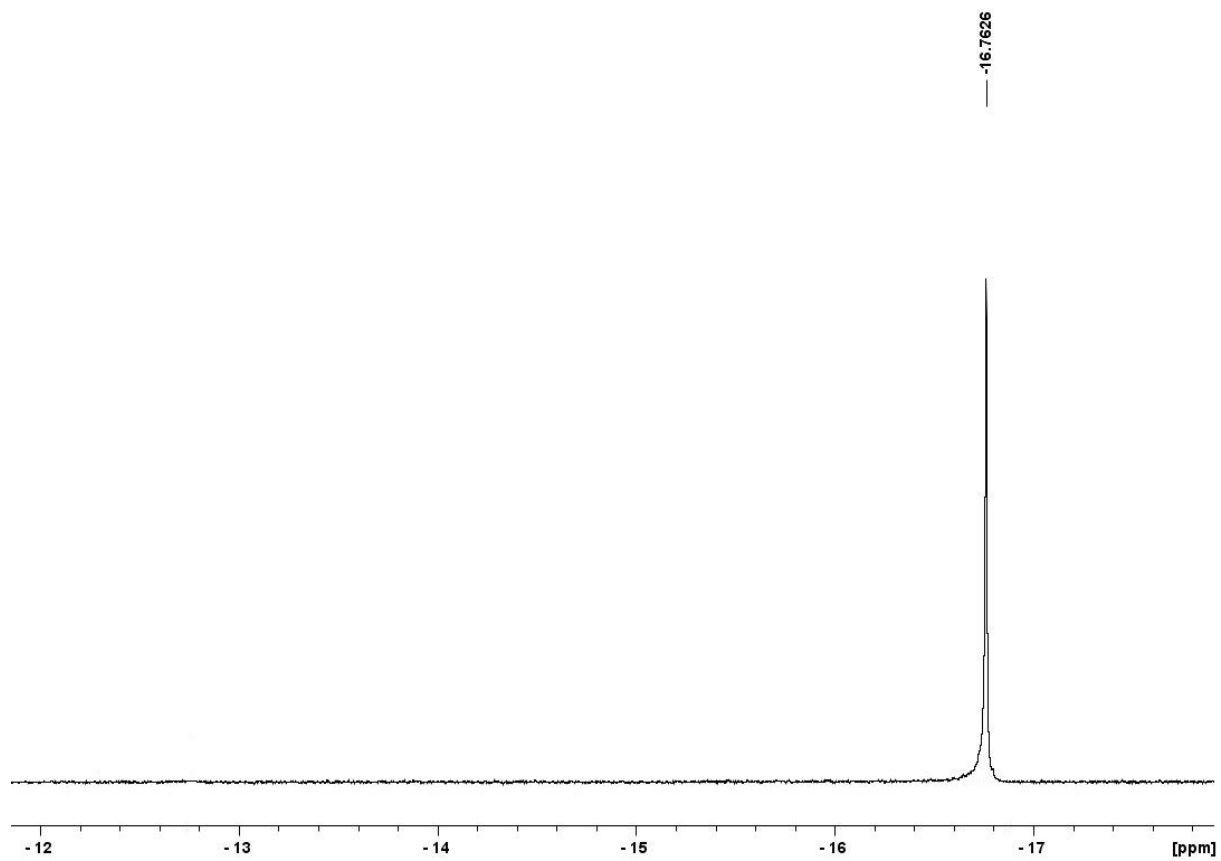


Figure S12. Hydride region of the ^1H NMR spectrum of $[\text{HOs}_3(\text{CO})_9(\mu_3\text{-C}_5\text{H}_4\text{NC}=\text{CHCO}_2)]$ (**3**) in CDCl_3 at 400 MHz.

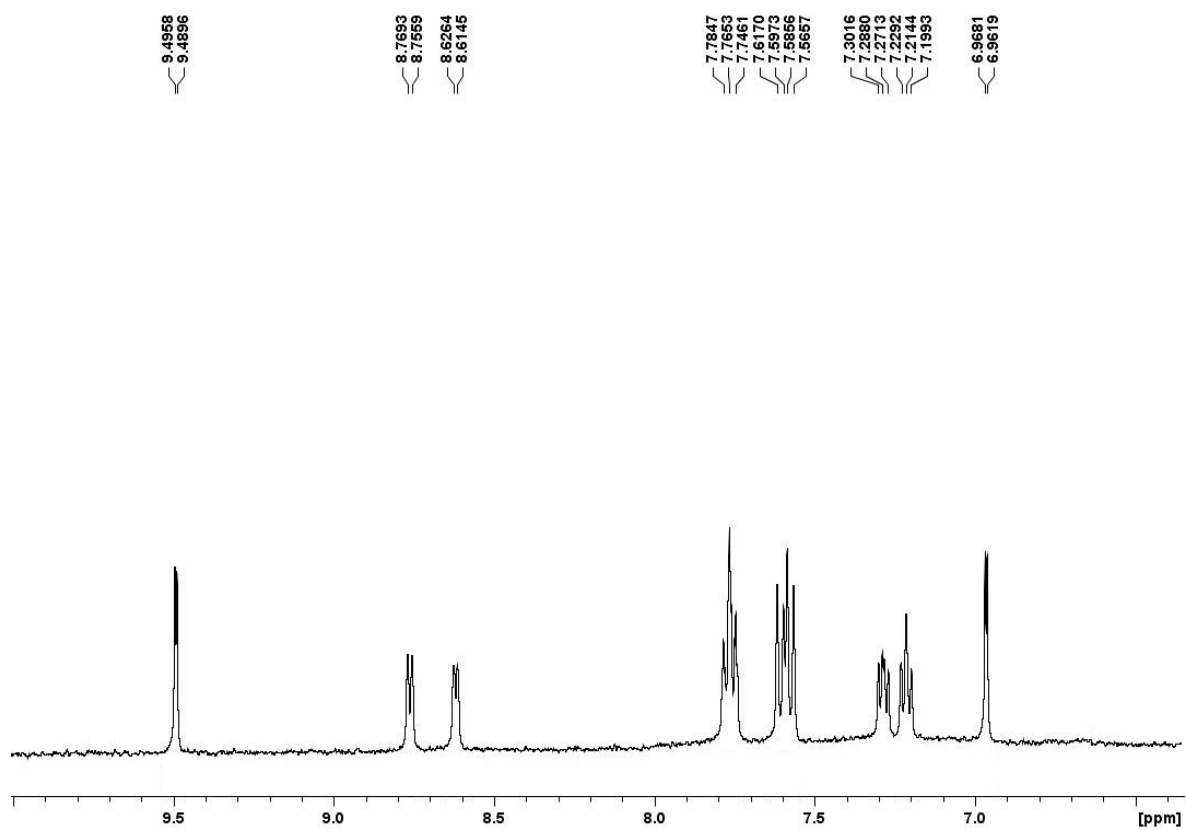


Figure S13. ^1H NMR spectrum of $[\text{Ru}_3(\text{CO})_7(\mu\text{-CO})\{\mu_3\text{-C}_5\text{H}_4\text{NC}=\text{CHC}(\text{C}_5\text{H}_4\text{N})=\text{CH}\}]$ (**5**) in CD_2Cl_2 at 400 MHz.

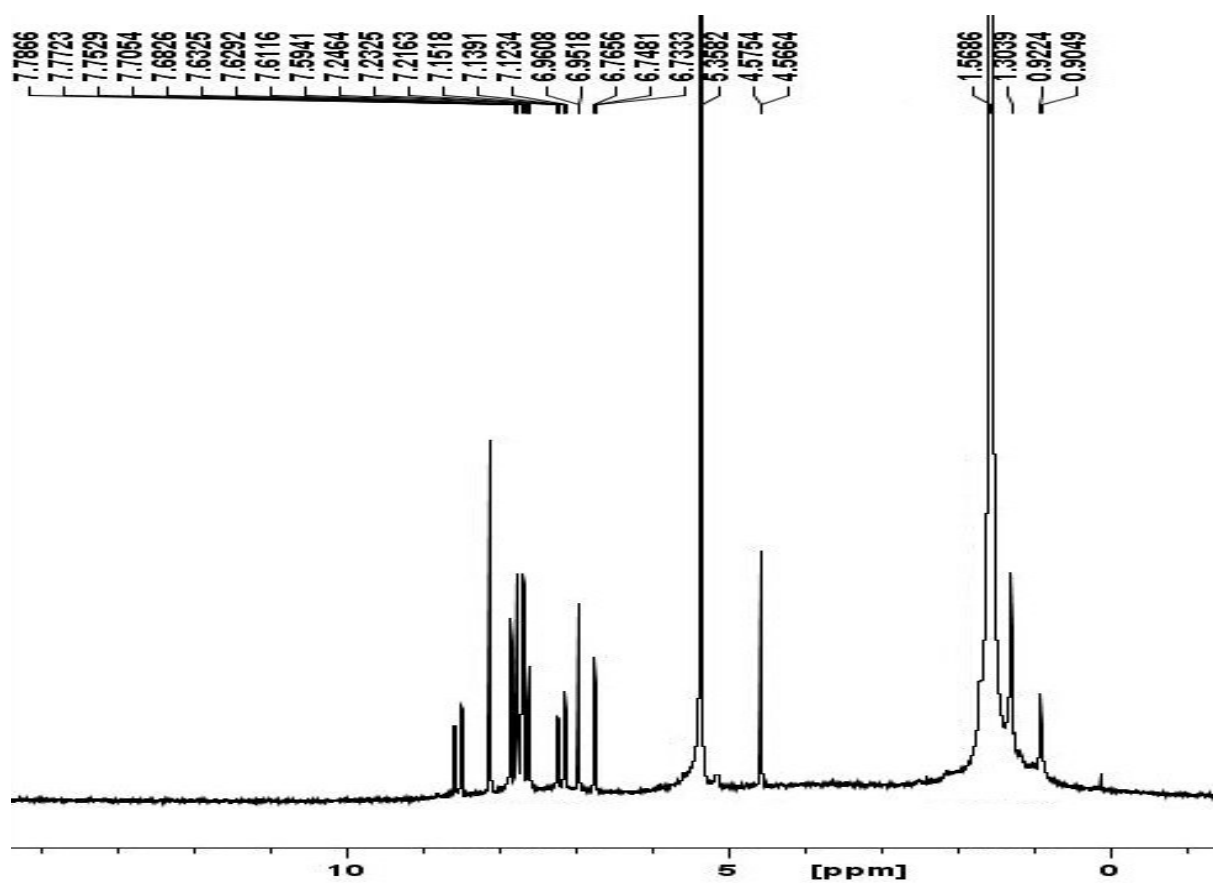


Figure S14. ¹H NMR spectrum of [Ru₃(CO)₇(μ-CO){μ₃-C₅H₄NCCHC(C₅H₄N)CHCHC(C₅H₄N)}] (**6**) in CD₂Cl₂ at 400 MHz. The spectrum also shows residual proton signal of CD₂Cl₂ (at δ 5.35) and H₂O (at δ 1.56). The compound was recrystallized from *n*-hexane/CH₂Cl₂ for purification during workup whose signals are also present in the spectrum (*n*-hexane at δ 1.30 and 0.92).

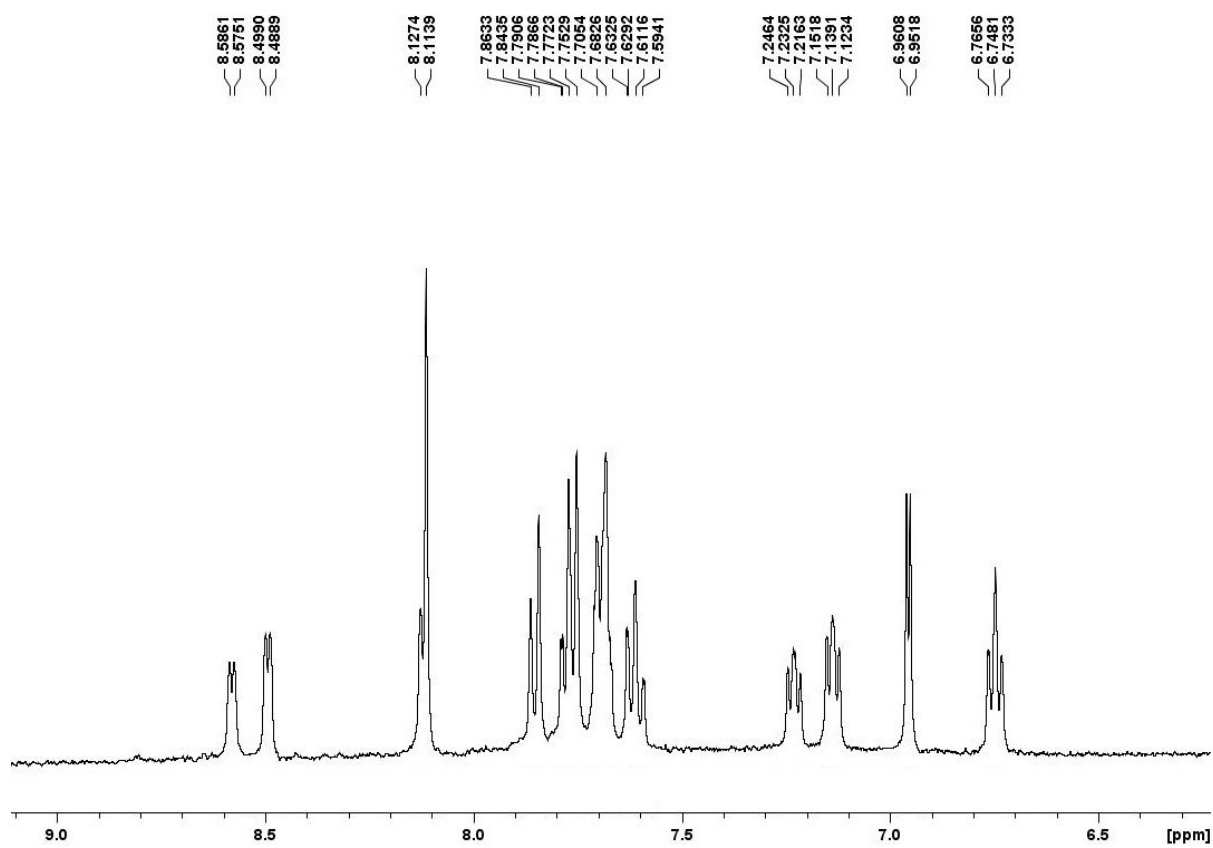


Figure S15. Aromatic region of the ^1H NMR spectrum of $[\text{Ru}_3(\text{CO})_7(\mu\text{-CO})\{\mu_3\text{-C}_5\text{H}_4\text{NCCHC}(\text{C}_5\text{H}_4\text{N})\text{CHCHC}(\text{C}_5\text{H}_4\text{N})\}]$ (**6**) in CD_2Cl_2 at 400 MHz.

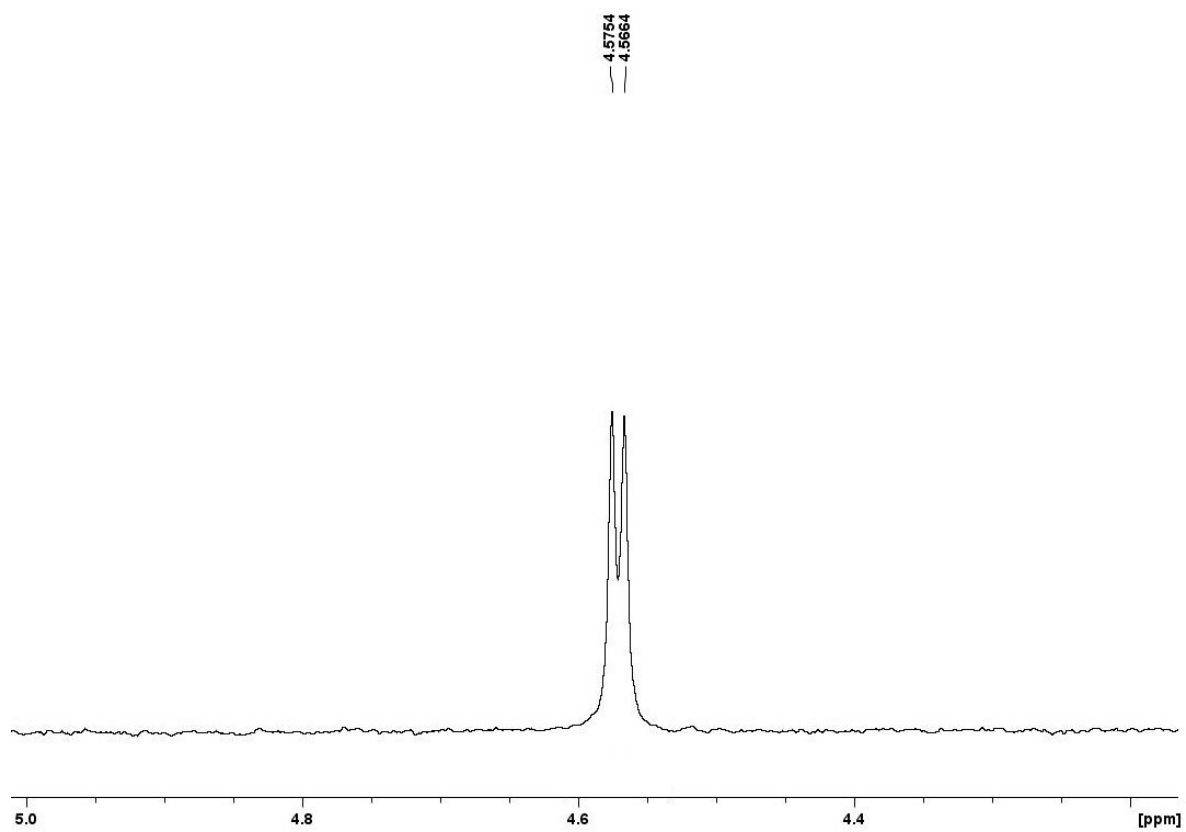


Figure S16. Aliphatic region of the ^1H NMR spectrum of $[\text{Ru}_3(\text{CO})_7(\mu\text{-CO})\{\mu_3\text{-C}_5\text{H}_4\text{NCCHC}(\text{C}_5\text{H}_4\text{N})\text{CHCHC}(\text{C}_5\text{H}_4\text{N})\}]$ (**6**) in CD_2Cl_2 at 400 MHz.