

Supporting

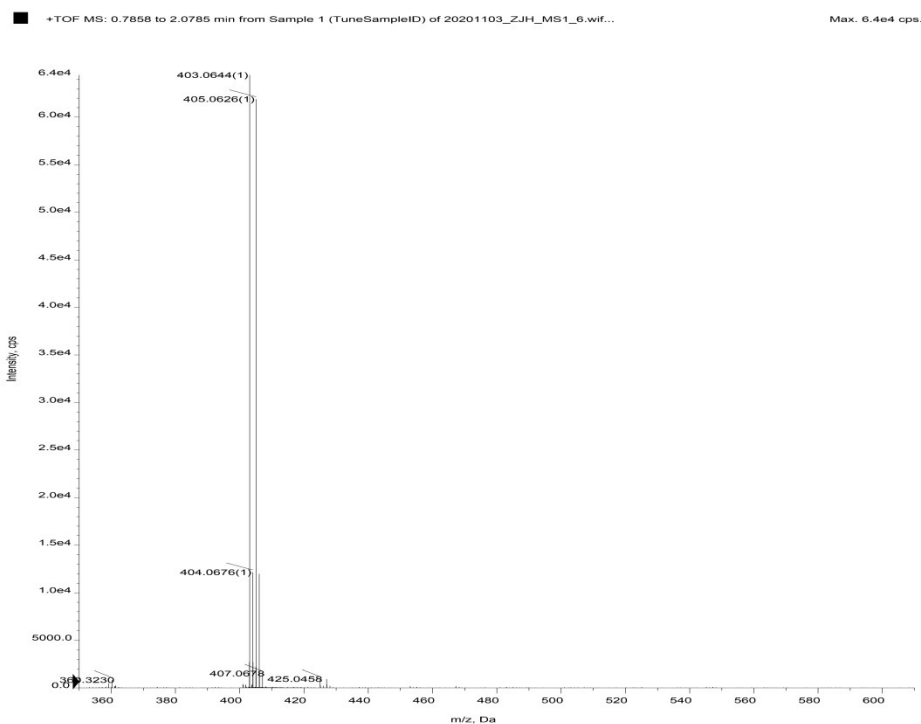


Fig. S1. TOF Mass spectrum of Compound 1.

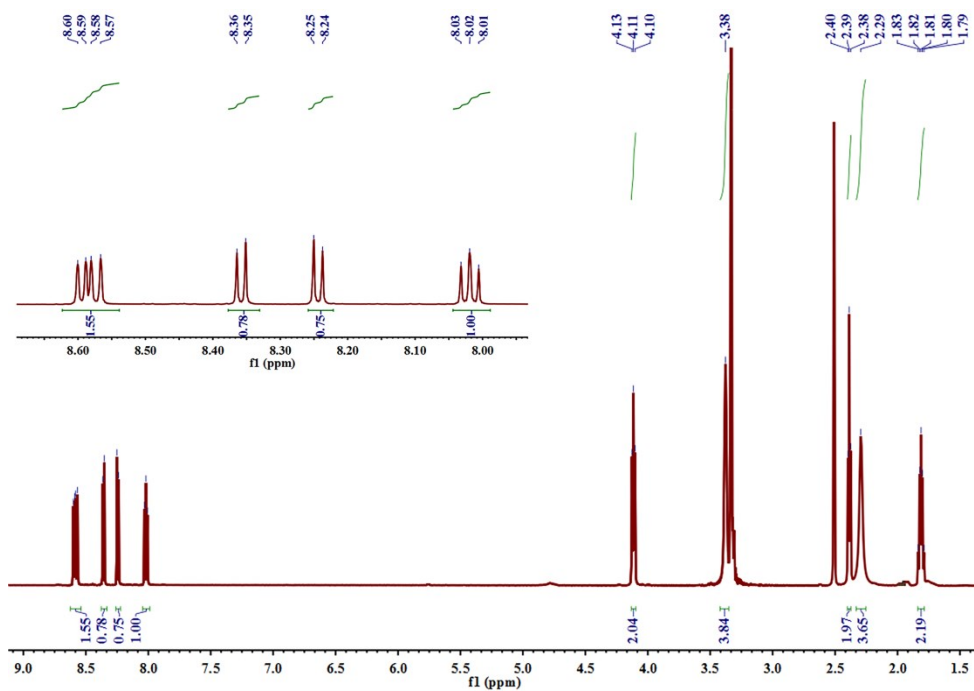


Fig. S2. ^1H NMR spectrum of Compound 1 ($\text{DMSO}-d_6$).

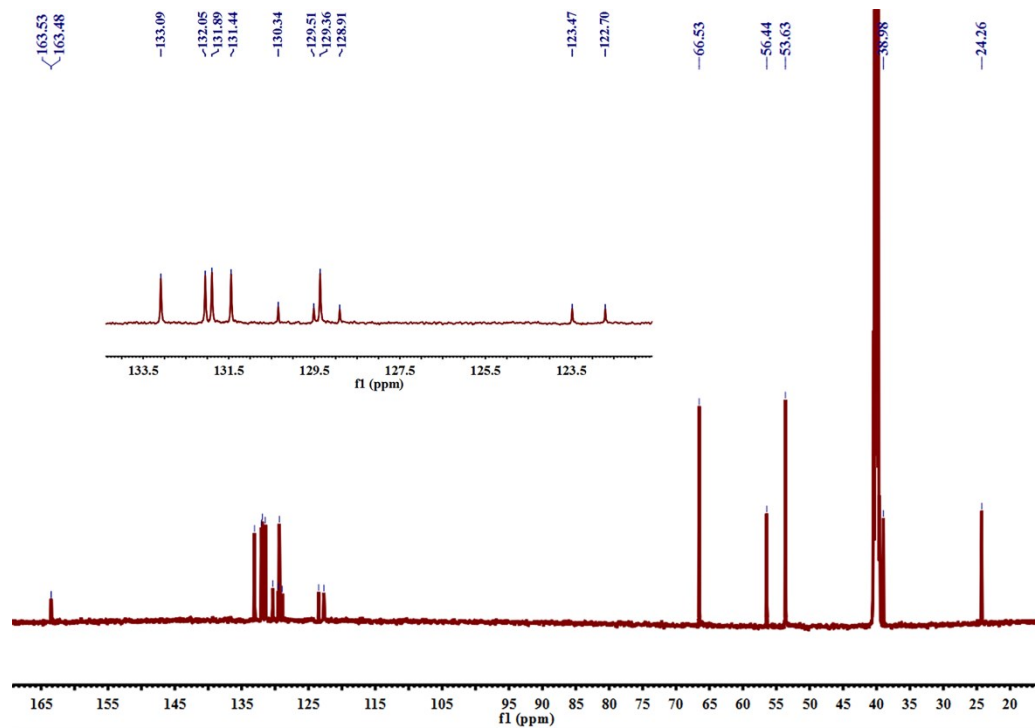


Fig. S3. ^{13}C NMR spectrum of Compound 1(DMSO- d_6).

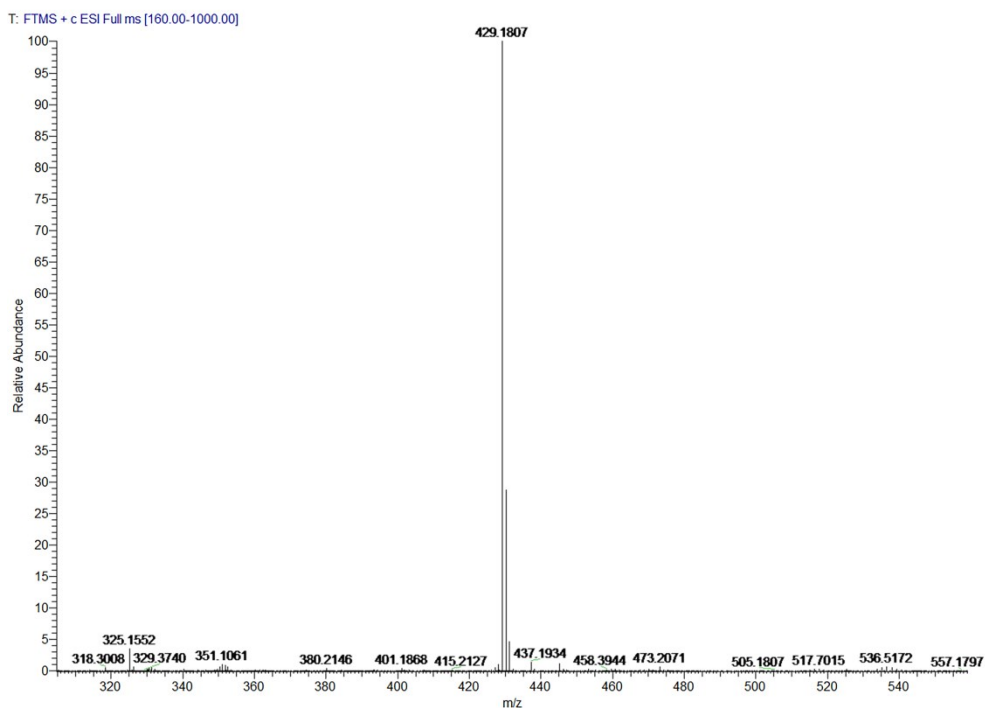


Fig. S4. FTMS spectrum of MFNI

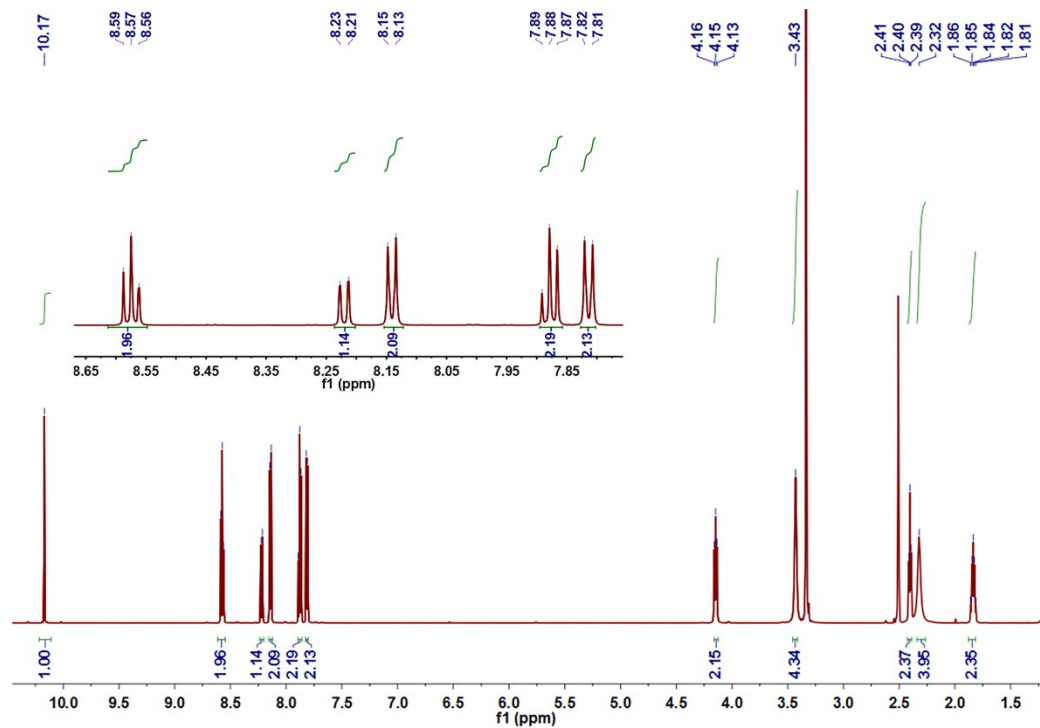


Fig. S5. ^1H NMR spectrum of MFNI ($\text{DMSO}-d_6$).

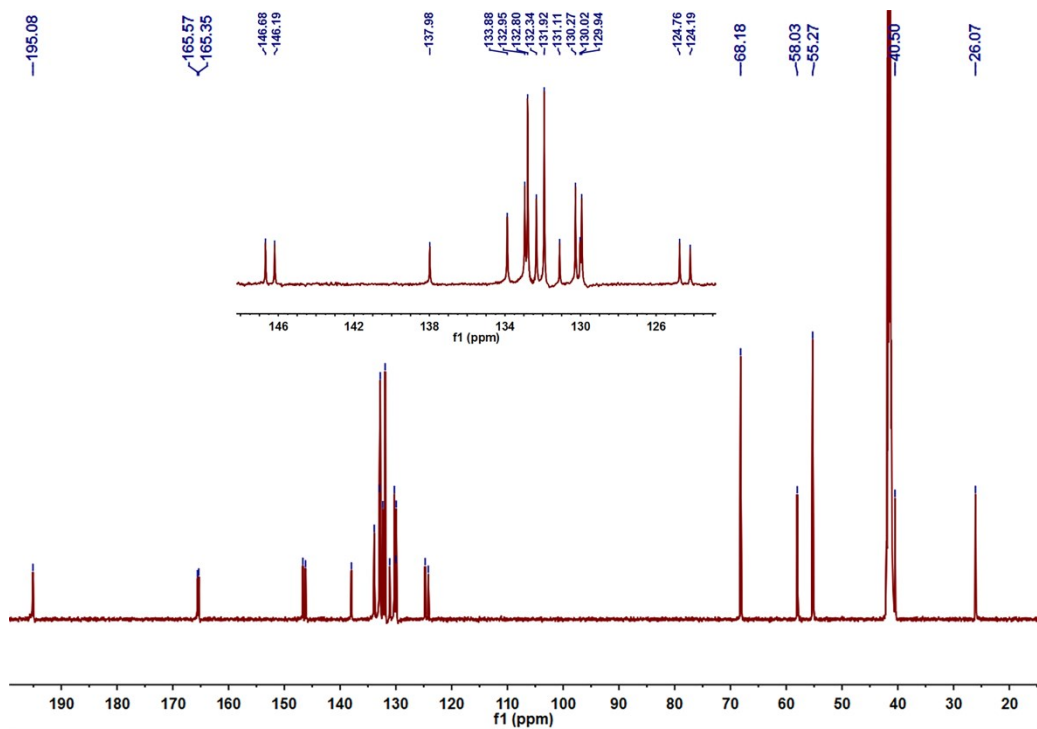


Fig. S6. ^{13}C NMR spectrum of MFNI ($\text{DMSO}-d_6$).

F1 #9 RT: 0.12 AV: 1 NL: 7.31E8
T: FTMS + c ESI Full ms [100.00-1000.00]

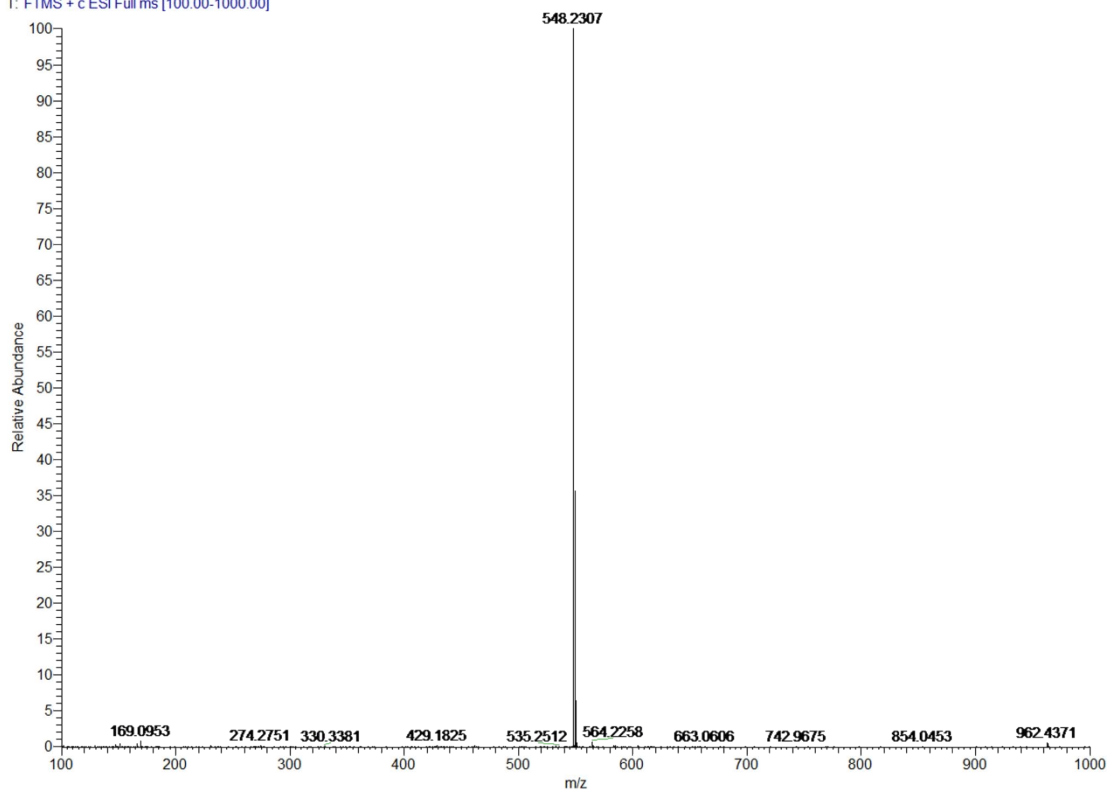


Fig. S7. FTMS spectrum of CuNI.

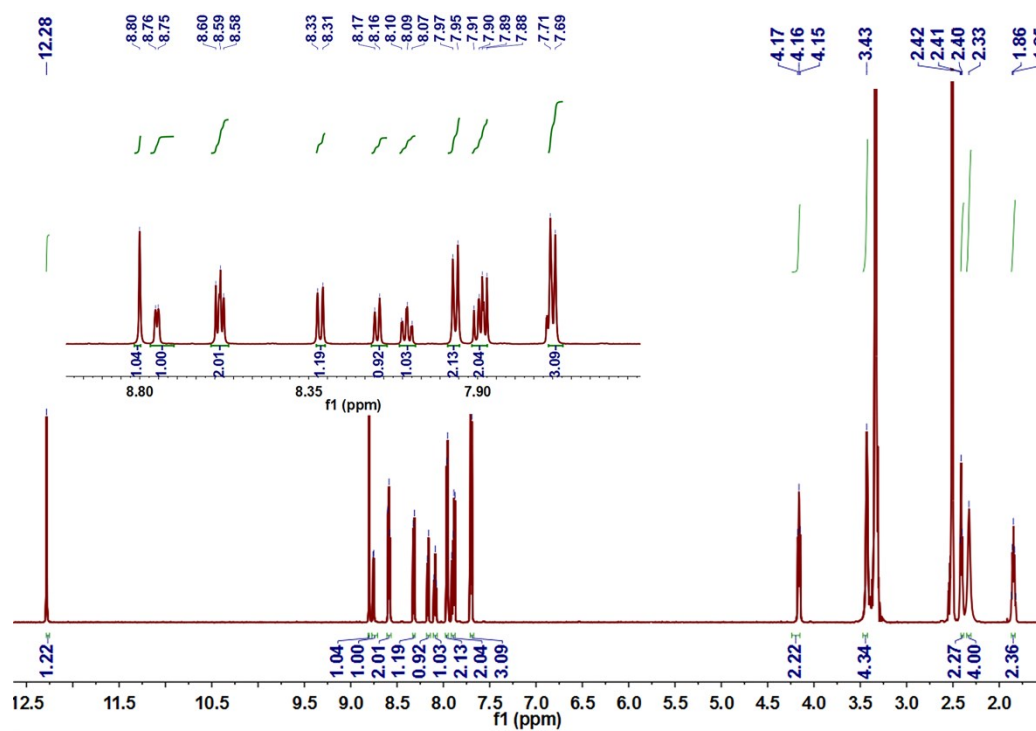


Fig. S8. ^1H NMR spectrum of CuNI (DMSO- d_6).

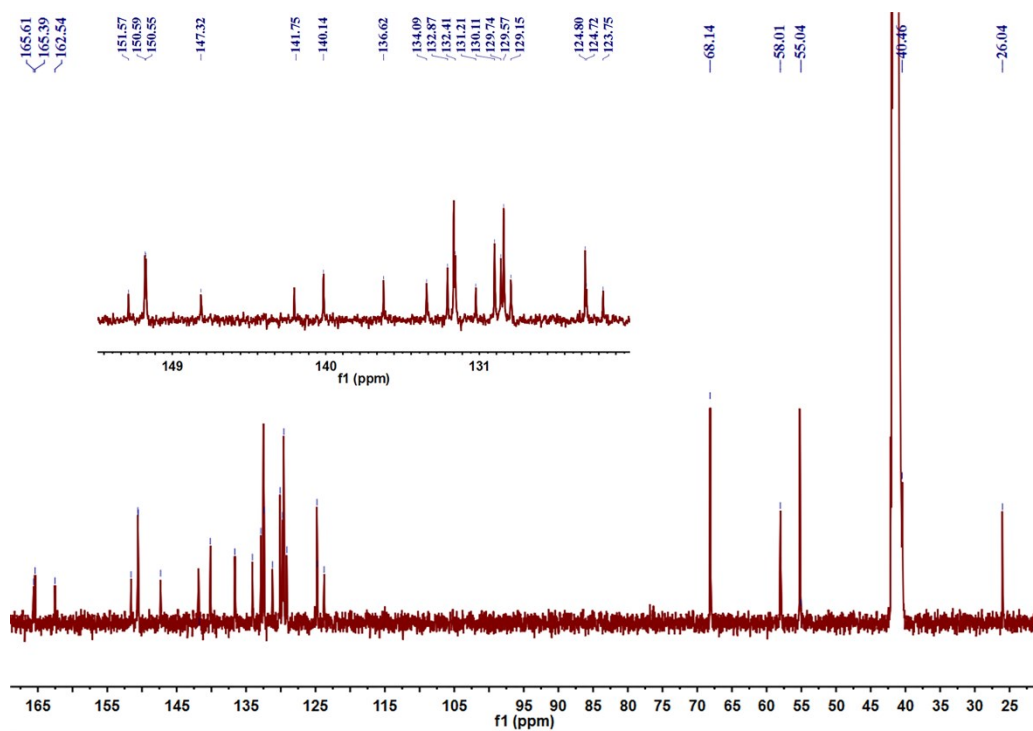
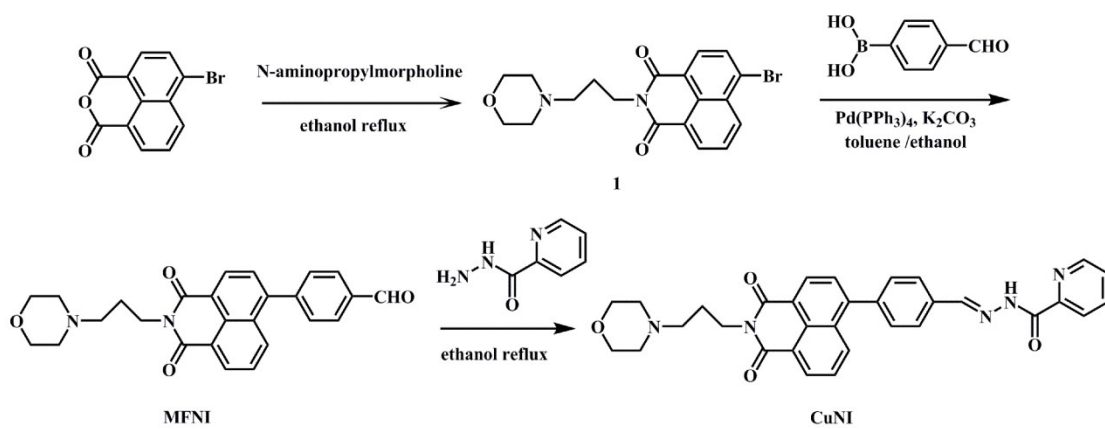


Fig. S9. ^{13}C NMR spectrum of **CuNI** ($\text{DMSO-}d_6$).



Scheme S1. The synthetic route of **CuNI**.

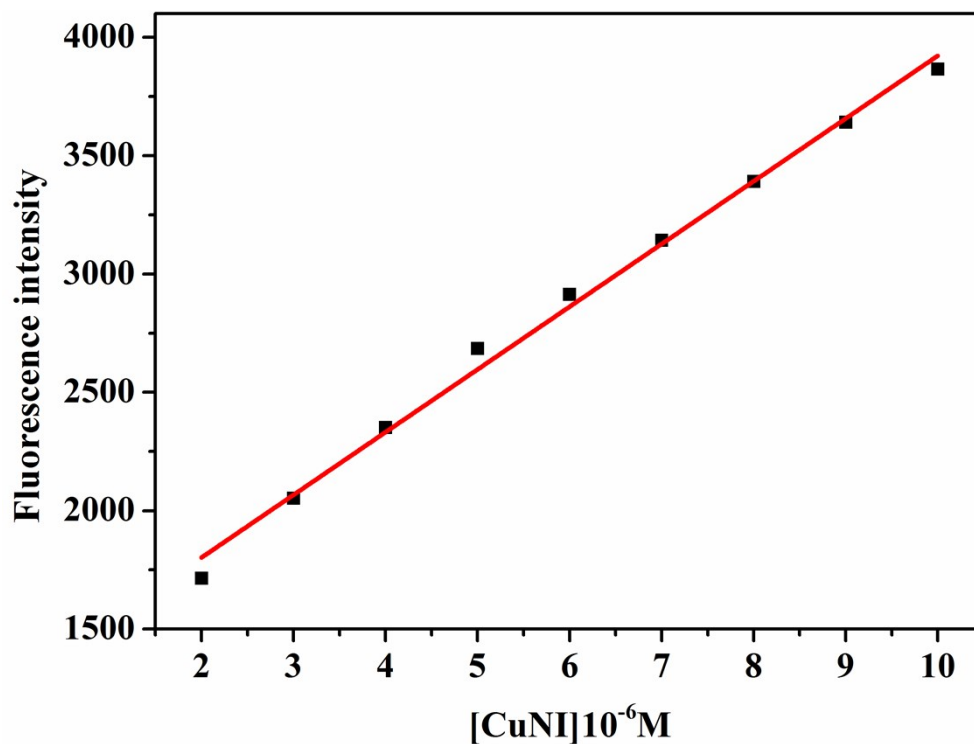


Fig. S10. Fluorescence changes of **CuNI** ($10 \mu\text{M}$) upon addition of Cu^{2+} (0.2~1 equiv.) in HOAc-NaOAc buffer solution (10 mM, pH 5, with 1% DMSO) at $25 \text{ }^\circ\text{C}$. ($I_{440 \text{ nm}}$, $\lambda_{\text{ex}} = 370 \text{ nm}$).

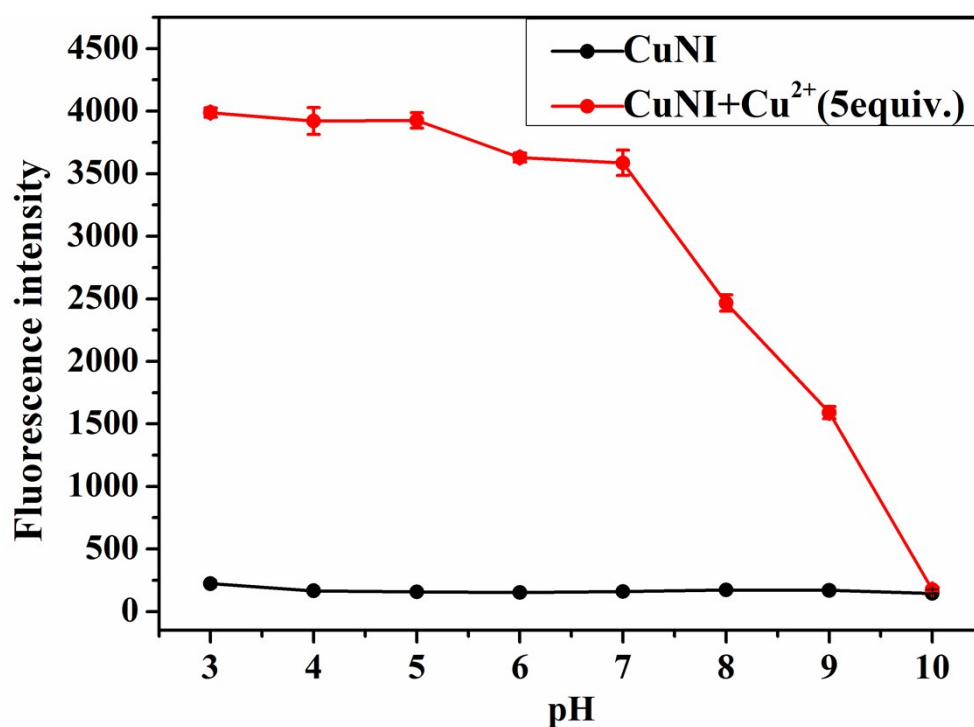


Fig. S11. The fluorescence intensity of **CuNI**($10 \mu\text{M}$) and **CuNI** ($10 \mu\text{M}$)+ Cu^{2+} (5equiv.) in pH 3-10 solution at $25 \text{ }^\circ\text{C}$ ($I_{440 \text{ nm}}$, $\lambda_{\text{ex}} = 370 \text{ nm}$).

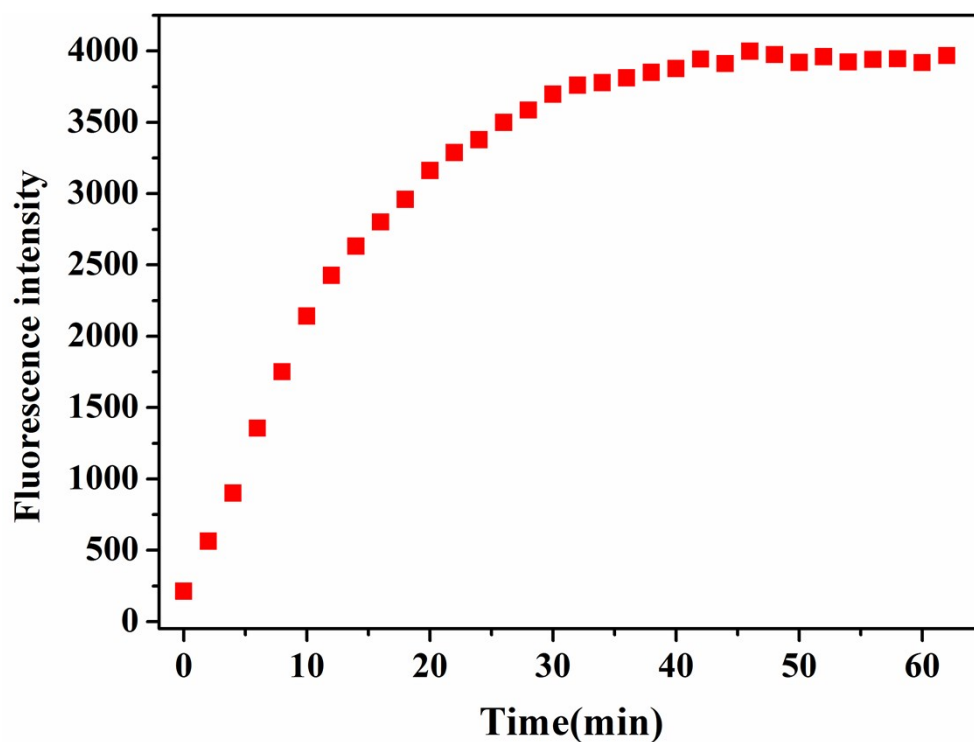


Fig. S12. The fluorescence emission intensity of **CuNI** (10 mM)+ Cu^{2+} (5 equiv.) content in HOAc-NaOAc buffer solution (10 mM, pH 5.0, 1% DMSO) at 25°C.

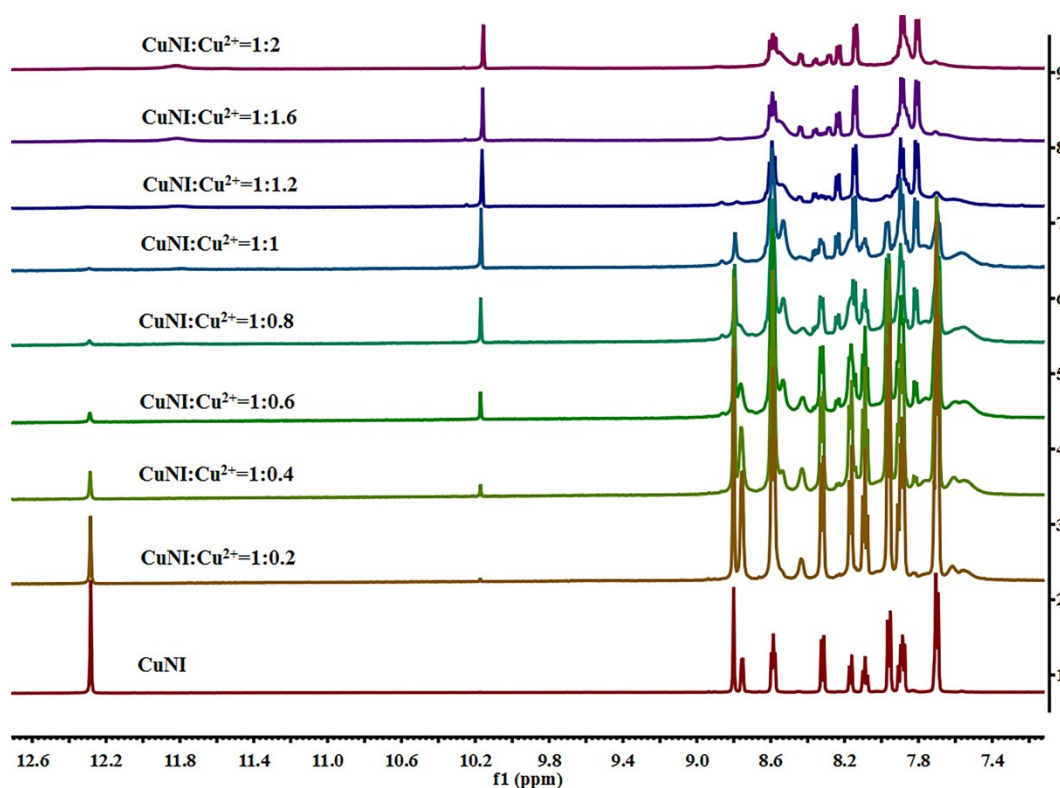


Fig. S13. ^1H NMR spectra of (1) **CuNI** in DMSO- d_6 , (2) **CuNI** in DMSO- d_6 + Cu^{2+} (0.2 equiv.) in D_2O , (3) **CuNI** in DMSO- d_6 + Cu^{2+} (0.4 equiv.) in D_2O , (4) **CuNI** in DMSO- d_6 + Cu^{2+} (0.6 equiv.) in D_2O , (5) **CuNI** in DMSO- d_6 + Cu^{2+} (0.8 equiv.) in

D₂O, (6) **CuNI** in DMSO-d6 + Cu²⁺ (1.0 equiv.) in D₂O, (7) **CuNI** in DMSO-d6 + Cu²⁺ (1.2 equiv.) in D₂O, (8) **CuNI** in DMSO-d6 + Cu²⁺ (1.6 equiv.) in D₂O, (9) **CuNI** in DMSO-d6 + Cu²⁺ (2.0 equiv.) in D₂O.

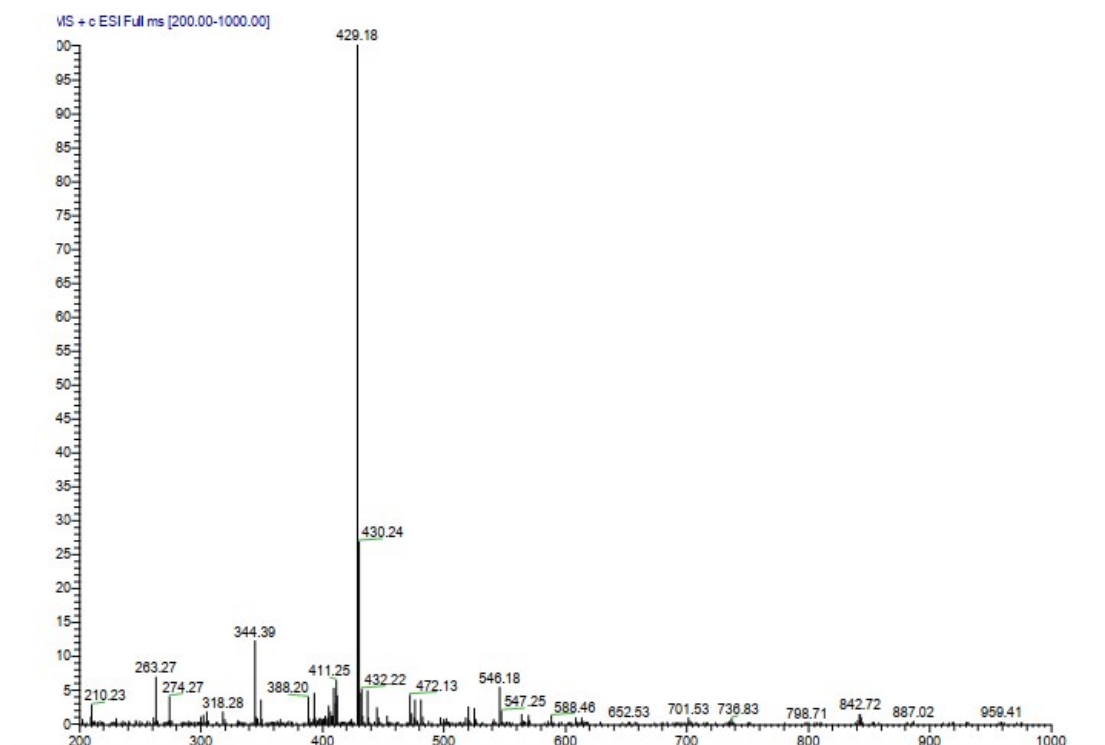


Fig. S14. FTMS spectrum of **CuNI** + Cu²⁺.

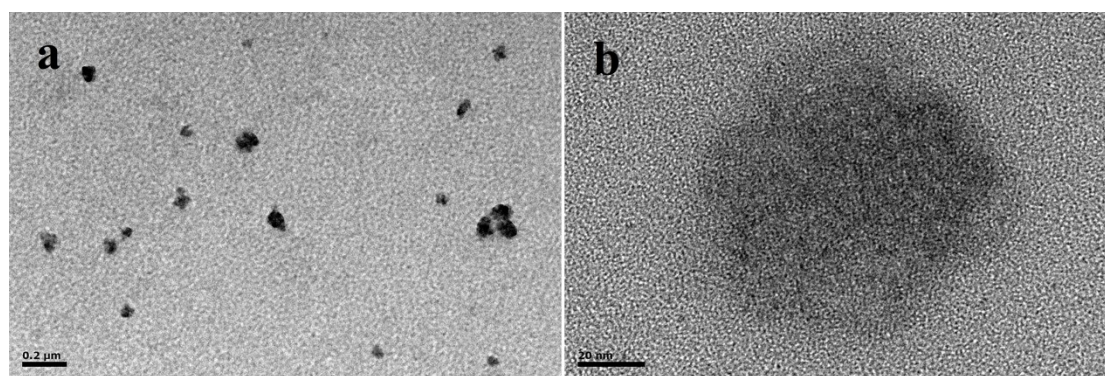


Fig. S15. (a) TEM of **CuNI** (10 μM) added with Cu²⁺ (5 equiv.) in HOAc-NaOAc buffer solution (10 mM, pH 5.0, with 1% DMSO). (b) Expansion of individual particles

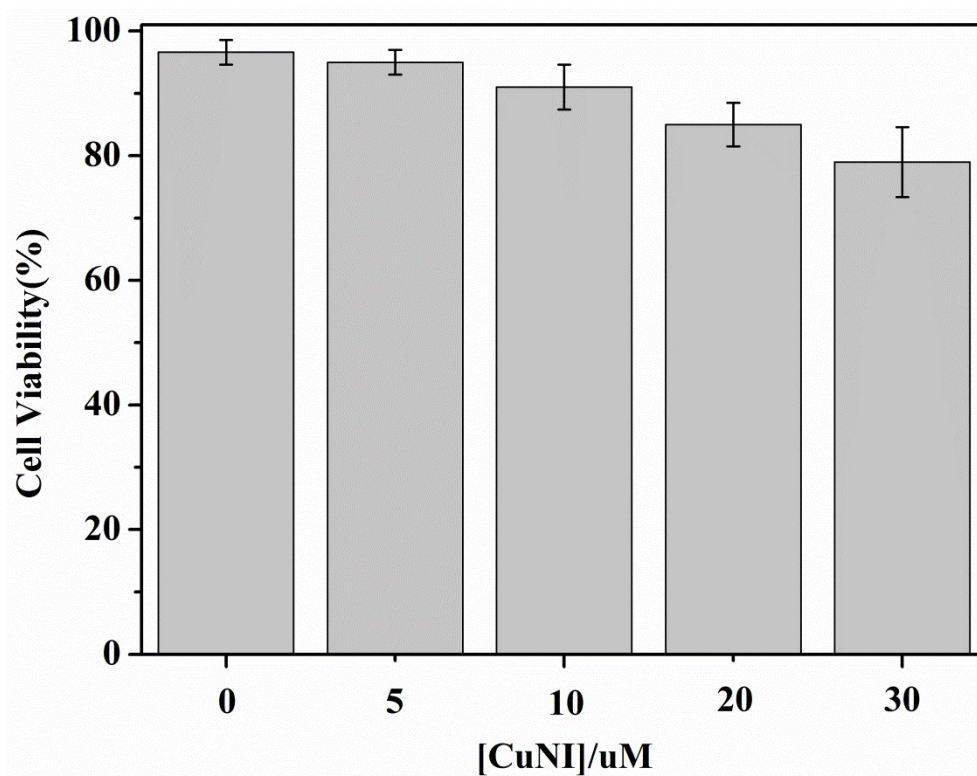


Fig S16. Cytotoxicity data of CuNI (HepG2 cells incubated for 24 h).