Supporting Information

Heterodimers Made of Metal-Organic Frameworks and Upconversion Nanoparticles for Bioimaging and pHresponsive Dual-drug Delivery

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Fig. S1. The particle size distribution maps of (a) UCNPs, (b) PVP-UCNPs, (c) UCMOFs, (d) UCMOFs@D and (e) UCMOFs@D@5 in water.



Fig. S2. The Zeta potentials of UCNPs, PVP-UCNPs, UCMOFs, UCMOFs@D, and UCMOFs@D@5 in water (200 µg mL⁻¹).



Fig. S3. XRD patterns of UCNPs, UCMOFs, UCMOFs@D@5, and standard hexagonal NaYF₄ (JCPDS: 16-033). *: The characteristic diffraction peaks of MOFs.



Fig. S4. Fourier transform infrared (FT-IR) spectra of OA-UCNPs, PVP-UCNPs, UCMOFs, UCMOFs@D, and UCMOFs@D@5.



Fig. S5. Thermogravimetric analysis (TGA) curves for PVP-UCNPs and UCMOFs.



Fig. S6. Overlap of absorption spectrum of DOX (pink) and the emission spectrum of UCMOFs (green) upon 980 nm laser excitation (500 mW \cdot cm⁻²)



Fig. S7. UV-visible absorption spectra of UCMOFs, UCMOFs@D, and UCMOFs@D@5.



Fig. S8. (a) UV/Vis absorption spectra of DOX with different concentrations, (b) linear relationship between absorbance at 480 nm and concentration of DOX.

Fig. S9. (a) UV/Vis absorption spectra for different concentrations of 5-FU, (b) linear relationship between absorption intensity at 261 nm and concentration of 5-FU.



Fig. S10. Confocal fluorescence images of HeLa cells after incubation with UCMOFs for 2, 4 and 6 h at 37 °C; Upconversion luminescence (UCL) was collected by a green channel at 500 - 600 nm and a red channel at 600 - 700 nm, $\lambda_{ex} = 980$ nm, 500 mW.



Fig. S11. The three-dimensional confocal luminescence reconstructions of HeLa cells after incubation with UCMOFs for 4 h collected as a series along the Z optical axis; upconversion luminescence (UCL) was collected through a green UCL channel at 500 - 600 nm; $\lambda_{ex} = 980$ nm, 500 mW.