

Scheme S1. The chemical structure of PNBC-b-PEO and its photocleavage into poly(L-cysteine)-b-PEO (PLC-b-PEO) in the presence of UCNPs upon 980 nm laser irradiation.



Figure S1. TEM photograph (A), HRTEM (B), diagonal size histograms (C) and side size histograms (D) of UCNPs.



Figure S2. WAXD patterns (A) and emission spectra under 980 nm laser excitation (B) of UCNPs in THF (3.2 mg/mL).



Figure S3. (A) the mean size distribution of PNBC₅₆-b-PEO-UCNPs-5:1; (B) the time-

resolved UV-vis spectra of PNBC₅₆-b-PEO-UCNPs-5:1 upon different times of 980 nm irradiation; (C, D) the TEM photographs of PNBC₅₆-b-PEO-UCNPs-5:1.



Figure S4. Mean size distribution (A) and TEM photograph (B) of PNBC₉-b-PEO/UCNPs/DOX.



Figure S5. CLSM images of HeLa cells incubated with PNBC₉-b-PEO-UCNPs-DOX at different time intervals (the cell nucleus was stained by Hoechst 33342).



Figure S6. The cell viability of HeLa cells after 5 min or 10 min of the 980 nm NIR irradiation (5 W/cm²).