## Biodistribution, Excretion, and Toxicity of Polyethyleneimine Modified NaYF<sub>4</sub>:Yb,Er Upconversion Nanoparticles in Mice *via* Different Administration Routes

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Figure S1. EDX spectrum of PEI@UCNPs. The Si element comes from the silicon substrate.



**Figure S2**. (a) Stability tests of PEI@UCNPs in PBS, FBS, DMEM, and water dispersions (concentration: 500  $\mu$ g mL<sup>-1</sup>). (b) XRD patterns of PEI@UCNPs incubated with PBS, FBS, DMEM, and water solutions for 24 h, respectively.



**Figure S3**. PEI@UCNPs synthesized by adding different amount of water: (a,b) 2 mL of water , and (c,d) 3 mL of water.



**Figure S4**. PEI@UCNPs synthesized by different solvothermal reaction time: (a) 6, (b)12, (c) 24, and (d) 48 h.



**Figure S5**. PEI@UCNPs synthesized by adding different amount of precursors: (a) 0.4 mmol of  $Er^{3+}$ , (b) 0.1 mmol of  $Yb^{3+}$ , and (c) 0.4 mmol of  $Y^{3+}$ .



**Figure S6**. (a) TGA plot of PEI@UCNPs. Inset in (a) shows the magnified TGA plot of PEI@UCNPs. (b) Relative cell viability of BEL-7402 cells after incubation with different concentrations of PEI for 24 h.



**Figure S7.** Cell imaging under (a) bright field and (b) 980 nm laser irradiation after being incubated with PEI@UCNPs ( $10 \ \mu g \ mL^{-1}$ ) for 24 h.



**Figure S8**. Changes in body weight of mice *via i.p.* and *i.v.* injected with PEI@UCNPs within 30 days. Error bars represent standard deviation calculated from three mice.



**Figure S9**. Organ biodistribution of <sup>64</sup>Cu-NOTA-PEI@UCNPs at 24 h after *i.v.* injection, determined by  $\gamma$ -counter (n=3). (% ID/g organ)