## Electronic Supplementary Information(ESI) for:

## Upconversion Nanoparticle-Based Fluorescence Resonance Energy Transfer System for Effective Sensing Caspase-3 Activity

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## Contents

Additional figures S1-S8



**Fig. S1** The UCL spectrum of NaGdF<sub>4</sub>: Yb<sup>3+</sup>, Er<sup>3+</sup>@NaGdF<sub>4</sub> UCNPs (0.3 mg mL<sup>-1</sup> in cyclohexane, black line) and absorption spectrum of RB-pCasCPP (0.5 mg mL<sup>-1</sup> in PBS, blue line).



**Fig. S2** TEM micrograph of NaGdF<sub>4</sub>: Yb<sup>3+</sup>, Er<sup>3+</sup>UCNPs (a), NaGdF<sub>4</sub>: Yb<sup>3+</sup>, Er<sup>3+</sup>@NaGdF<sub>4</sub> UCNPs (b) and NaGdF<sub>4</sub>: Yb<sup>3+</sup>, Er<sup>3+</sup>@NaGdF<sub>4</sub> UCNP@RB-pCasCPP (c).



**Fig. S3** X-ray diffraction patterns of NaGdF<sub>4</sub>: Yb<sup>3+</sup>, Er<sup>3+</sup> UCNPs (a) and NaGdF<sub>4</sub>: Yb<sup>3+</sup>, Er<sup>3+</sup>@NaGdF<sub>4</sub> UCNPs (b).



**Fig. S4** XPS survey spectra of oleate capped NaGdF<sub>4</sub>: Yb<sup>3+</sup>, Er<sup>3+</sup>@NaGdF<sub>4</sub> UCNPs (a) and NaGdF<sub>4</sub>: Yb<sup>3+</sup>, Er<sup>3+</sup>@NaGdF<sub>4</sub> UCNP@RB-pCasCPP (b), and P 2p(c) and N 1s (d) XPS spectra of NaGdF<sub>4</sub>: Yb<sup>3+</sup>, Er<sup>3+</sup>@NaGdF<sub>4</sub> UCNP@RB-pCasCPP.



**Fig. S5** FTIR spectra of oleate capped NaGdF<sub>4</sub>: Yb<sup>3+</sup>, Er<sup>3+</sup>@NaGdF<sub>4</sub> UCNPs (black line) and NaGdF<sub>4</sub>: Yb<sup>3+</sup>, Er<sup>3+</sup>@NaGdF<sub>4</sub> UCNP@RB-pCasCPP (blue line).



Fig. S6 DLS size distribution of NaGdF<sub>4</sub>: Yb<sup>3+</sup>, Er<sup>3+</sup>@NaGdF<sub>4</sub> UCNP@RB-pCasCPP.



**Fig. S7** Digital images of NaGdF<sub>4</sub>: Yb<sup>3+</sup>, Er<sup>3+</sup>@NaGdF<sub>4</sub> UCNP@RB-pCasCPP redispersed in different media after 12 h.



Fig. S8 Cell viability as a function of the concentration of NaGdF<sub>4</sub>: Yb<sup>3+</sup>,  $Er^{3+}$ @NaGdF<sub>4</sub> UCNP@RB-pCasCPP.