Experimental Details

Synthesis of β-$NaYF_4$: 20% Yb, 2% Er nanoparticles

All chemicals were of analytical grade and used without further purification. In a typical synthesis, $YCl_3 \cdot 6H_2O$ (0.78 mmol), $YbCl_3 \cdot 6H_2O$ (0.20mmol), $ErCl_3 \cdot 6H_2O$ (0.02 mmol) were added to a 100 mL three-necked flask containing oleic acid (6 mL) and 1-octadecene (15 mL). The solution was stirred and heated to 150 °C under vacuum for 30 min to get a homogenous solution and then cooled down to room temperature. A solution of NH_4F (4 mmol) and NaOH (2.5 mmol) dissolved in methanol (10 mL) was added to the flask and stirred for 30 min at 50 °C and then heated slowly to 70 °C until all the methanol evaporated. Subsequently, the solution was heated to 300 °C for 1.5 h under nitrogen protection and then cooled down to room temperature. The nanoparticles were precipitated by adding ethanol, centrifuged and washed with ethanol for three times. The isolated nanoparticles were redispersed in THF (10 mL) for further experiments.

Synthesis of RB-hydrazide. 6 mL (excess) hydrazine hydrate (80 %) was added dropwise to a solution of rhodamine B (2.40 g, 5 mmol) in absolute ethanol (30 mL) with vigorous stirring at room temperature. After the addition, the stirred mixture was refluxed in an oil bath for 12 h. The solution changed from dark purple to light orange and became transparent. Then the solvent was removed under reduced pressure, a

large amount of water was added, the resulting precipitate was filtered and washed by water. After drying in vacuo, the reaction afforded a light pink solid (1.84 g,yield 81 %).

Characterization The X-ray diffraction (XRD) patterns of UCNPs were performed on a Rigaku D/Max-Ra x-ray diffractometer using a Cu target radiation source (λ = 0.15418 nm). The transmission electron microscopy (TEM) images were recorded with a JEM-2010 transmission election microscope made by the Japanese JEOL Company. Dilute colloids of the nanoparticles dispersed in THF were drop-cast on thin, carbon formvar-coated copper grids and air dried before imaging. Solid-state ^{29}Si MAS NMR spectra were recorded at 79.46 MHz, using a Bruker Avance 400 spectrometer. Optical measurements were acquired on a Hitachi F-4500 fluorescence spectrophotometer under the excitation of a 980 nm laser diode. All spectrophotometic spectra were performed with a dispersion of sample in THF and then brought into a quartz cell for measurement. Emission lifetimes were measured at 539 nm (from Er^{3+}) with a Lecroy Wave Runner 6100 Digital Oscilloscope (1GHz) using a tunable laser as the excitation source (Continuum Sunlite OPO).

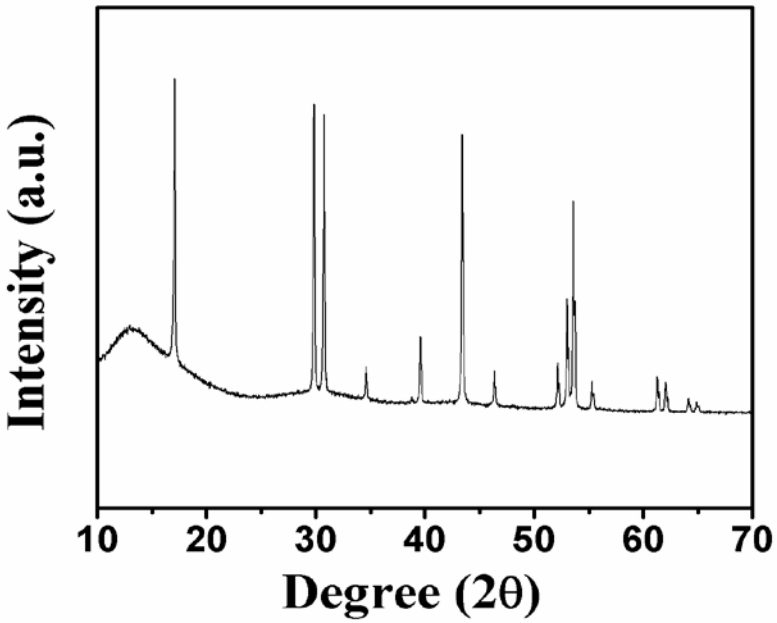

Fig. S1 XRD pattern of sample $NaYF_4$: Yb^{3+} / Er^{3+}.

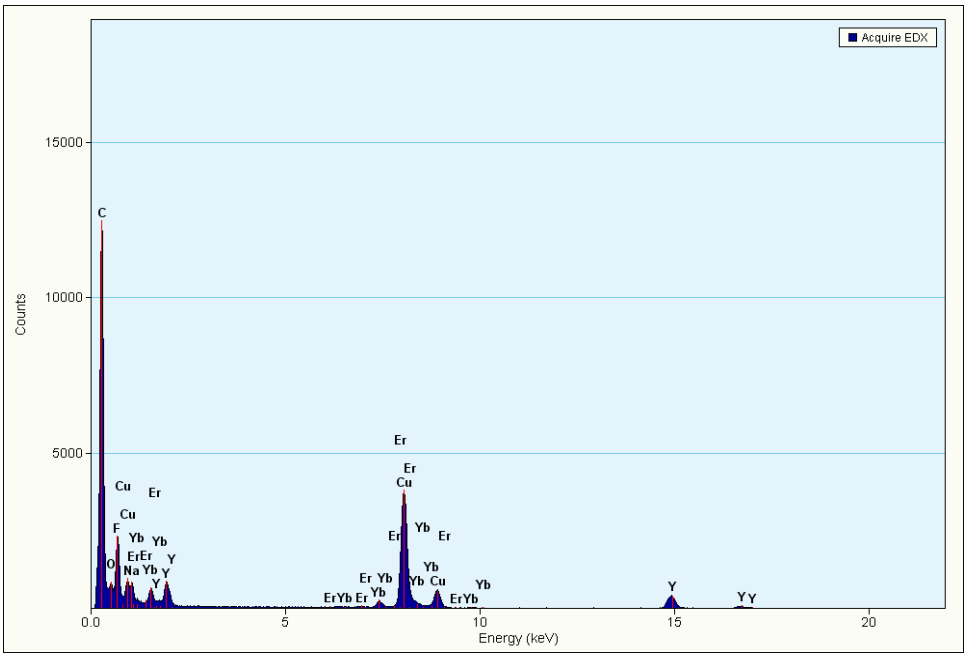

Fig. S2 EDX spectra of $NaYF_4$: Yb^{3+} / Er^{3+} nanocrystals.

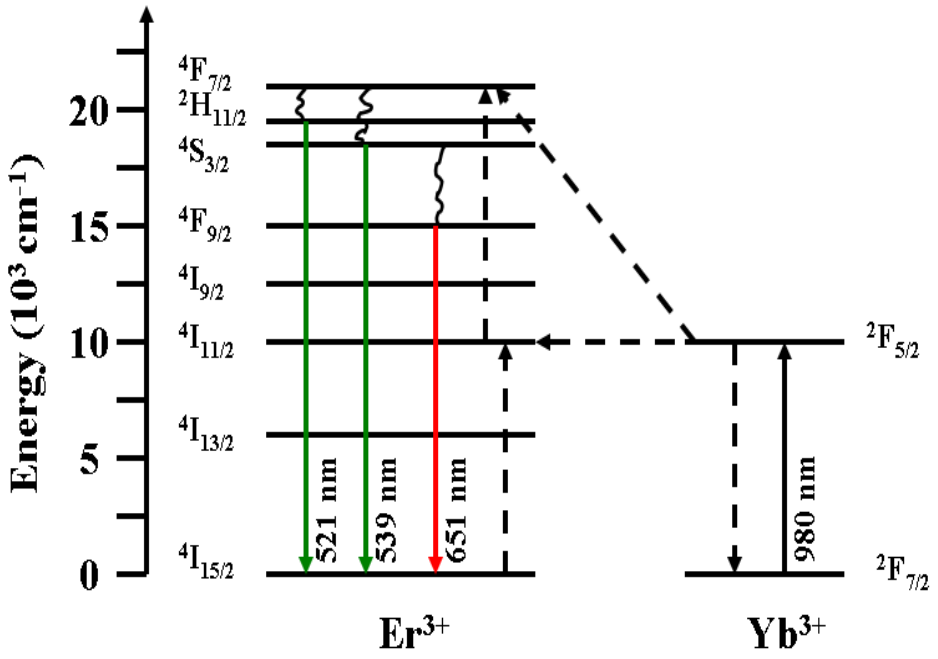

Fig. S3 Energy transfer upconversion scheme of NIR 980 nm radiation to populate $^2H_{11/2}$ + $^4S_{3/2}$ and $^4F_{9/2}$ levels of Er^{3+} in $NaYF_4$: Yb^{3+} / Er^{3+} nanocrystals.

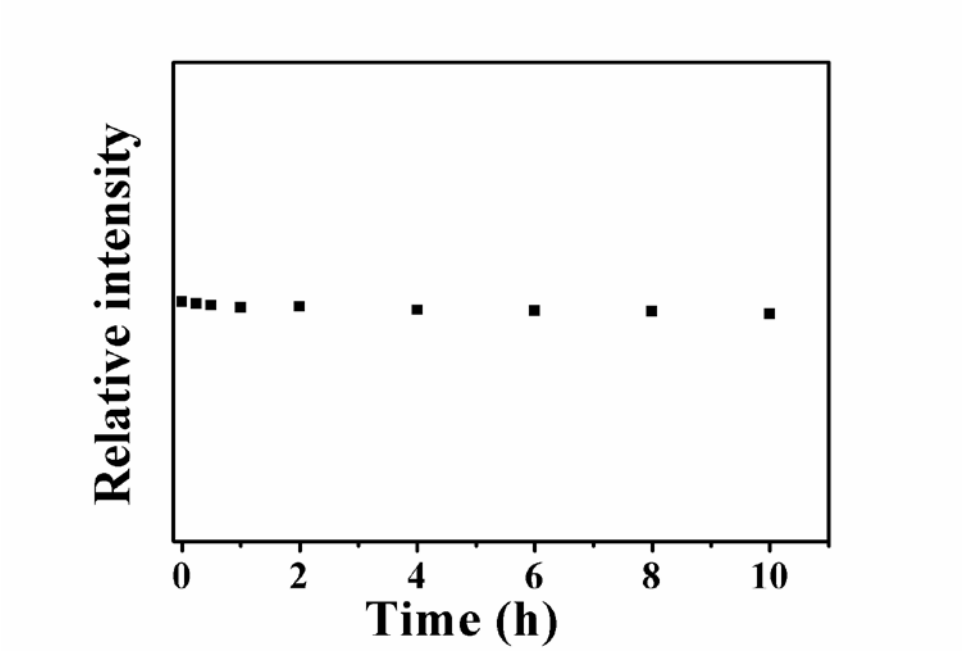

Fig. S4 The relative fluorescent intensity of RB-hydrazide in the presence of Cu(II) (2×10^{-6} M) for different times in buffer (pH = 7) with CH_3CN .

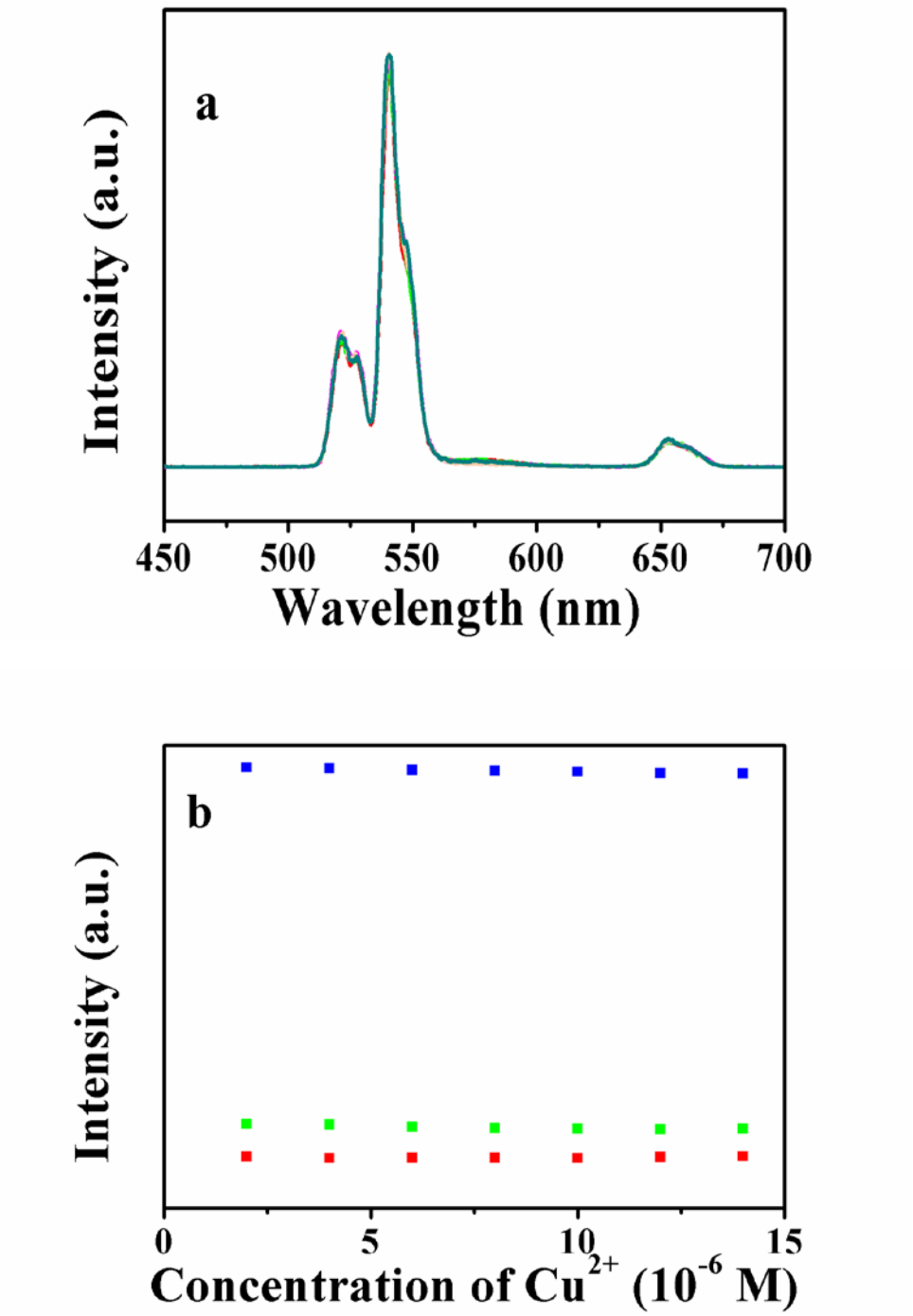

Fig. S5 (a) Luminescence emission of UCNPs upon addition of RB-hydrazide (5×10^{-6} M to 2.5×10^{-5} M) when no Cu(II); (b) the same for Cu(II) (10^{-5} to 10^{-6} M) when no RB-hydrazide.(red: λ = 651 nm, green: λ = 521 nm, blue: λ = 539 nm)

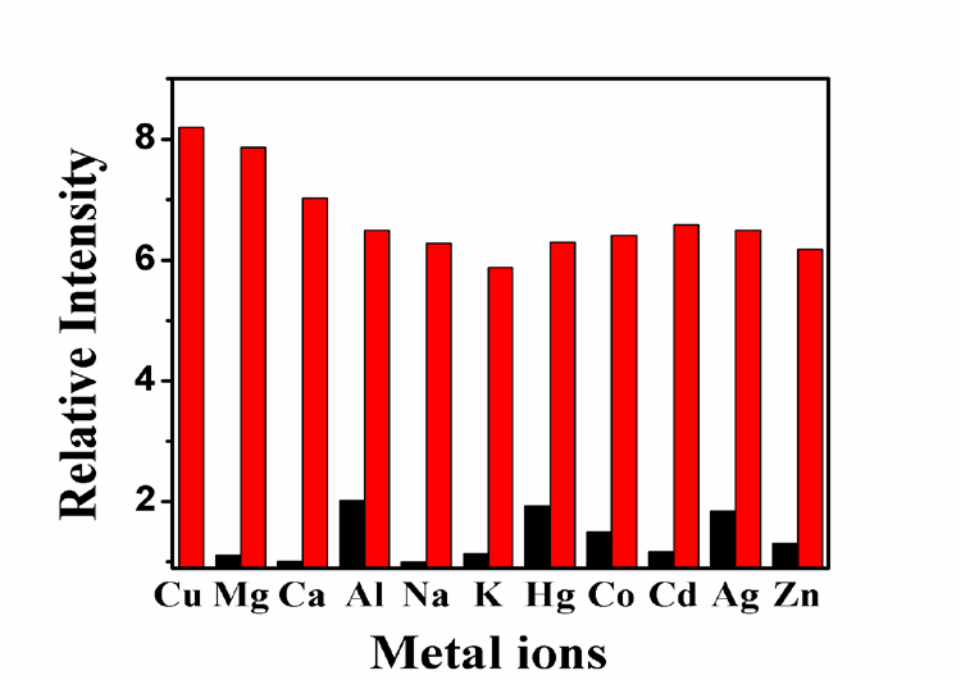

Fig. S6 The relative fluorescence intensity of RB-hydrazide in the presence of various interfering ions (black bars) and coexistence with Cu(II) (red bars) in aqueous solution. λex = 980 nm and λem = 578 nm.