

# Supporting Information

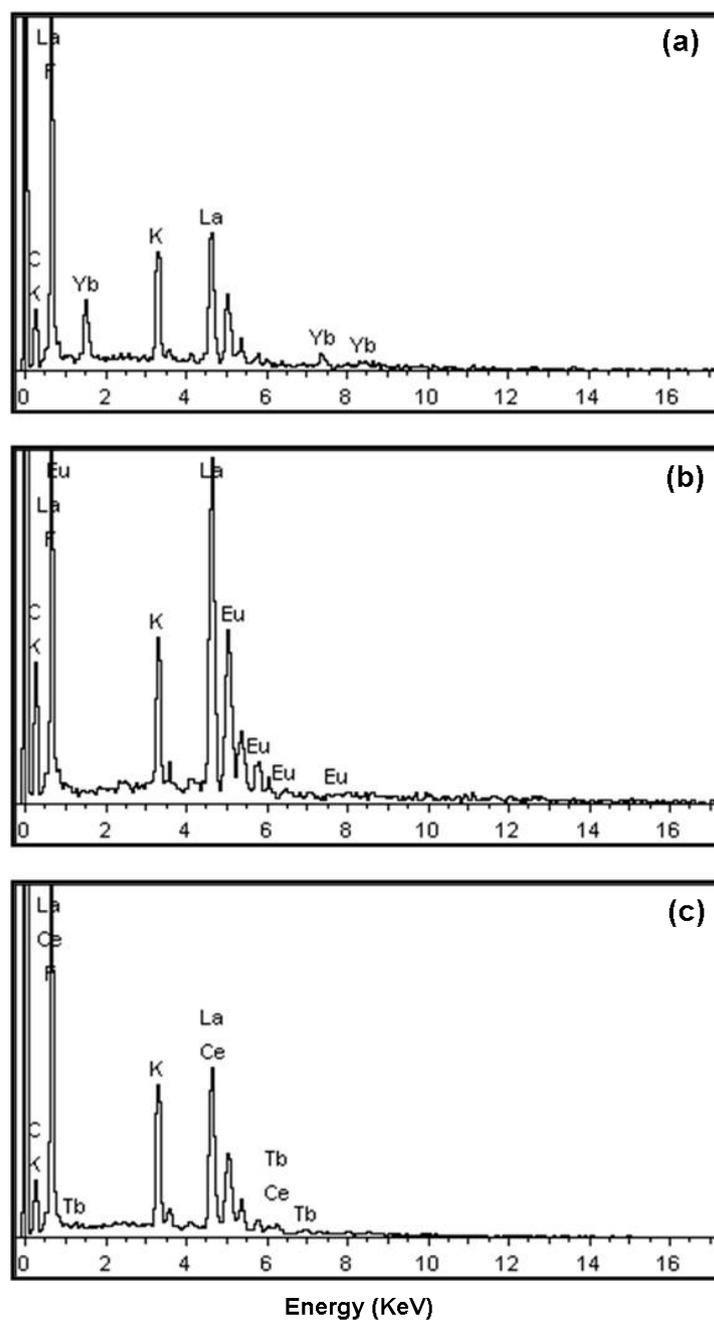
## Controlled synthesis and optical spectroscopy of lanthanide-doped KLaF<sub>4</sub> nanocrystals

Rui Liu,<sup>ab</sup> Datao Tu,<sup>a</sup> Yongsheng Liu,<sup>a</sup> Haomiao Zhu,<sup>a</sup> Renfu Li,<sup>a</sup> Wei Zheng,<sup>ab</sup> En Ma,<sup>a</sup> and  
Xueyuan Chen<sup>\*a</sup>

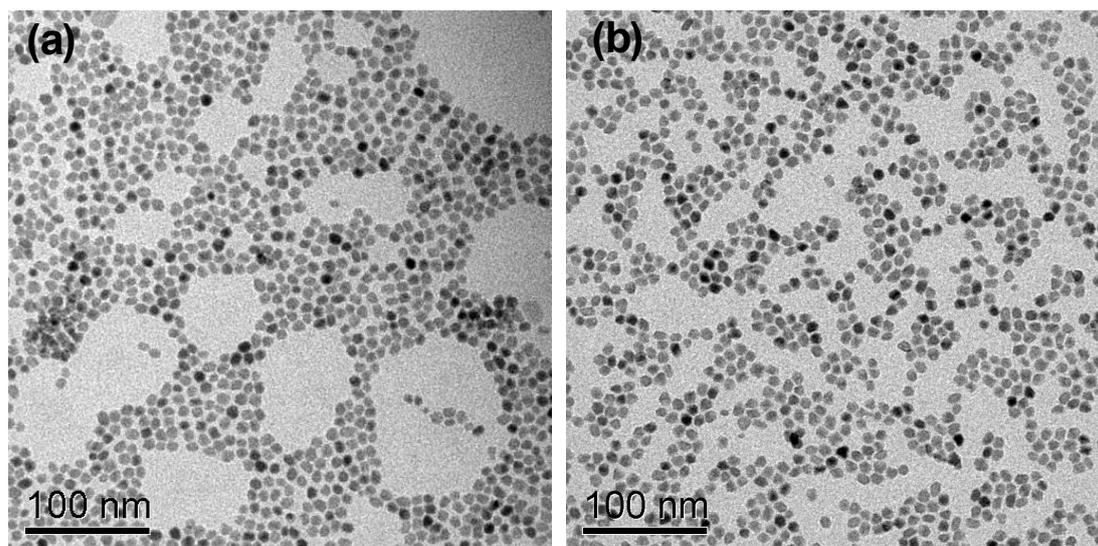
<sup>a</sup> *Key Laboratory of Optoelectronic Materials Chemistry and Physics, Fujian Institute of Research on the Structure of Matter, Chinese Academy of Sciences, Fuzhou, Fujian 350002, China.*

<sup>b</sup> *Graduate University of the Chinese Academy of Sciences, Beijing 100049, China.*

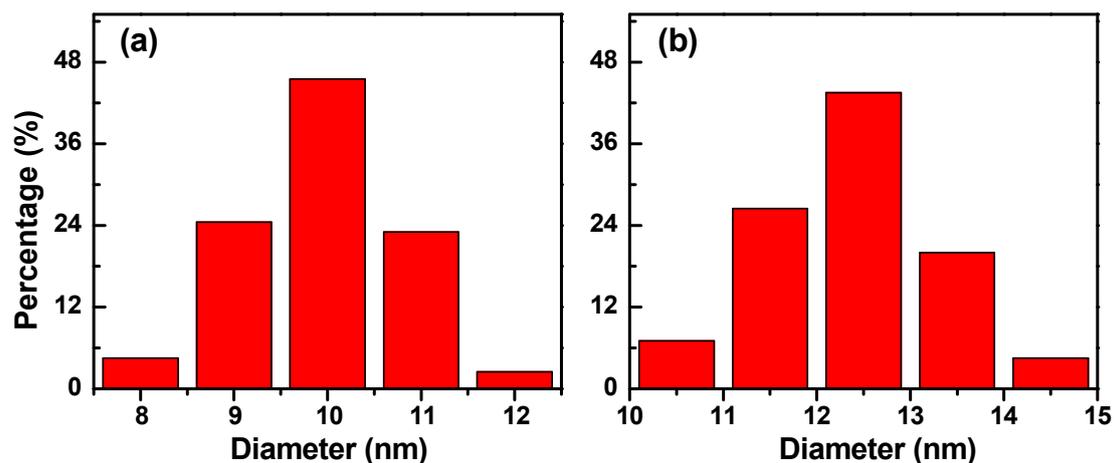
\* Corresponding author, E-mail: [xchen@fjirsm.ac.cn](mailto:xchen@fjirsm.ac.cn)



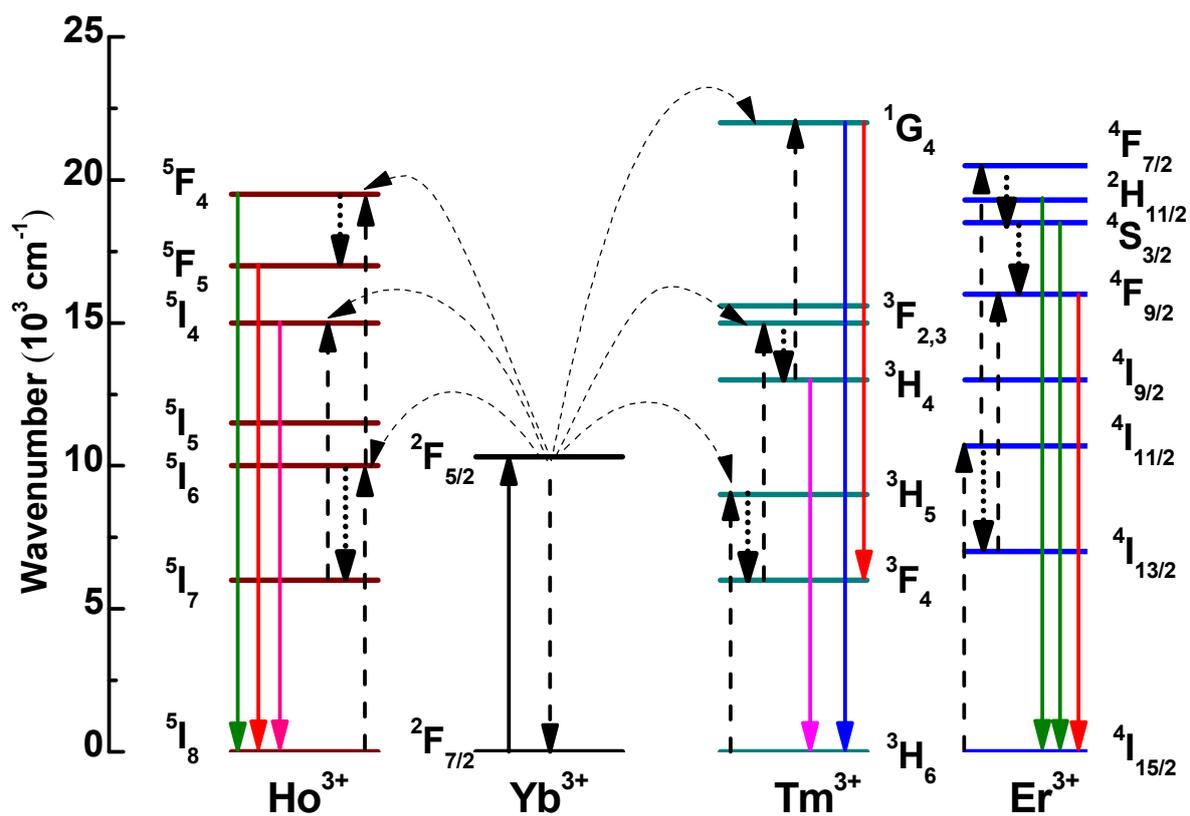
**Fig. S1** Energy dispersive X-ray spectrum (EDS) analysis of (a)  $\text{KLaF}_4:0.02\text{Er}^{3+}/0.18\text{Yb}^{3+}$ , (b)  $\text{KLaF}_4:0.05\text{Eu}^{3+}$  and (c)  $\text{KLaF}_4:0.05\text{Ce}^{3+}/0.05\text{Tb}^{3+}$  nanocrystals (NCs), revealing successfully doping of  $\text{Er}^{3+}/\text{Yb}^{3+}$ ,  $\text{Eu}^{3+}$  or  $\text{Ce}^{3+}/\text{Tb}^{3+}$  into  $\text{KLaF}_4$  host.



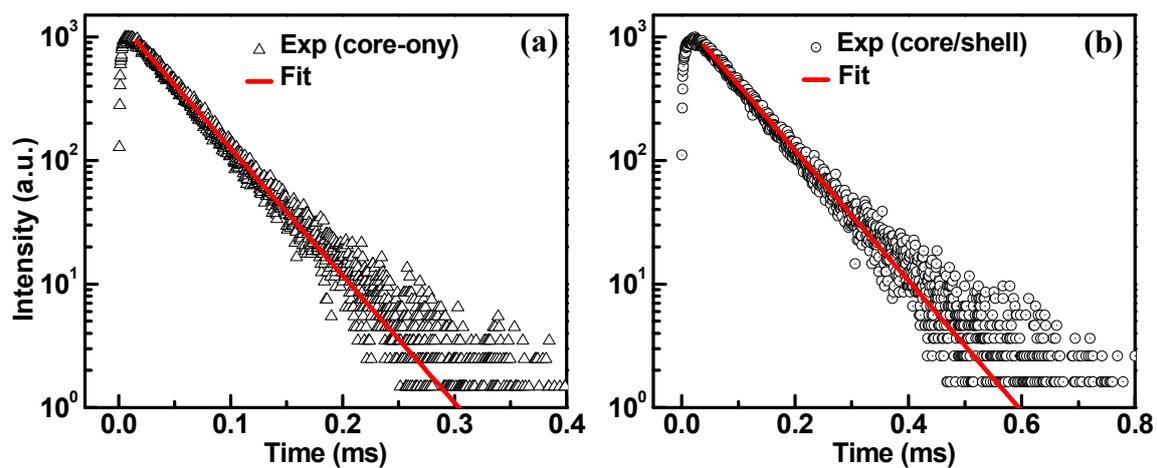
**Fig. S2** TEM images of (a) KLaF<sub>4</sub>:0.05Eu<sup>3+</sup> and (b) KLaF<sub>4</sub>:0.05Ce<sup>3+</sup>/0.05Tb<sup>3+</sup> NCs.



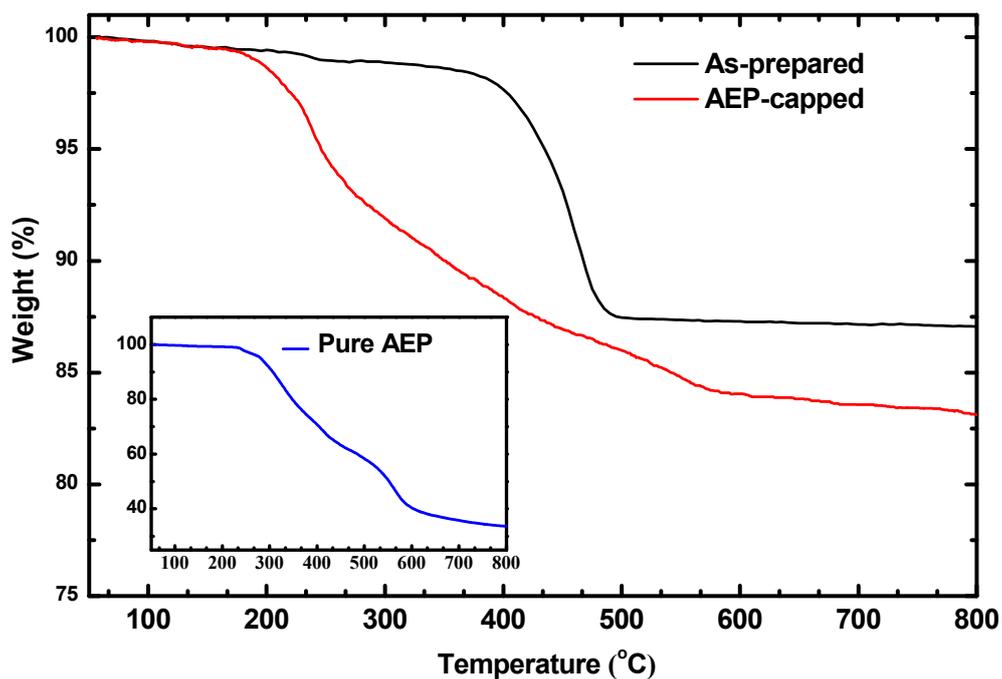
**Fig. S3** Histograms of size distribution of (a)  $\text{KLaF}_4:0.02\text{Er}^{3+}/0.18\text{Yb}^{3+}$  core-only and (b)  $\text{KLaF}_4:0.02\text{Er}^{3+}/0.18\text{Yb}^{3+}@\text{KLaF}_4$  core/shell NCs, estimated from corresponding TEM images of Figs. 2a-b by randomly counting 200 nanoparticles. The average diameter was increased from 9.9 nm in core-only NCs to 12.4 nm in core/shell NCs, which demonstrates a thin  $\text{KLaF}_4$  layer ( $\sim 1.3$  nm) coated on the inner core.



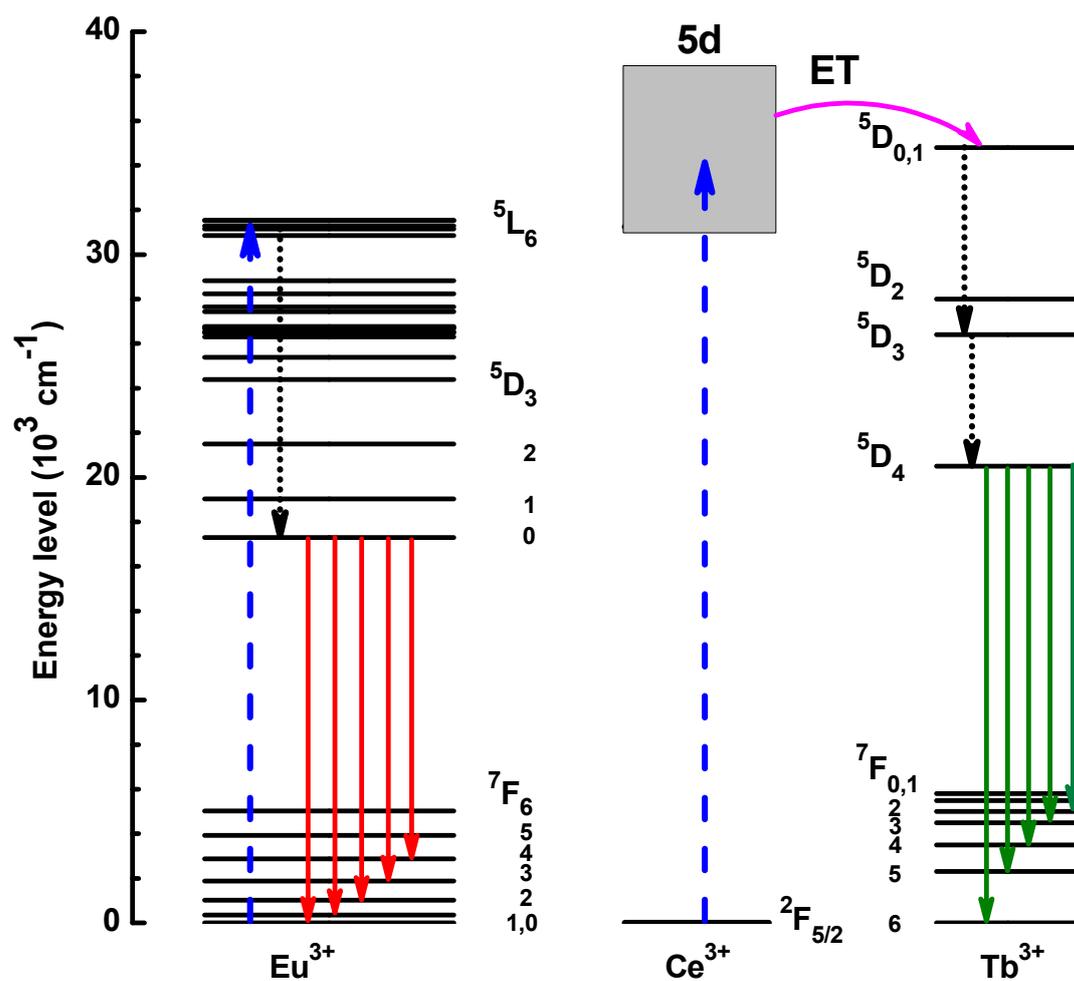
**Fig. S4** Schematic energy level diagrams showing typical upconversion (UC) processes for (a) Ho<sup>3+</sup>, (b) Tm<sup>3+</sup> and (c) Er<sup>3+</sup> via the sensitization of Yb<sup>3+</sup>. The dashed, dotted and full arrows represent the excitation, nonradiative relaxation, and emission processes, respectively..



**Fig. S5** UC luminescence decay curves of (a)  $\text{KLaF}_4:0.02\text{Er}^{3+}/0.18\text{Yb}^{3+}$  core-only and (b)  $\text{KLaF}_4:0.02\text{Er}^{3+}/0.18\text{Yb}^{3+}@\text{KLaF}_4$  core/shell NCs by monitoring the  ${}^2\text{H}_{11/2} \rightarrow {}^4\text{I}_{15/2}$  transition of  $\text{Er}^{3+}$  at 521 nm upon excitation at 980 nm. By fitting with a single exponential function, the UC lifetimes of  ${}^2\text{H}_{11/2}$  in core-only and core/shell NCs were determined to be 41 and 84  $\mu\text{s}$ , respectively.



**Fig. S6** Thermogravimetric analysis (TGA) of as-prepared and AEP-capped KLaF<sub>4</sub> NCs, respectively. The inset shows the TGA curve of pure AEP. The major stages of weight loss of as-prepared and AEP-capped KLaF<sub>4</sub> NCs are different. Similar TGA behaviors observed for AEP-capped NCs and pure AEP indicate the AEP capping on the surface of the NCs after ligand exchange.



**Fig. S7** Schematic energy level diagrams showing downconversion processes for (a)  $\text{Eu}^{3+}$  and (b)  $\text{Ce}^{3+}/\text{Tb}^{3+}$  ions in  $\text{KLaF}_4$  NCs. The dashed, dotted and full arrows represent the excitation, nonradiative relaxation, and emission processes, respectively.