Supporting Information

A general strategy for the synthesis of rare earth fluoride nano

(micro)crystals

Songtao Liu,^a Gejihu De, ^{abc*} Xian Wang, ^a Yueshan Xu, ^a Yuanyuan Liu, ^a Jianxun Wang, ^a Chunyan Cheng. ^a

^a College of Chemistry and Environment Science, Inner Mongolia Normal University, Hohhot 010022, People's Republic of China
^b Physics and Chemistry of Functional Materials, Inner Mongolia Normal University, Hohhot 010022, People's Republic of China
^c State Key Laboratory on Integrated Optoelectronics, Jilin University, Jilin 130012, People's Republic of China

E-mail: degjh@imnu.edu.cn

Samples	Structure	Excited level	Wavelength (nm)	Lifetime (µs)
KY ₃ F ₁₀	Tetragonal	⁴ S _{3/2}	544	39.31
KYF ₄	Hexagonal	⁴ S _{3/2}	544	733.01
K ₂ YF ₅	Orthorhombic	⁴ S _{3/2}	544	996.67
KY ₃ F ₁₀	Tetragonal	⁴ F _{9/2}	655	58.88
KYF ₄	Hexagonal	⁴ F _{9/2}	655	1045.69
K_2YF_5	Orthorhombic	⁴ F _{9/2}	655	1055.88

 $STable \ 1 \ Crystal \ structure, \ Excited \ level, \ Monitoring \ wavelength \ and \ Lifetime \ of \ as-obtained \ KYF_4: 20\%Yb^{3+}/2\%Er^{3+} \ nanocrystals.$



Figure S1: XRD data of the products obtained at different molar ratios KF-to-RE: (a) 2:1; (b) 2.25:1; (c) 2.5:1; (d) 3:1; (e) 3.5:1; (f) 4:1. Other synthesis conditions: 45 mL OA/15 mL HA, $V_{H20}/V_{CF3COOH}=9/2$, 230 °C for 48 h. Vertical black, red and blue lines depict the XRD references for hexagonal LaF₃ (JCPDS No: 72-1435), cubic KLaF₄ (JCPDS No: 75-2020), and hexagonal (KLaF₄)_{1.5} (JCPDS No: 75-1927), respectively.



Figure S2: XRD patterns of the KGdF₄ nanocrystals synthesized at different molar ratios KF-to-RE: (a) 3:1; (b) 3.75:1; (c) 12:1; (d) 18:1; (e) 24:1;. Other synthesis conditions: 45 mL OA/15 mL HA, $V_{H2O}/V_{CF3COOH}=9/2$, 230 °C for 48 h. The standard data of orthorhombic of K₂GdF₅ (JCPDS No: 77-1924), KGdF₄ (JCPDS No: 33-1007) and K_{0.265}Gd_{0.735}F_{2.47} (JCPDS No: 80-0511) are also shown.



Figure S3: XRD data of the products obtained at different molar ratios NaF-to-RE: (a) 1:1; (b) 1.5:1; (c) 2:1; (d) 3:1. Other synthesis conditions: 45 mL OA/15 mL HA, $V_{H2O}/V_{CF3COOH}=9/2$, 180 °C for 48 h. Vertical black, red and black lines depict the XRD references for hexagonal NaYF₄ (JCPDS No: 16-0334), cubic NaYF₄ (JCPDS No: 39-0725), respectively.



Figure S4: XRD data of the products obtained at different molar ratios LiF-to-RE: (a) 1:1; (b) 2:1; (c) 3:1; (d) 4:1. Other synthesis conditions: 45 mL OA/15 mL HA, $V_{H2O}/V_{CF3COOH}=9/2$, 180 °C for 48 h. Vertical black, red and blue lines depict the XRD references for tetragonal LiYF₄ (JCPDS No: 17-0874), orthorhombic YF₃ (JCPDS No: 32-1431), respectively.



Figure S5: TEM image and particle size distribution of KYF_4 nanocrystals obtained in 45 mL OA/15 mL HA, $V_{H2O}/V_{CF3COOH}=9/2$, 250 °C for 48 h at molar ratio of KF/RE=2:5.75.



Figure S6: TEM image and particle size distribution of LaF₃ nanocrystals obtained in 45 mL OA/15 mL HA, $V_{H2O}/V_{CF3COOH}=9/2$, 230 °C for 48 h at molar ratio of KF/RE=2:1.



Figure S7: TEM and HRTEM images of NaYF₄ (cubic) and NaYF₄ (hexagonal) Nanocrystals obtained in 45 mL OA/15 mL HA, V_{H20}/V_{CF3COOH}=9/2, 180 °C for 48 h at different molar ratios NaF-to-RE: (a-c): 1:1; (d-f): 10:1.



Figure S8: TEM and HRTEM images of YF_3 and $LiYF_4$ nano (micro)crystals obtained in 45 mL OA/15 mL HA, $V_{H2O}/V_{CF3COOH}=9/2$, 180 °C for 48 h at different molar ratios LiF-to-RE: (a-c): 1:1; (d-f): 4:1.



Figure S9: The energy level diagram of Yb^{3+} sensitized Er^{3+} and upconversion mechanisms under near-infrared (980 nm) excitation.