One-step synthesis of water-soluble hexagonal NaScF$_4$:Yb/Er nanocrystals with intense red emission

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**Fig. S1** EDS pattern of the as-synthesized NaScF₄:Yb/Er UCNCs.

**Fig. S2** XRD patterns of NaScF₄:Yb/Er NCs obtained at different reaction times (a) 30 min; (b) 1 h; (c) 1.5 h; (d) 2 h; (e) 4 h; the standard peaks of (f) hexagonal NaScF₄ and (g) orthorhombic ScF₃, respectively.

**Fig. S2** shows the XRD patterns of the products prepared at different reaction times. The XRD patterns of the sample obtained at the initial stage (30 min) revealed the presence of impurities. The impure peaks at 2θ=22.2 degree mainly correspond to the orthorhombic ScF₃. When the reaction time extended, impure peaks gradually disappeared. The pure hexagonal phase (NaScF₄) was obtained at 2 h. The diffraction intensity became stronger as reaction time extended, indicating that crystallinity increased. Therefore, the time-dependent experiments demonstrated that hexagonal NaScF₄:Yb/Er NCs had been synthesized in 2 hours.
**Fig. S3** XRD patterns of Na₅ScF₈₊ₓ:Yb³⁺/Er³⁺ (20/2 mol %) UCNCs obtained under (a) Na:Sc=3:1 (b) Na:Sc=2:1 and (c) Na:Sc=1:1, the standard peaks in pure hexagonal NaScF₄ (d) and monoclinic (e) Na₃ScF₆ phases, respectively.

**Fig. S4** (a) SEM image and (b) the corresponding histogram of NaYF₄:Yb³⁺/Er³⁺ (20/2 mol%) NCs. The particle size and dopant concentration of the NaYF₄ and NaScF₄ NCs were similar.

**Fig. S5** UC energy transfer mechanisms of the as-prepared NaScF₄:Yb/Er UCNCs.
Fig. S6 XRD patterns of NaScF$_4$:Yb/Er (m/2 mol %) NCs doped with different Yb$^{3+}$ ion concentrations (a) m = 20, (b) m = 30, (c) m = 40, (d) m = 50, (e) m = 60 and the standard peaks of hexagonal NaScF$_4$.

When NaScF$_4$:Er/Yb NCs doped with various level of Yb$^{3+}$, the XRD patterns (Fig. S6) match well with that of standard hexagonal structured NaScF$_4$. Compared with standard peaks of hexagonal NaScF$_4$, the slight shifts of the peaks were caused by the incorporation of Yb$^{3+}$ ions into the NaScF$_4$ NCs.

Fig. S7 XRD patterns of NaScF$_4$:Yb/Er (20/n mol %) NCs doped with different Er$^{3+}$ ion concentrations (a) n = 2, (b) n = 4, (c) n = 6, (d) n = 8, the standard peaks of (e) hexagonal NaScF$_4$ and (f) orthorhombic ScF$_3$, respectively.

When the concentration of Er$^{3+}$ ion extended to 4 mol % or more, impure peaks indexed to the orthorhombic ScF$_3$ would appear (Fig. S6). Therefore, the appropriate dopant concentrations of Er$^{3+}$ is about 2 mol% in the system. No impure peaks occurred in NaScF$_4$:Er/Yb NCs when doping concentration of Yb$^{3+}$ was gradually enhanced, which was probably because of the similar radius between Sc$^{3+}$ and Yb$^{3+}$ ions.
**Fig. S8** UC luminescence spectra of NaScF₄:Yb/Er NCs doped with different Er³⁺ ion concentrations in the range of 2–8 mol%.

**Fig. S9** STEM image of NaScF₄:Yb/Er (40/2%) @ NaScF₄:Yb (10%) NCs (a) and EDS line scan (b) showing the signal intensity variation of Yb across a randomly selected core–shell NCs. The Yb content at particle edge is lower than that in the interior, which is very consistent with the designed core–shell structure.