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Preparation of rare-earth doped NaYF₄ luminescent nanoparticles by high-energy ball milling process

Electronic Supplementary Information

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Figure S1 XRD patterns KTb_3F_{10} crystal after different "wet" mechanical milling treatment; the positions of the Bragg peaks for the KTb_3F_{10} (*ICDD PDF* 01-074-2165, sp. gr. *Fm* $\overline{3}m$) and KTb_2F_7 (*ICDD PDF* 00-057-0574, sp. gr. *Fm* $\overline{3}m$) are indicated.



Figure S2 XRD patterns of the "wet" milled α -NaYF₄ crystals at different duration of mechanical treatment. The positions of the Bragg peaks for sp. gr. *Fm* $\overline{3}m$ (*ICDD PDF* 01-077-2042) and *P*6₃/*m* (*ICDD PDF* 00-064-0156) are indicated.

FTIR analysis

FTIR spectra of the oleic acid, «dry»-milled β -NaYF₄ and oleate-capped 16 h-milled β -NaYF₄ NPs were measured on a Bruker Alpha FTIR spectrometer using the KBr method. The spectra in transmission mode were recorded in a wavenumber range of 4000-500 cm⁻¹.



Figure S3. FTIR spectra of (a) pure oleic acid, (b) β -NaYF₄ NPs, produced *via* 16 h oleatemediated milling, and (c) «dry»-milled (oleate-free) β -NaYF₄ NPs.

The signals, observed for pure oleic acid, are corresponded to the characteristic vibrations of various functional groups: $-CH_{val}$ and $-COO-H_{val}$ (3150-2800 cm⁻¹), -C=O (1711 cm⁻¹), $-CH_{def}$ and $-COO_{def}$ (1480-1400 cm⁻¹). In the FTIR spectrum of β -NaYF₄ NPs, fabricated by 16 h oleate-mediated milling, analogous peaks are observed, confirming the functionalization of the NPs surface by oleic acid molecules.