

## SUPPORTING INFORMATION

### **Upconversion luminescence in sub-10 nm $\beta$ -NaGdF<sub>4</sub>: Yb<sup>3+</sup>, Er<sup>3+</sup> nanoparticles: An improved synthesis in anhydrous ionic liquids**

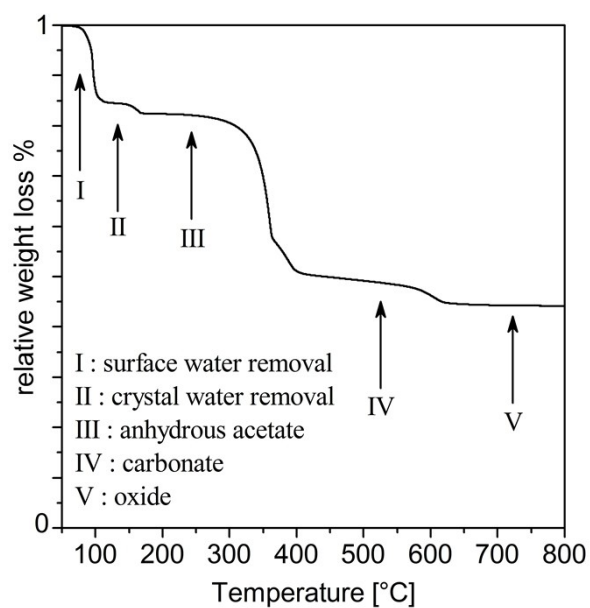
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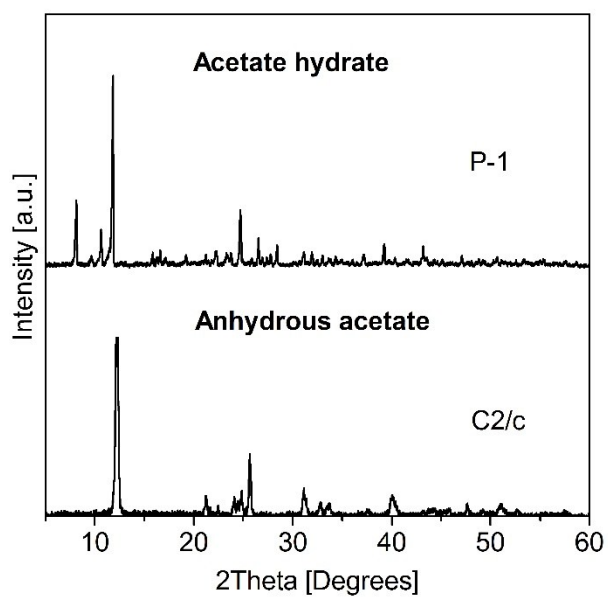
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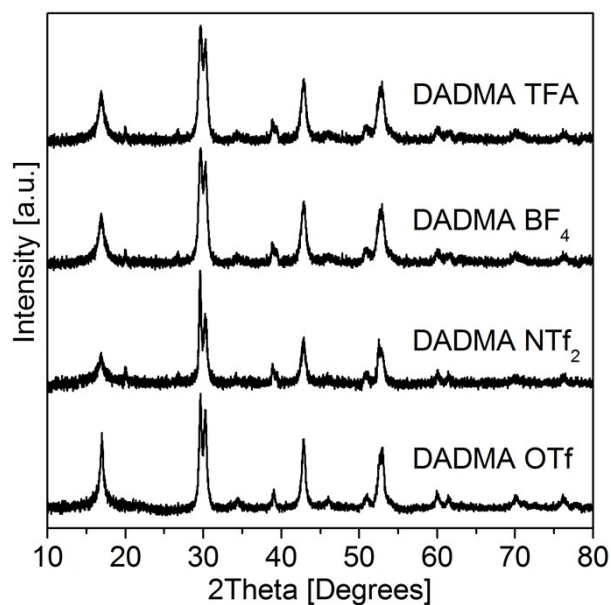
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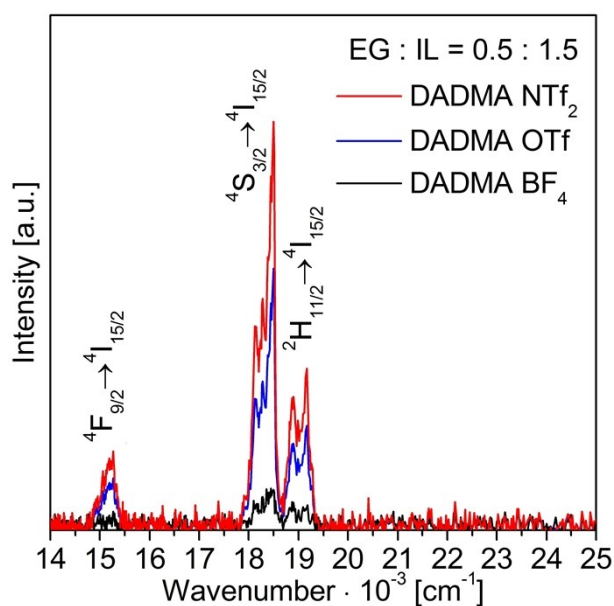
**Fig. S1:** Thermogravimetric analysis of the rare earth acetate hydrate precursor  $\text{RE}(\text{OAc})_3 \cdot \text{aq.}$  with  $\text{RE} = \text{Gd}_{0.8}, \text{Er}_{0.02}, \text{Yb}_{0.18}$ . The sample was heated by 5 K/min in a  $\text{N}_2$  flow of 20 ml/min.



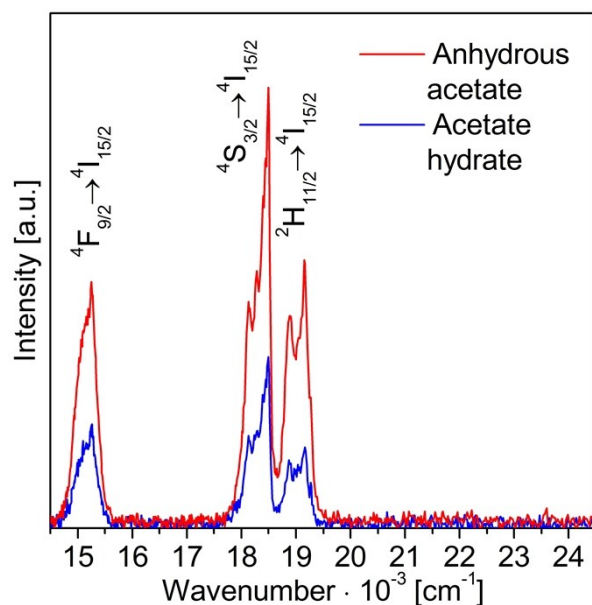
**Fig. S2:** Powder X-ray diffraction (XRD) patterns of the rare earth acetate hydrate  $\text{RE}(\text{OAc})_3 \cdot \text{aq.}$  (top) and the anhydrous rare earth acetate  $\text{RE}(\text{OAc})_3$  (bottom) with  $\text{RE} = \text{Gd}_{0.8}, \text{Er}_{0.02}, \text{Yb}_{0.18}$ . The space groups are specified in the figure and structures discussed in Ref. S1.



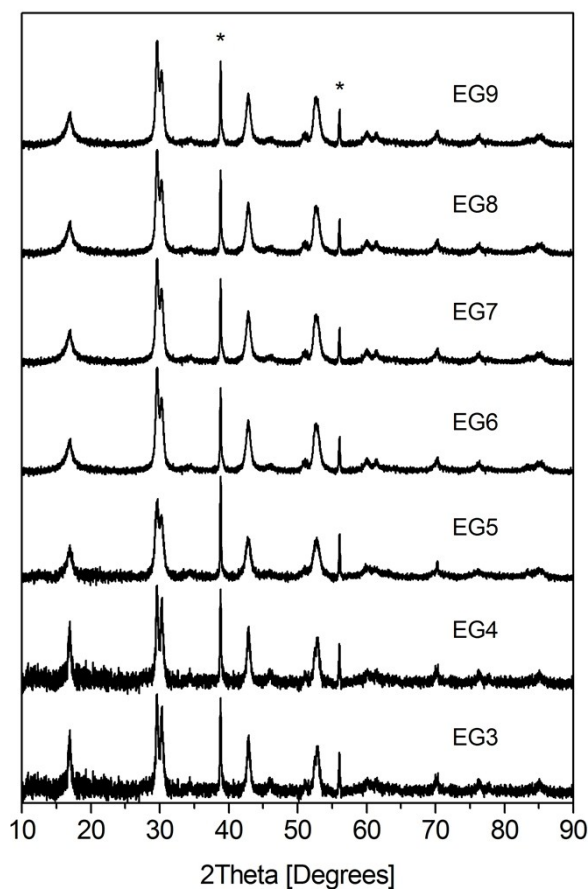
**Fig. S3:** Powder XRD patterns of nanocrystalline  $\beta$ -NaGdF<sub>4</sub>: 18% Yb<sup>3+</sup>, 2% Er<sup>3+</sup> samples synthesized in 0.5:1.5 vol. ethylene glycol (EG) / ionic liquid (IL) solutions with IL = diallyldimethylammonium (DADMA) trifluoroacetate (TFA), DADMA BF<sub>4</sub>, DADMA bis(trifluoromethanesulfonyl)amide (NTf<sub>2</sub>), and DADMA trifluoromethanesulfonate (OTf). Samples were synthesised from 60 mg RE(AcO)<sub>3</sub>, 20 mg NaCl, and 80 mg NH<sub>4</sub>F at 120°C for 30 min.



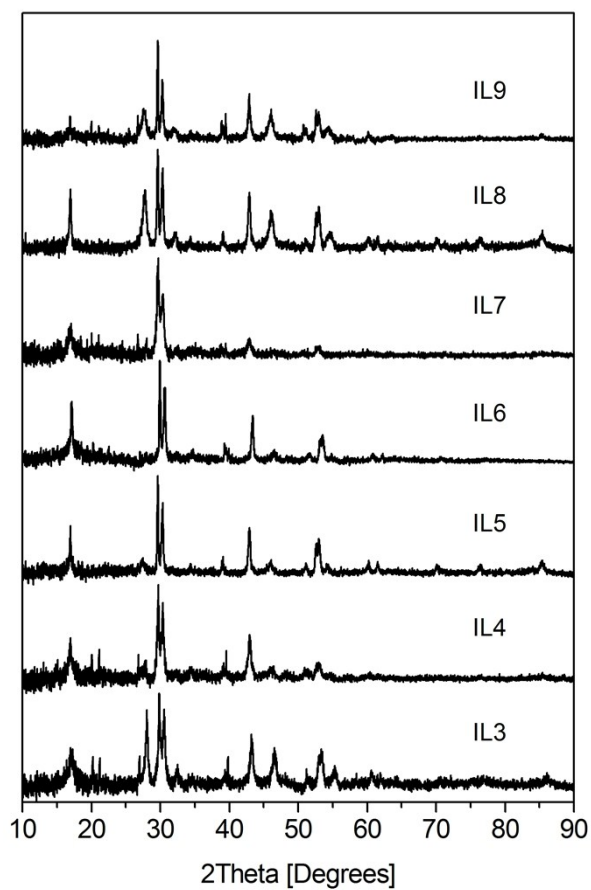
**Fig. S4:** UC luminescence of  $\beta$ -NaGdF<sub>4</sub>: 18% Yb<sup>3+</sup>, 2% Er<sup>3+</sup> nanoparticles from 0.5:1.5 vol. EG/IL solutions with IL = DADMA BF<sub>4</sub> (black trace), DADMA OTf (blue trace), and DADMA NTf<sub>2</sub> (red trace). Nanoparticles from the EG/DADMA TFA synthesis are not shown due to their very weak emission intensity. The UC luminescence was excited at 970 nm with 580 mW (unfocused) laser power.



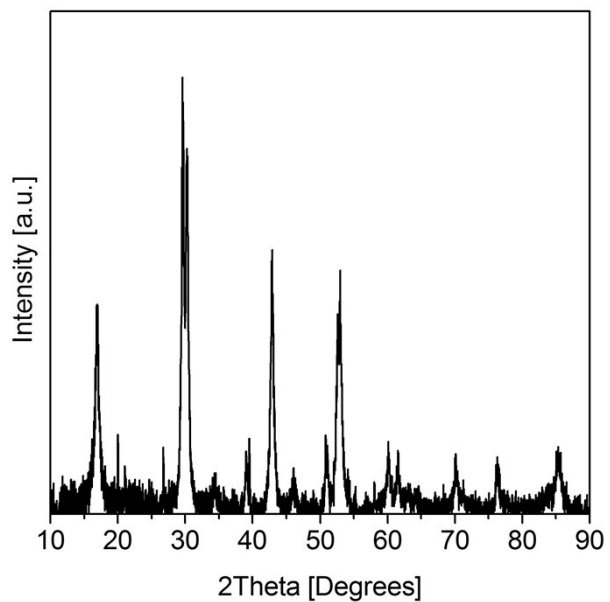
**Fig. S5:** Upconversion luminescence of  $\beta$ -NaGdF<sub>4</sub>: 18% Yb<sup>3+</sup>, 2% Er<sup>3+</sup> nanoparticles from a 0.5:1.5 vol. EG/IL synthesis with IL = DADMA NTf<sub>2</sub> and the rare earth acetate hydrate (blue trace) or the anhydrous rare earth acetate (red trace). Samples were synthesised from 30 mg RE(AcO)<sub>3</sub>, 10 mg NaCl, and 120 mg NH<sub>4</sub>F at 200°C for 30 min. The UC luminescence was excited at 970 nm with 580 mW (unfocused) laser power.



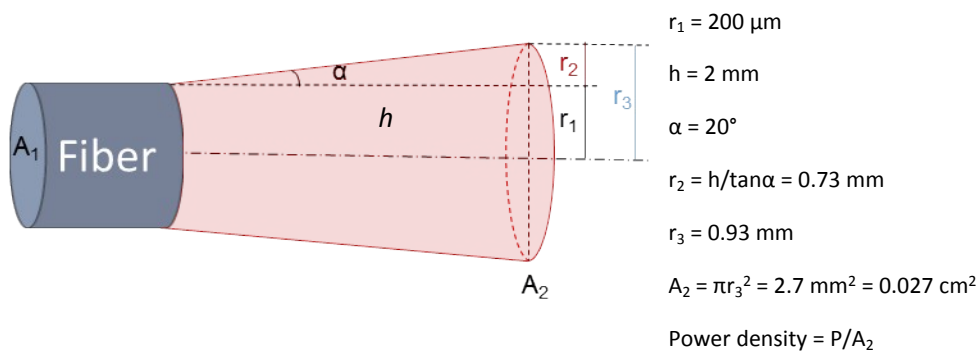
**Fig. S6:** Powder XRD patterns of  $\beta$ -NaGdF<sub>4</sub>: 18% Yb<sup>3+</sup>, 2% Er<sup>3+</sup> nanoparticles from EG/DADMA OTf syntheses. Sample names refer to Tab. 1. NaF peaks are marked by asterisks for the top trace.



**Fig. S7:** Powder XRD patterns of  $\beta$ -NaGdF<sub>4</sub>: 18% Yb<sup>3+</sup>, 2% Er<sup>3+</sup> nanoparticles from IL syntheses. Sample names refer to Tab. 1.



**Fig. S8:** Powder XRD pattern of  $\beta$ -NaGdF<sub>4</sub>: 18% Yb<sup>3+</sup>, 2% Er<sup>3+</sup> /  $\beta$ -NaGdF<sub>4</sub> core-shell nanoparticles from sample CS\_IL4.



**Fig. S9:** Evaluation of the power density from the fiber geometry. The divergence angle  $\alpha = 20^\circ$  and the fiber radius  $r_1 = 200 \mu\text{m}$  determine the illuminated area  $A_2$ . The resulting power densities are reported in Tab. S1.

**Table S1:** Evaluated power density for the measured laser power used in this work.

Power [mW]	Power density [W/cm <sup>2</sup> ]
70	2.6
330	12.2
580	21.5

#### Reference

S1 C. Heinrichs, PhD thesis, Universität zu Köln, Synthese und Charakterisierung wasserfreier Selterdmetall-Nitrate, -Acetate und -Oxyacetate, 2013.