## **Electronic Supplementary Information**

## Novel heterometal-organic complexes as first single source precursors for up-

## converting NaY(Ln)F<sub>4</sub> (Ln = Yb, Er, Tm) nanomaterials

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**Fig. S1** FT-IR spectra of (a) diglyme complexes **1** & **4**-**6**, and (b) triglyme (**2**) and tetraglyme complexes (**3**, **7**, **8**).



Fig. S2  $^{1}$ H NMR spectra of 1 (a), 2 (b) and 3 (c).

3



Fig. S3 TGA (a) and DTA (b) of NaYF<sub>4</sub>:  $Yb^{3+}$ ,  $Er^{3+}/Tm^{3+}$  NCs obtained at different temperature.





**Fig. S4** EDX analysis of the thin films obtained by spin-coating of the diglyme precursors **1**, **4** and **6**. Above: NaYF<sub>4</sub>: Yb<sup>3+</sup>, Er<sup>3+</sup> films on Si wafer & calcined at 400 °C under nitrogen. Below: thin films consisting of the NaF and Y(Ln)OF phases on glass substrate (calcined at 400 °C in air).



**Fig. S5** Emission spectra of NaYF<sub>4</sub>: Yb<sup>3+</sup>, Er<sup>3+</sup> NCs when taken in solid or solution states.



**Fig. S6** Effect of aging of NaYF<sub>4</sub>: 20%  $Yb^{3+}$ , 2%  $Tm^{3+}$  NCs.



**Fig. S7**: Evolution of the intensity of the difference bands with the excitation power: (a) hexagonal phase of NaYF<sub>4</sub>: Yb<sup>3+</sup>, Ln<sup>3+</sup> NCs taken as a solid powder, and b) the cubic phase of NaYF<sub>4</sub>: Yb<sup>3+</sup>, Tm<sup>3+</sup> NCs taken in water.