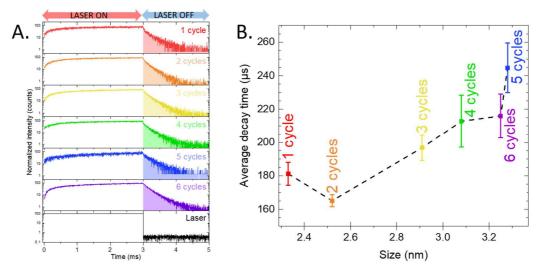
## Investigation of photophysical properties of ultra-small upconverting nanoparticles

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Up-Converting NanoParticles (UCNPs) have a multimodal fluorescence emission when excited in the near IR, which is a great advantage for fluorescence bio-imaging. Small and ultrasmall (sub-10nm and smaller) inorganic nanoparticles are promising tools for nano-imaging. However, reducing the size of UCNPs while preserving their luminescence is challenging. Based on a microwave heating cycling process, it is now possible to get emissive  $\beta$ -crystal phase particles of about 3 nm. Therefore, the influence of the number of heating cycles on size and emission properties needs to be examined in order to determine how much it is possible to decrease the size of the particle while preserving the  $\beta$ -crystal phase that ensures the emission properties. In this aim, steady state and time-resolved spectroscopy were employed to investigate the emission properties of these Tm³+-based UCNPs as a function of the number of heating cycles and compared to the crystal phase structure measured by XRD. Average lifetimes of the 800 nm emission decays show a meaningful step increase that is assigned to the apparition of  $\beta$ -crystal phase (figure 1.A. 1.B).



**Figure 1: A.** Time-resolved 800 nm luminescence of the core particles prepared after 1 to 6 heating cycles (colored curves) using a 976 nm 3 ms-burst cw excitation (black curve). **B.** Average lifetimes calculated for the decays of the 800 nm transition ( ${}^{3}H_{4} \rightarrow {}^{3}H_{6}$ ).

[1] B. Amouroux, C. Roux, J.-D. Marty, M. Pasturel, A. Bouchet, M. Sliwa, O. Leroux, F. Gauffre, C. Coudret, *Inorg. Chem.* **2019**, 58, 5082-5088