# Capillary flow reactor synthesis of upconversion colloidal nanoparticles

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**Introduction**:

Most of the alkali rare-earth fluoride nanocolloids were synthesized using conventional three neck flask process under argon gas blanket by thermal decomposition of trifluoroacetates precursors in high boiling point solvents at 300-350°C. Main disadvantage of conventional synthesis process is temperature ununiformed over the volume of the solvent, one has to use dangerous and hazardous molten salt heater to maintain the uniform temperature and also one cannot change reaction factors like temperature gradients, mixing rate, heating and cooling rate during the synthesis. Complicated injection method is used to remove the solution from the reaction flask at higher temperatures. Abrupt thermal decomposition is required to avoid complicated intermediate compounds that affects the formation of hexagonal $NaYF_4$. Microreactor synthesis can be used to overcome such disadvantages. One can easily attain the higher temperature within the few micrometer flow distance and can change the reaction temperature during the process.

**Synthesis method and results:**

Synthesis of $NaGdF_4$:Yb:Tm has been carried out using capillary flow reactor arrangement madeup of stainless tube with 2 mm iner diameter (Fig.1). Sodium and rare earth trifluoroacetates precursors dissolved in oleylamine and thoroughly degassed at 160°C before being drawn into the syringes. The ratio between Na-trifluoroacetate (Na(TFA)) and Gd, Yb, Tm-trifluoroacetates (Re(TFA)) in the initial solution was kept to 1:1 and 2:1. Glass syringes was used to load the precursor solution, liquid flow was controlled with automated syringe flow controller. The precursor solution allowed to travel through two different coil setup, one was used for the reaction which was in the sand heating bath at high temperature (300°C) for the particles formation and the another in low temperature (80°C) to quench the reaction solution. Argon gas bubbling was used to control the carbon sediment in the reaction. Final solution was washed with several times with ethanol and synthesized nanoparticles have been analyzed with XRD and TEM for the conformation of particle formation. XRD analysis of $NaGdF_4$:Yb:Tm synthesized with different ratio of precursor in oleylamine solvent confirms that it belongs to cubic phase 1:1::Na(TFA):Re(TFA)) and hexagonal phase (2:1::Na(TFA):Re(TFA)) (Fig.2).

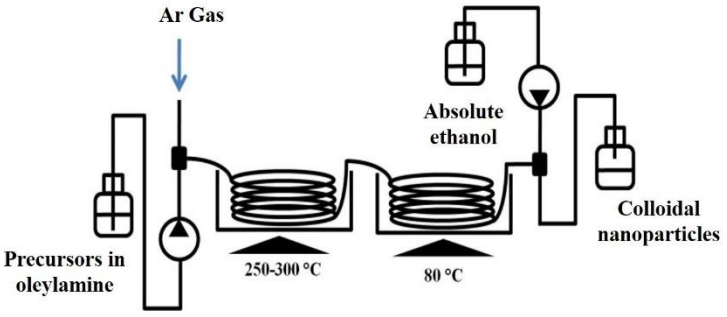


Fig. 1 schematic diagram of flow reactor setup

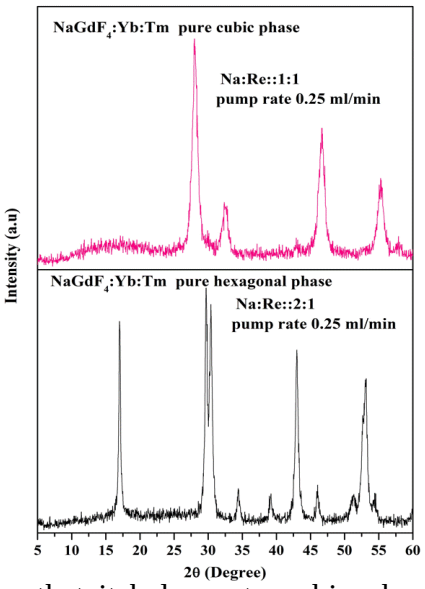


Fig. 2 XRD patterns of 1:1::Na(TFA):Re(TFA) (red curve) and 2:1::Na(TFA):Re(TFA) (black curve)